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Manual
on Laboratory Testing
for Uranium Ore Processing

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MANUAL
ON LABORATORY TESTING
FOR URANIUM ORE PROCESSING

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MANUAL
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FOR URANIUM ORE PROCESSING

FOREWORD

Laboratory testing of uranium ores is an essential step in the economic evaluation of uranium occurrences and in the development of a project for the production of uranium concentrates. Although these tests represent only a small proportion of the total cost of a project, their proper planning, execution and interpretation are of crucial importance.

The main purposes of this manual are to discuss the objectives of metallurgical laboratory ore testing, to show the specific role of these tests in the development of a project, and to provide practical instructions for performing the tests and for interpreting their results. Guidelines on the design of a metallurgical laboratory, on the equipment required to perform the tests and on laboratory safety are also given.

This manual is part of a series of Technical Reports on uranium ore processing being prepared by the IAEA's Division of Nuclear Fuel Cycle and Waste Management. A report on the Significance of Mineralogy in the Development of Flowsheets for Processing Uranium Ores (Technical Reports Series No. 196, 1980) and an instruction manual on Methods for the Estimation of Uranium Ore Reserves (No. 255, 1985) have already been published.

CONTENTS

1. INTRODUCTION

Laboratory testing of ores is an important step in the development of a metallurgical project. Its main purpose is to study the behaviour of a particular ore in selected process environments and to provide information needed, first, to make a preliminary assessment of the economic feasibility of a proposed project and later, if the project is viable, as the basis for further development of a suitable metallurgical process and for the design of an industrial plant.

This section reviews the main metallurgical processes currently in use for uranium extraction. A brief account of standard industrial practice for the development of metallurgical projects is then given, in order to set the laboratory testing of ores in its proper context. The section concludes with a discussion of the objectives of laboratory ore testing and an overview of the objectives of pilot plant testing.

1.1. SUMMARY REVIEW OF URANIUM ORE PROCESSING TECHNOLOGY

The technology for uranium extraction is essentially modern, although some of its elements have evolved from the technology for the extraction of other metals such as gold and vanadium. It has reached a state of maturity although processes continue to be refined to improve their efficiency and flexibility and to lower their costs and reduce their environmental impact.

The extraction of uranium from its ores generally involves the following unit operations (Fig. 1).

(a) Size reduction

The ore is crushed and ground to a suitable range of particle sizes required for effective leaching and to produce a material that can be slurried and pumped through the processing circuits.

(b) Leaching

Uranium is leached (dissolved) from the ground ore using either acid or alkaline (carbonate) solutions with or without heating and with or without the addition of oxidants such as atmospheric oxygen, sodium chlorate, ferric oxide, manganese dioxide and peroxides. Leaching can be done in mechanically agitated vessels, in Pachuca tanks or in autoclaves. Other techniques such as acid pugging, heap leaching, in-place leaching and in situ leaching (solution mining) are also used.

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              Ore
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               ▼
      ┌──────────────────┐
      │  Size reduction  │
      └──────────────────┘
               │
               ▼
        ┌──────────────┐
        │  Leaching    │
        └──────────────┘
               │
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      ┌──────────────────┐
      │  Solid–liquid    │
      │  separation      ├────────▶ Tailings
      │  and washing     │
      └──────────────────┘
               │
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      │  Purification    │
      │  and concentration│
      └──────────────────┘
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      ┌──────────────────┐
      │  Precipitation   │
      │  and solid–liquid│
      │  separation      │
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      │  Drying          │
      │  or calcining    │
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           Uranium
          concentrate
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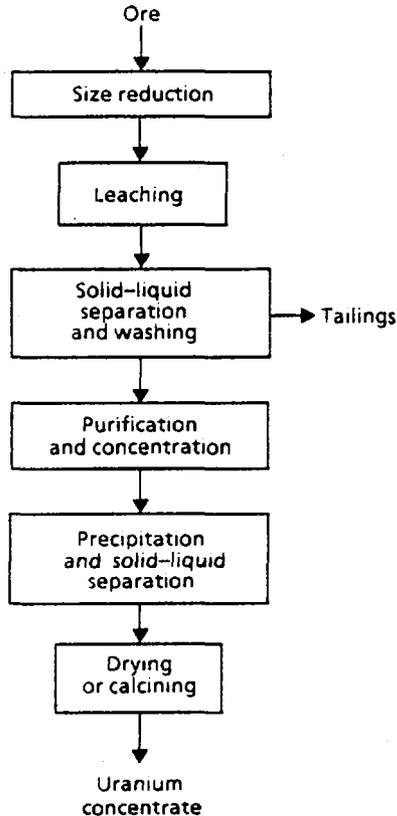
FIG. 1. Generalized process for uranium extraction.

(c) Solid–liquid separation and washing

The pregnant liquor is separated from the leached ore, which is then washed to remove the remaining dissolved uranium. A variety of separation techniques are available, including counter-current decantation, filtration and separation by cyclones.

(d) Purification and concentration

The pregnant liquor contains a low concentration of uranium (usually of the order of 1 g/L) and many impurities. It must therefore be concentrated and purified using ion exchange resins or liquid–liquid extraction (more commonly known as solvent extraction) or the two in tandem.

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(e)   Precipitation and solid–liquid separation

A uranium concentrate (yellow cake) is precipitated from the pregnant solution in one or two stages using any one of a number of reagents, such as sodium hydroxide, ammonium hydroxide, magnesium hydroxide or hydrogen peroxide. The product is either filtered or dewatered by settling and centrifugation.

(f)   Drying or calcining

The product is dried or calcined at temperatures not exceeding 600°C. A wide variety of equipment can be used for this purpose: multiple hearth driers, single hearth driers, drum type units, screw type driers and radiant heat driers.

Another unit operation which is sometimes used is preconcentration of the ore prior to leaching. This has been widely studied but has found only limited application in industrial practice because in many cases the uranium is finely disseminated in the ore. Some of the techniques that have been investigated are: sorting (including radiometric sorting), gravity separation, heavy media separation, separation by differences in magnetic susceptibility and flotation.

The unit operations described in the preceding paragraphs are combined in a suitable form to constitute an actual industrial process. The main processes used by the uranium industry are:

— Acid leaching in mechanically agitated tanks followed by purification and concentration by solvent extraction,
— Alkaline leaching in mechanically agitated tanks followed by purification and concentration by ion exchange,
— Alkaline leaching in Pachuca tanks followed by purification and concentration by ion exchange,
— Acid leaching by pugging and curing followed by purification and concentration by solvent extraction,
— Heap leaching followed by solvent extraction,
— In situ leaching followed by ion exchange.

There are many variations of these basic processes. In the last analysis each plant has a unique process because each ore is unique. Several monographs on uranium extraction technology are available and should be consulted [1–3].

1.2. DEVELOPMENT OF A METALLURGICAL PROJECT

A project consists of a series of activities intended to achieve a specific objective within certain cost and time limitations. A project for the construction of an ore

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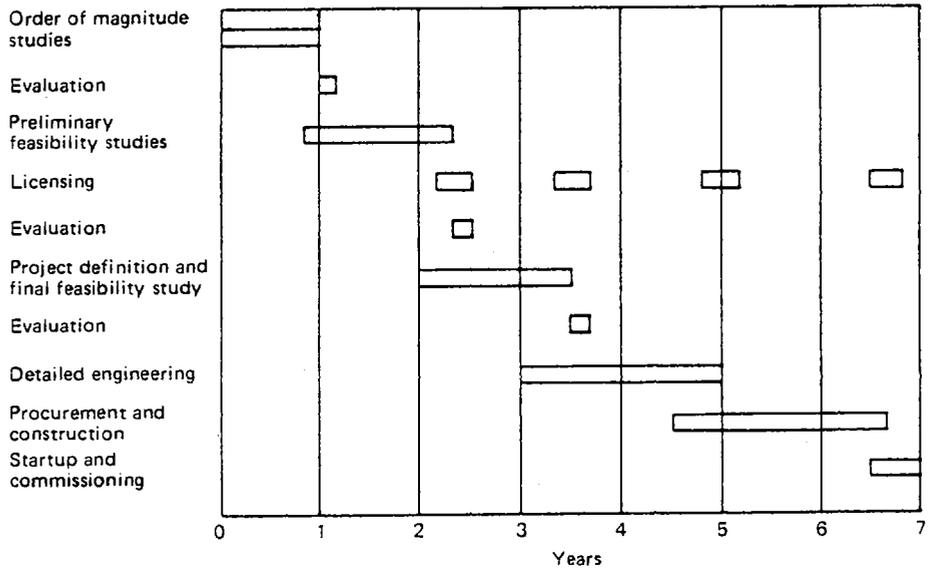
FIG. 2. Phases of project development: an example.

processing plant, after a potential ore body has been discovered, can be considered to include the following main phases (Fig. 2).

(1)   Order of magnitude studies

Rough estimates of the ore body's grade and tonnage are prepared, on the basis of drilling, trenching or other sampling techniques. One or several mining methods are tentatively selected and an estimate is made of the production rate, waste to ore ratio, mining losses and ore dilution. Mineralogical examinations and bench scale metallurgical tests are made using core samples. Preliminary material balances are developed and used to prepare tentative process flow sheets. Order of magnitude estimates of capital and operating costs for the mine and the mill are prepared largely on the basis of published cost data and the costs of similar existing equipment and plants. These estimates are normally expected to be accurate to within 30%.

(2)   Preliminary feasibility studies

The ore body is extensively drilled to establish its type, morphology (in three dimensions), size (also in three dimensions) and degree of homogeneity. The mean ore grade and the ore reserves are calculated. Mining methods are proposed and the grade and tonnage of mineable ore, mining losses, the mine's production rate, the

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expected mine life, the ratio of ore to waste rock and ore dilution are estimated. Detailed mineralogical studies and bench scale metallurgical tests are performed using representative samples, with the primary purpose of selecting and defining a suitable metallurgical process. The tests include all the unit operations necessary to constitute a complete process. The results of the tests are used to define the main process parameters (fineness of grind, solid/liquid ratio, temperatures, residence times, levels of oxidant, pH regimes, etc.) and process efficiency. The size of the plant is tentatively selected and complete flow sheets and material and energy balances are prepared. The main objective of the material balances is to determine the design basis for establishing the size of equipment and other main equipment parameters. The main equipment items are selected from the many options available and a preliminary layout of the plant is prepared. Utility requirements (electrical power, water, fuel and steam) and reagent consumption (sulphuric acid, oxidants, ion exchange resins, extractants, etc.) are calculated.

All this information is now used to prepare new estimates of capital and operating costs which are expected to be accurate to within 20%.

(3) Licensing

Licensing of uranium mines and mills is required in most countries and has become an important phase in the development of a project. In general terms it is necessary to demonstrate that appropriate measures are being taken or will be taken to minimize the environmental impact of the mine and mill and to protect the health of the workers and of the public within the limitations of currently available technology and reasonable costs. Environmental and radiological safety requirements must be taken into consideration from the earliest stages of the project because these requirements will influence the design, construction and operation of the mine and mill. Before any major commitment is made it is essential to have reasonable assurances that the mine and mill will be allowed to operate.

(4) Project definition and final feasibility study

Depending on the nature and complexity of the ore body, additional drilling or other development work is usually required to confirm the reported morphology, ore grade and reserves. Suitable mining methods are developed, taking into account the exact locations of the ore or of the waste zones to be mined, the tonnages and grades of the ore zones and the periods in which these zones are to be mined. The cut-off grade, mining losses, ore to waste ratio, ore dilution and extraction rate are defined. The process flow sheet is refined by further testing, which may include modelling and pilot plant studies. The definitive flow sheet, with the corresponding material and energy balances, is prepared. The selection of equipment and the plant

layout are verified and piping and instrument diagrams and electrical diagrams are prepared. Structural sketches and building sketches are drawn up. A site for the plant is selected and a general study is made of the site and of the infrastructure needed to support the mine and the mill. Definitive estimates of capital and operating costs, accurate to within 10%, are prepared. The project can now be submitted for approval to the management and the institutions that would finance the project.

(5)   Detailed engineering

The final engineering design of all plant equipment and facilities is completed on the basis of conceptual and preliminary engineering designs previously prepared. The final process design includes vessel design, machine design or selection, definitive plant layout, piping design, instrument and control design and electrical design. Final site design and structural, civil and architectural designs are also completed.

(6)   Procurement and construction

The final engineering drawings and specifications are translated into the actual plant. The mine and site are prepared, construction equipment is brought in and construction personnel are hired. Plant equipment and all material items needed are ordered and delivered to the site. The facilities are built by a field task force which is usually a contracting company.

(7)   Startup and commissioning

All the equipment and the piping, electrical, materials handling and control systems are checked and any problems corrected. The plant is started, operating first at reduced capacity and finally at full rated capacity. Custody and control of the plant are transferred to the operator.

(8)   Decommissioning

At some point operations will end and the mine, mill and tailings dams must be decommissioned. Concrete provisions for this final stage of the project must be made during the design and construction stages. It is also necessary to prepare in advance a decommissioning plan, schedule and budget and to ensure that there will be funds available to execute the plan.

These phases of development are not strictly sequential. They usually overlap, as shown in Fig. 2. This is only an example of a typical time sequence, but wide variations can occur in actual projects. For instance, licensing may take much longer than one year in some countries. In addition, and concurrently with the main activi-

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ties outlined above, it is necessary to institute and implement a programme of quality assurance.

Experience has shown that it is very important to go through all the main project phases in the order outlined above. Bypassing phases is seldom beneficial in the long run and usually results in additional expenses and delays.

An evaluation of the project is made at the conclusion of the order of magnitude studies, of the preliminary feasibility studies and of the final feasibility study. A decision is taken at each of these points to continue the project, or to defer or abandon it. Many factors must be considered when making these decisions. Some of the factors involved are as follows:

(a) *Geological:* the physical nature of the deposit, including its size and shape, the distribution of mineralized areas, the characteristics of the host rocks and the structural environment;

(b) *Mineralogical:* the chemical and physical characteristics of the uranium minerals as they affect the ease of extraction of the metal from the host rock;

(c) *Infrastructure:* the availability of water, electricity, reagents, parts, roads, railways, other means of transportation and communication, other services required to operate the mine and mill, and facilities and services needed by the personnel;

(d) *Technological:* the mining and processing techniques available and applicable to the deposit;

(e) *Economic:* the assurance that a minimum required rate of return on investment capital can be realized, taking into consideration capital and operating costs, taxes, terms of contracts, the current and expected demand and market price of uranium and the probable economic outlook;

(f) *Human:* the availability of personnel and of technical and administrative skills, and the adoption of an effective operational plan;

(g) *Environmental:* the effects of mining and milling on the environment and the requirements of governmental regulations;

(h) *Legal:* national and local regulations regarding mineral rights, royalty regimes, use of land and water, taxation and other matters;

(i) *Political:* the international, national and local political factors which may affect access to the ore body and the conditions under which the project may be implemented and the mine and mill allowed to operate.

All of these factors directly affect the feasibility of mining and processing uranium. Other factors not mentioned above may also have an effect and should be considered when appropriate.

The time needed for the development of a project depends upon many factors, such as:

— The size of the project, i.e. the rated throughput (tonnes of ore per day) of the plant;

— The complexity of the project;

— The degree of novelty of the project, that is, how much it differs from similar projects that have already been completed and proven;

— The availability of services such as roads, railways and other means of transportation and communication, and electricity, water and other supplies;

— The availability of skilled manpower;

— The experience of the various engineering teams in charge of the project;

— The form in which the project is planned, organized and implemented;

— Adequate and timely financing of the project.

1.3. OBJECTIVES OF LABORATORY TESTING OF URANIUM ORES

Laboratory testing of uranium ores is an essential step in the development of a project for the production of uranium concentrates. Its main purposes are:

(a)   To study in a systematic manner the behaviour of a particular ore in selected process environments, i.e. to determine whether uranium can be efficiently extracted from the ore in question using any of the proven processes used by the industry;

(b)   To assess the physical and chemical variability of the ore to be processed;

(c)   To provide data for the selection and definition of a metallurgical process for an industrial plant;

(d)   To provide data for the preliminary selection of the type and size of the equipment that will be needed;

(e)   To provide data for order of magnitude and preliminary evaluations of the economic feasibility of the project.

Although laboratory tests represent only a small proportion of the total cost of a project, they are of crucial importance. Two main types of laboratory test may be considered:

Preliminary tests, to be performed at an early stage in the development of a prospect, with the primary purpose of helping to determine the technical and economic feasibility of processing the ore. These tests may be performed on grab samples or on core samples that need not be representative of the ore body as a whole. Also, at this stage it is only necessary to cover the first process stages: size reduction, leaching and solid–liquid separation. If it is possible to dissolve most of the uranium present in the samples and if it is possible to obtain a clear pregnant liquor, it will generally be possible to purify and concentrate this liquor and to obtain a uranium concentrate of good quality. On the other hand, several of the major process alternatives may be considered at this stage: acid or alkaline leaching, heap leaching, etc.

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Detailed tests, to be performed at a more advanced stage in the development of the project, once its economic potential has been established with some certainty, with the primary purpose of selecting and defining a suitable process. At this stage the ore body should be reasonably well defined and evaluated. The type of ore body and its morphology (in three dimensions) should be known. Tentative mining methods should have been proposed so that the grade and tonnage of the mineable ore, the production rate, the ratio of ore to waste rock, mining losses and ore dilution can be estimated. It is also important at this stage to have a good mineralogical and petrographic characterization of the ore. The detailed tests must be performed on samples that are reasonably representative of the ore body as a whole and should include all the unit operations necessary to constitute a complete process.

1.4. OBJECTIVES OF PILOT PLANT TESTING

A detailed discussion of pilot plant testing is beyond the scope of this report. However, a brief discussion of the objectives of pilot plant testing is pertinent, if only to show the specific role of laboratory testing.

A pilot plant is a functional model of an industrial plant, built at a reduced scale, but using the same materials of construction and capable of reproducing the conditions of the plant being modelled. A pilot plant is a scaled-down version of the expected final plant. It is not a scaled-up version of laboratory apparatus [4]. It should be noted, however, that a fully integrated pilot plant may not always be needed or even desirable. Pilot scale testing may only be needed for some parts of the process, especially those that differ substantially from standard industrial practice or that have never been tested before, or those that bench scale work has shown to be troublesome.

The overall objective of pilot plant testing is to ensure that the decision to proceed with the full scale project is based on a proven process and on a reliable economic evaluation. Other, more particular objectives of the pilot plant are:

— To optimize the operating parameters of the process,
— To study the effects of recirculating process streams and of accumulation of impurities over long periods,
— To obtain process information necessary to specify and design the full scale plant,
— To test process control systems and procedures,
— To test materials of construction,
— To optimize the design of the equipment,
— To obtain sufficient information to prepare detailed and reliable estimates of capital and operating costs and to prepare a reliable economic evaluation of the project,

— To gain operating experience and to train the personnel that will operate the full scale plant,

— To verify that the process can be used in a safe manner,

— To produce a reasonable amount of product for characterization studies.

The design, construction and operation of a pilot plant are expensive and time consuming. They are only undertaken when there are already strong indications that the project is economically viable. A pilot plant may be considered after:

(a) An ore body with significant proven reserves at a workable grade under prevailing market conditions has been identified;

(b) The ore body has been sufficiently developed so that representative ore samples are available in enough quantity to feed the pilot plant continuously during the test period, which may be from several months to a year or more;

(c) A metallurgical process has been selected and its main parameters have been defined through bench scale testing;

(d) Preliminary feasibility studies have been performed and these studies have indicated that the project is potentially viable.

2. SAMPLING AND CHARACTERIZATION

Selection and preparation of the ore test samples are two of the most critical steps of the overall metallurgical evaluation process. Process evaluation results can be no better than the samples being tested. For meaningful results the test samples must contain, in unchanged percentages, all of the constituents in the original ore lot. Disregarding these criteria can seriously reduce the value of the process evaluation data.

2.1. SAMPLE SELECTION AND COMPOSITING

No single uranium ore is identical to another because the rocks that make up an ore are complex assemblages of minerals and can differ widely in composition and texture. Even within a given uranium deposit, the ore can vary significantly from one part of the deposit to another. A flow sheet cannot be realistically optimized for all variations and is always a compromise of many factors. When samples are being collected for process development studies, the potential variability should always be kept in mind. Two different classes of samples should be collected and studied. With one class an attempt should be made to simulate an overall composite mill feed, and the second class should be a group of samples which represent the variability that may be encountered in various parts of the deposit. It is therefore highly desirable

for the metallurgist to be part of the team which selects even the preliminary metallurgical test samples. This team might include geologists, mineralogists, mining engineers and process metallurgists. The chances of a successful process development can be significantly increased if the metallurgist is directly involved in the sample selection. It is particularly important that a metallurgist be present at the mine site while samples are being collected for process development studies. This will help the metallurgist to gain a better appreciation of the limitations of the sampling procedure and of the variability which may be encountered.

Metallurgical test samples can come from a variety of sources. The following types of sample are often encountered:

— Outcrop or surface samples from the target area
— Percussion drill samples
— Trench samples
— Diamond drill samples
— Channel samples or other types of sample from underground workings.

The type of deposit determines which of these samples may be available. For example, all five types of sample may be quite easily obtained from shallow sandstone deposits, while only diamond drill samples may be initially available from deeper deposits of the disseminated type.

Often the first samples to become available are from an oxidized zone of the deposit and may substantially differ from ores found in lower, unoxidized zones. It is therefore desirable that the individual zone samples be collected and kept separate; composites should not be prepared until the variability is assessed and at least a preliminary ore reserve distribution can be estimated. The metallurgist should work closely with the geologists and mineralogists to determine how to composite materials from various parts of the deposit. Even after these decisions are made, portions of the original samples should be retained so that a more detailed study of the metallurgical or mineralogical characteristics can be made if difficulties are encountered during the process development studies.

2.2. SAMPLE IDENTIFICATION

A sample numbering and identification system should be set up at the very start of the metallurgical evaluation programme. It is often desirable that the system for identifying the head or feed samples be an extension of the identification system used by the geologists and mining engineers to designate the various ore types or horizons. A permanent record system should be kept which references the sample number to all critical information such as the location where the sample was taken, the sampling dates, all analysis data, and mineralogical information. The head sample containers should be labelled with at least the full sample identification number. The label

should be permanent. It should be written with paint or with a waterproof marker. Paper sacks should be labelled directly on the sack. It is almost always desirable to use double paper sacks, i.e. one inside the other. Cloth sacks should be labelled both inside and out. If a tag is tied to the outside, a second tag should also be placed inside the sack. It is a good idea to label steel drums with paint on the outside of the drum and also to put a cardboard label inside the drum. Placing the cardboard label in a sealed plastic sack will keep the label dry if the ore is somewhat damp. If a large sample requires more than one sack or drum, each label should show the total number of sacks or drums so that one sack will not be taken to be the entire sample when actually there are more.

2.3. SAMPLE PREPARATION

Correct sampling and preparation of an ore lot for analysis or testing are necessary for obtaining a smaller quantity that contains, in unchanged percentages, all the constituents of the original lot.

Every time an ore is handled, the particles tend to segregate according to size and specific gravity. In considering the reliability of any sampling method this tendency to segregate should be constantly borne in mind. Any detail of the sampling procedure that may tend to make possible the selection or rejection of either the finer or coarser particles should be considered inaccurate. So absolutely does the accuracy of a sampling method depend on freedom from selection or rejection that it may be forgotten that uranium or other metals are to be determined by the process and the method may be examined solely as to its ability to take from an ore lot the same proportions of all the various sizes of particles.

The process of preparing an ore sample for laboratory testing can be thought of as a sequence of two-step operations which are repeated until the desired amount of sample is obtained. In the first step, the ore lot is crushed to the appropriate size and in the second step, the crushed ore is mixed and divided into two equal portions each of which contains the same proportions of all the various sizes of particles. The crushing size depends upon both the type of ore being sampled and the weight of the ore lot available. The desirable particle size for a given amount of a uniform low grade ore can be considerably larger than the particle size necessary for the same weight of an inhomogeneous, relatively high grade ore. Table I presents sample weights and crushing sizes that have been found to be appropriate for relatively homogeneous sandstone uranium ores containing 0.1–0.3% U_3O_8.

Various hand sampling techniques have been used to prepare uranium ores for test work, but coning and quartering followed by riffling has probably been the most widely used procedure. Other techniques, including grab sampling, shovel sampling, pipe sampling and mechanical sampling, have also been used. Mechanical sampling

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TABLE I. SMALLEST PERMISSIBLE SAMPLE WEIGHTS FOR VARIOUS CRUSHING SIZES

When crushed to:		Smallest permissible weight	
(in)	(mm)	(lb)	(kg)
2	50.8	2000	908
1	25.4	1000	454
0.75	19.1	250	114
0.5	12.7	125	56.8
0.25	6.35	60	27.2

techniques are the most accurate, but suitable mechanical sampling equipment is not often available during the initial phases of an ore evaluation programme.

2.3.1. Coning and quartering

Taggart [5, pp. 19-27, 19-28] has presented the following general description of the coning and quartering technique; the procedure is applicable for nearly all uranium ores.

"This is one of the oldest forms of hand sampling. It was for many years the standard method of sampling throughout the western United States, especially for batches of ore whose value was to be determined between buyer and seller. It can be used on lots up to 50 tons.[1] Where larger lots are to be sampled, the first cut is made by some other method and the sample thus obtained is further reduced by coning and quartering.

"**Procedure** consists in piling the ore into a conical heap, spreading this out into a circular cake, dividing the cake radially into quarters, taking opposite quarters as sample and rejecting the other two. The ore should be crushed through 2-in. or smaller ring.[2] The operation should be carried on in a room of sufficient size to allow convenient handling of material. The floor should be smooth and free from cracks, preferably smooth concrete or steel sheets; it should first be swept thoroughly clean to avoid salting from a previous lot. The ore is dumped on the floor in two or four piles, or in a circular ring. Shovelers then pile this ore into a right-conical heap conveniently placed, taking care to drop each shovelful directly onto the apex. The object of coning

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[1] 1 ton (short) = 907.2 kg.
[2] 1 in. = 2.54 cm.

is to form a heap in which segregation shall be symmetrical with respect to the vertical axis. To insure this, successive shovel loads should be so taken as to contain similar sizes similarly segregated on the shovel and should be so dropped onto the cone that the segregation therein is symmetrical. This end is attained by having the shovelers take successive shovel loads from adjacent places around the periphery of the heaps from which they are shoveling and drop these loads from successively adjacent points around the cone that they are building. Each man should take a shovel load of the same size as each other shoveler, and shovel loads should be made smaller as the size of the heap being shoveled becomes less. When the material is placed in a ring the men move around the ring as they shovel into the cone. When the ore is all heaped up into a cone, the floor is carefully swept and the fines collected are placed on the apex, not swept up against the bottom of the pile. The men then start at points near the bottom of the cone and, with their shovels held tangentially, drag the material down radially so as to form a truncated cone or flat circular cake. In this operation they should work around the cone. The cake is then marked off into quarters with a stick or board along diameters at right angles. Opposite quarters are shoveled out as reject. The remaining quarters, called the sample, are then shoveled into one or more piles or into a ring depending on the amount of material, and the coning and quartering operations are repeated until the sample is so small that further crushing is necessary before further reduction in bulk can be made. A cross made of sheet iron or wood with sharpened edges is often used to mark the truncated cone into quarters. This is placed on top of the cake with the center of the cross directly over the center of the cone and is then pressed into the ore until it touches the floor. Such an arrangement is preferable to marking off with a single board. Lines of division will be more exact and the cross can be left in place to hold the sides of the sample quarters vertical while the reject quarters are being shoveled away. Or the cross may be laid on the floor before starting the pile and each shovelful dropped over the intersection. The cone is then spread out to the thickness of the cross and the reject shoveled away as above.

"**Coning** does not mix the ore uniformly. As material is shoveled onto the cone the coarser pieces roll down the sides and come to rest on the floor while the finest particles remain near the apex. Pieces of intermediate size arrange themselves on the slopes of the pile according to their size. The ultimate result desired is that the segregation be symmetrical with respect to the axis of the cone. If this condition is attained, any sector taken should correctly represent the whole.

"**Bench or cobbing system,** sometimes used to get better distribution of the material in the cone, consists in first making a small cone of some of the ore and spreading it out into a cake, then making another cone on the center of the cake and spreading it out and repeating until all the ore is thus disposed

of. This method tends to reduce the effects of accidental errors in flattening the cone.''

Taggart [5, pp. 19-29, 19-30] and Perry [6, pp. 1098, 1099] have presented the following example for sampling coal; the same sequence of operations has also been found to be applicable for most low and medium grade uranium ores (0.03–0.3% U_3O_8). The procedure is a combination of several hand sampling techniques. A symbolic representation of the steps is shown in Fig. 3. The numbers in the text refer to the step numbers in the figure.

"*1,000-lb gross sample.*[3] Crushed to 1 in (1); mixed by shoveling into a conical pile (2), then shoveled into a pile 5 to 10 ft long[4] (3) and alternate shovels taken (4) giving a *500-lb sample* (5), which is crushed to 3/4 in (6) and treated as above, giving a *250-lb sample* (7–10). This is crushed to 1/2 in (11), mixed, by shoveling into a conical pile (12) and then shoveled into another cone (13), flattened and quartered, giving a *125-lb sample* (14–16), which is crushed to 3/8 in (17), placed on a tightly woven canvas or rubber covered rolling cloth (6 ft × 8 ft), mixed by rolling (18), coned by drawing up the ends of the cloth (19), then flattened and quartered, giving a *60-lb sample* (20–22). This is crushed to 1/4 in (23), placed on the rolling cloth and the preceding operation repeated, giving a *30-lb sample* (24–28) which is crushed to 3/16 in and again subjected to rolling, coning, flattening, and quartering, giving a *15-lb sample* (29–34).''

Each of the final 15 lb splits can be saved in separate containers and subsequently ground to the size desired for leaching or other types of test. An alternative procedure is to take the entire 60 lb sample (step 22), crush the material to −10 mesh (<1.651 mm) and then split down the sample using a Jones splitter or equivalent type of riffle splitter.

2.3.2. Splitting

A large variety of splitters or riffles are available; the Jones splitter, shown in Fig. 4, is one of the most commonly used. It is used to divide a sample into two parts and consists of a number of equally sized chutes (A) discharging to opposite sides. The Jones and other splitters are available in a number of sizes. In splitting a sample, a splitter should be used that has chute widths somewhat larger than the largest particle in the sample; this will ensure a free flow through the chutes.

It is most important that the material be fed to the splitter from a scoop or shovel (B) of the same width as the splitter. The lip of the scoop should be placed

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[3] 1 lb = 0.4536 kg.
[4] 1 ft = 0.3048 m.
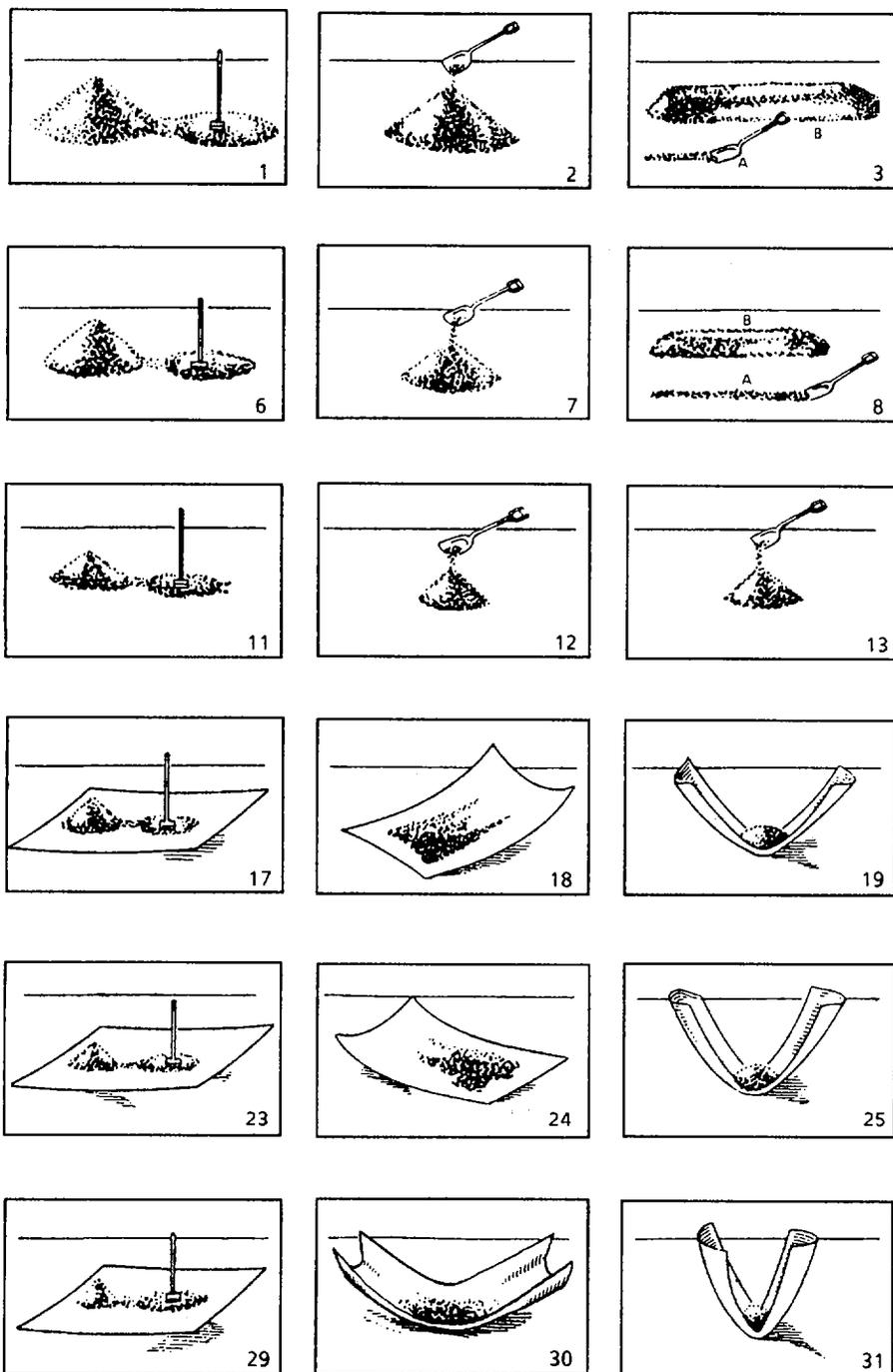
FIG. 3. Coning, quartering and rolling [6].
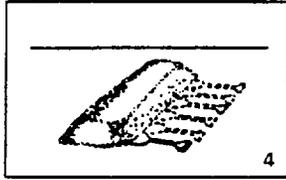
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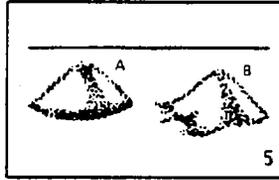
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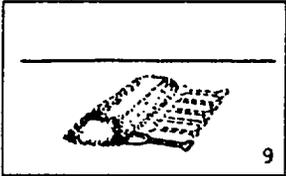
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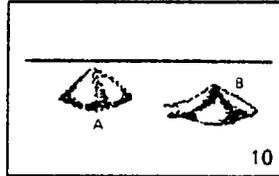
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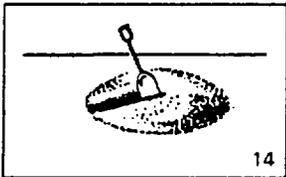
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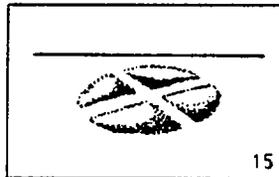
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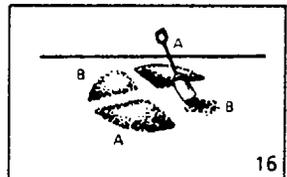
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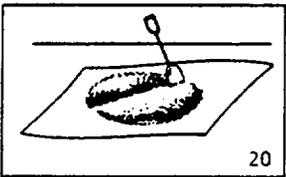
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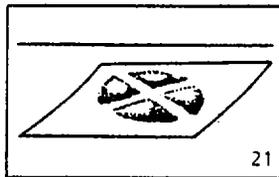
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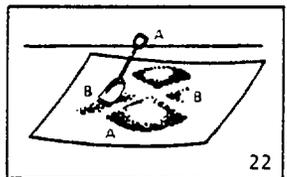
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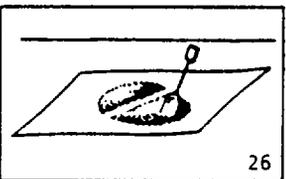
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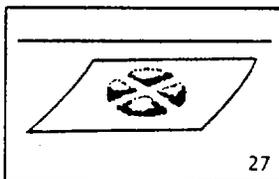
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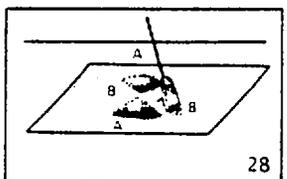
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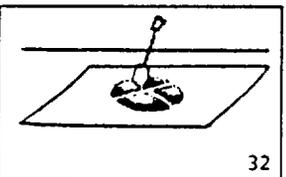
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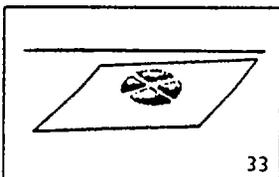
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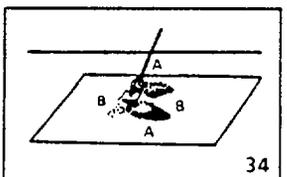
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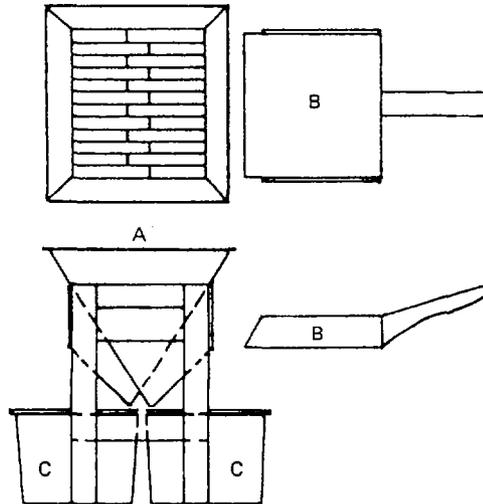
FIG. 4. Jones splitter. A: splitter; B: scoop for spreading ore over riffles; C: pans for receiving split sample and rejects.

at the edge of the splitter hopper and the material then dumped into the splitter. The most common error is to pour the material into the splitter in a small continuous stream which is moved back and forth across the splitter; this should never be done. The pans (C) that are provided with the splitter to catch the products are almost always of the same width as the splitter and can also be used to feed the splitter if a suitable scoop is not available.

As an example of the splitting technique, it is supposed that a sample has been reduced to about 32 kg and ground to -10 mesh. A final sample weighing about 1 kg is wanted. After the pans, splitter and floor area have been thoroughly cleaned, the pans are placed below the splitter and the material is fed to the splitter with the proper scoop. With successive deliveries the material is fed first from one side of the splitter and then the other. It is likely that this splitting has filled a total of at least four pans (two on each side), so two from one side are dumped into a holding container and the other two are split again. If the first pan is dumped into the splitter from the right hand side, the second is dumped from the left. The original sample has now been reduced to two pans, each containing approximately 8 kg. One pan is dumped into the holding container and the other again put through the splitter. This process is continued, the quantity being reduced by about half each time, until the desired amount of sample is reached. A large splitter would be used for the initial 32 kg, but as the amount of material to be split becomes smaller, say less than a quarter-panful, the final divisions should be made with a smaller splitter. The splitter should be rapped smartly between splits to ensure that the slots are clean.

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The Jones splitter can be used not only to reduce the quantity of material and produce a single sample for analysis but also to divide a sample into a number of representative portions for leaching or other test work. For example, if 16 leach tests (500 g each) are to be made on an ore, the ore lot is split, as previously described, until one of the pans contains a little over 8 kg of ore. This material is then ground to the desired size, mixed and put through the splitter, as previously described, until two approximately 500 g samples have been produced by the last split. In this case all of the previous reject splits are saved separately and the splitting operation is performed on each of these until the entire 8 kg has been split into the desired 16 500 g samples. When this operation is performed carefully, each portion will be of nearly the same weight and only small subtractions or additions will be needed to bring the portion to the exact weight wanted. Also, one portion may be used as a head sample for all of the tests.

2.3.3. Sampling test products

Obtaining a representative sample of dried leaching residues or other test products for analysis requires particular care. Thorough mixing of the material is particularly important, and since the test products are usually quite fine, several different mixing techniques are applicable.

The method most frequently used is called rolling, and consists of rolling the material back and forth upon itself on a piece of glazed paper, rubber or oilcloth, ordinarily referred to as a rolling cloth. The rolling cloth should be square, or nearly so, and a little larger than the area covered by the material so that there will be no spillage while rolling. In this mixing method, the material is placed in the centre of the cloth and diagonally opposite corners of the cloth are grasped with either hand. Then, first with one corner and then the other, the cloth is drawn horizontally towards the opposite corner. This process is repeated using the other two corners, then the original two corners, etc., until the material has been rolled about a hundred times. The order of drawing across the cloth corners may be in either clockwise or anticlockwise sequence around the cloth instead of taking opposite corners. In this method the material is made to roll over and mix with itself. A common error made by persons unfamiliar with the proper technique of rolling is that they merely lift or shake the corners of the cloth instead of drawing it across the material. When this is done, the particles merely slide along the cloth and no mixing is accomplished. It is also important that the size of the cloth fit the size of the sample. It is practically impossible to mix a small amount of material on an oversize cloth because the material will always tend to slide rather than roll.

After mixing, the sample portion required for analysis may be obtained by splitting with a small riffle or by spreading the material on a rolling cloth with a spatula and then taking small portions with the tip of the spatula (being certain to go through to the cloth each time) from scattered points throughout the pile. Normally

the size of this analytical sample should be approximately 100 g. This material should be pulverized to −120 mesh (<0.125 mm) and mixed again by rolling a hundred times before being placed in a properly labelled sample container.

2.4. MINERALOGICAL CHARACTERIZATION

Both the metallurgical and economic processing characteristics of a uranium ore depend upon the mineralogical composition and texture of the ore. Processing factors influenced by the mineralogy of the ore, as distinct from the uranium grade, include the following:

— The degree of grinding required to liberate or expose the uranium minerals;
— The potential for physical beneficiation of the uranium minerals;
— The preferred lixiviant system and the potential level of reagent consumption;
— The probable rheological properties of the leach pulps and the resultant effects on agitation, thickening, filtration, etc.;
— The probable ionic composition and concentration of the leach liquor.

The relationships between these various factors and the overall processing characteristics of any given uranium ore are often quite complex and beyond the scope of this manual. The reader is referred to IAEA Technical Reports Series No. 196, Significance of Mineralogy in the Development of Flowsheets for Processing Uranium Ores [2]. It is desirable, however, to keep the following in mind:

Uranium minerals: At least 185 different uranium minerals have been identified in the literature and include oxides, silicates, phosphates, sulphates, carbonates and molybdates. Some of the more common minerals are listed in Table II. Often, however, only a portion of the total uranium content of an ore can be ascribed to specific uranium minerals. For example, in some relatively low grade sandstone ores, less than 20% of the uranium content can be identified as specific minerals. Also, uranium can be present in either the tetravalent or hexavalent form. Oxidation is required for dissolution of minerals containing tetravalent uranium, but the hexavalent uranium goes into solution without the addition of oxidants. Several uranium bearing minerals are refractory. Chemical analysis will indicate their total uranium content, but some or even most of this uranium cannot be recovered economically. In some rocks more than 80% of the uranium can be refractory.

Gangue minerals: Most of the reagent requirement for leaching uranium ores is directly related to the gangue constituents. Technical Reports Series No. 196 [2, pp. 25, 26] summarizes the chemical response of the gangue materials as follows:

"*Quartz:* Quartz is a non-reactive mineral and the fact that it is the major component in some of the world's largest uranium occurrences, such as the

conglomerates of South Africa and Canada, contributes (by default of other reactive minerals) in no small measure to the economic exploitability of these ores.

"*Carbonates:* Although some dolomites and siderites react relatively slowly, carbonate minerals in general consume their stoichiometric equivalent of acid at the pH required for the dissolution of uranium minerals, and their presence as a major component has usually been the determining factor in initiating the consideration of alkaline carbonate leaching. Gypsum by contrast is a problem in the alkaline circuit, reacting to form calcium carbonate and forming sodium sulphate in solution.

"*Phosphates:* Apatite is not as reactive in acid as carbonate and its reactivity is variable. However, to a greater or lesser degree, it consumes acid at relatively low acidities and its rate of dissolution at pH1.5 or less is significant. In addition, the phosphate going into solution can complex the ferric ions and thus inhibit their role in the oxidation process. Another effect is that, in subsequent processing, phosphate ions can cause reprecipitation of uranium if the pH is not maintained below 2.0. Finally, some phosphate may find its way to the yellow cake in unacceptable amounts.

"*Silicates:* Of the silicate minerals little systematic work has been done to indicate a particular order of reactivity. The Bowen's Reaction Series can give some indication. Certainly, of the 'primary' and unweathered rock-forming minerals, it is reasonable to expect that the more basic the mineral the more reactive to acid it might be. This is illustrated in the reactivity of the allanite host mineral in the Mary Kathleen ore of Australia.
"However, the silicate minerals occurring in most of the world's large uranium deposits are those associated with weathered material in sedimentary rocks, in metasediments and their weathering products.
"It is these minerals — biotite, chlorite, sericite and various clay minerals and their various proportions — which in the absence of highly reactive minerals, tend to effect the greatest control in the treatment process, both leaching and subsequent operations. All of them react in acid to a greater or lesser extent at pH below 2.0 and can be particularly reactive below pH1.5. The final choice of acidity, time and temperature of leach, to optimize the recovery and economic value of the recovery, is to a large extent controlled by the differential rate of attack on these ore minerals as compared with the uranium. These optimum conditions can only be determined by experimental testing procedures.

"*Iron oxides:* The reactivity of the various iron oxides and hydrous oxides is also variable but the rate of dissolution is usually significant at the pH range used in acid leaching of uranium ores. In fact these minerals and the

TABLE II. URANIUM MINERALS

Oxides

Becquerelite	$7UO_3 \cdot 11H_2O$
Billietite	$BaO \cdot 6UO_3 \cdot 11H_2O$
Cerianite	CeO_2
Clarkeite	$(Na,K,Ca,Pb)U_2O_7 \cdot nH_2O$
Curite	$PbO \cdot 8UO_3 \cdot 4H_2O$
Fourmarierite	$PbO \cdot 4UO_3 \cdot 5H_2O$
Ianthinite	$2UO_2 \cdot 7H_2O$ (?)
Masuyite	$UO_3 \cdot 2H_2O$
Pitchblende	UO_2
Richetite	Hydrated oxide of U and Pb (?)
Schoepite	$2UO_3 \cdot 5H_2O$
Thorianite	ThO_2
Uraninite	UO_2
Uranosphaerite	$(BiO)(UO_2)(OH)_3$ (?)
Vandenbrandeite	$Cu(UO_2)O_2 \cdot 2H_2O$
Vandendriesscheite	$PbO \cdot 7UO_3 \cdot 12H_2O$

Fluorides

Schroeckingerite	$NaCa_3(UO_2)(CO_3)_3(SO_4)F \cdot 10H_2O$

Carbonates

Andersonite	$Na_2Ca(UO_2)(CO_3)_3 \cdot 6H_2O$
Bayleyite	$Mg_2(UO_2)(CO_3)_3 \cdot 18H_2O$
Liebigite	$Ca_2(UO_2)(CO_3)_3 \cdot 10H_2O$
Rabbittite	$Ca_3Mg_3(UO_2)_2(CO_3)_6(OH)_4 \cdot 18H_2O$
Rutherfordine	$(UO_2)(CO_3)$
Schroeckingerite	$NaCa_3(UO_2)(CO_3)_3(SO_4)F \cdot 10H_2O$
Sharpite	$(UO_2)(CO_3) \cdot H_2O$ (?)
Studtite	Hydrated carbonate of U (?), $UO_4 \cdot 4H_2O$
Swartzite	$CaMg(UO_2)(CO_3)_3 \cdot 12H_2O$
Voglite	$Ca_2Cu(UO_2)(CO_3)_4 \cdot 6H_2O$ (?)

Sulphates

Johannite	$Cu(UO_2)_2(SO_4)_2(OH)_2 \cdot 6H_2O$
Uranopilite	$(UO_2)_6(SO_4)(OH)_{10} \cdot 12H_2O$
Zippeite	$2UO_3 \cdot SO_3 \cdot 5H_2O$

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TABLE II. (cont.)

Molybdates

Umohoite $(UO_2)(MoO_4) \cdot 4H_2O$

Arsenates

Abernathyite $K_2(UO_2)(AsO_4)_2 \cdot 8H_2O$

Kahlerite $Fe(UO_2)_2(AsO_4)_2 \cdot 8H_2O$

Metazeunerite $Cu(UO_2)_2(AsO_4)_2 \cdot 8H_2O$

Novacekite $Mg(UO_2)_2(AsO_4)_2 \cdot 8{-}10H_2O$

Troegerite $H_2(UO_2)_2(AsO_4)_2 \cdot 8H_2O$

Uranospathite $Cu(UO_2)_2(AsO_4,PO_4)_2 \cdot 16H_2O$ (?)

Uranospinite $Ca(UO_2)_2(AsO_4)_2 \cdot 10H_2O$

Walpurgite $Bi_4(UO_2)(AsO_4)_2O_4 \cdot 3H_2O$

Zeunerite $Cu(UO_2)_2(AsO_4)_2 \cdot 10{-}16H_2O$

Phosphates

Autunite $Ca(UO_2)_2(PO_4)_2 \cdot 10{-}12H_2O$

Bassetite $Fe(UO_2)_2(PO_4)_2 \cdot 8H_2O$

Cheralite $(Th,Ca,Ce,U,Pb)(PO_4,SiO_4)$

Dewindtite $Pb_3(UO_2)_6(PO_4)_4(OH)_6 \cdot 10H_2O$ (?)

Dumontite $Pb_2(UO_2)_3(PO_4)_2(OH)_4 \cdot 3H_2O$

Fritzscheite $Mn(UO_2)_2(PO_4,VO_4)_2 \cdot 8H_2O$ (?)

Meta-autunite $Ca(UO_2)_2(PO_4)_2 \cdot 8H_2O$

Metatorbernite $Cu(UO_2)_2(PO_4)_2 \cdot 8H_2O$

Meta-uranocircite $Ba(UO_2)_2(PO_4)_2 \cdot 8H_2O$

Monazite $(Ce,La,Th)(PO_4)$

Parsonsite $Pb_2(UO_2)(PO_4)_2 \cdot H_2O$

Phosphuranylite $Ca(UO_2)_4(PO_4)_2(OH)_4 \cdot 7H_2O$

Renardite $Pb(UO_2)_4(PO_4)_2(OH)_4 \cdot 7H_2O$

Sabugalite $HAl(UO_2)_4(PO_4) \cdot 16H_2O$

Saléeite $Mg(UO_2)_2(PO_4)_2 \cdot 8{-}10H_2O$

Torbernite $Cu(UO_2)_2(PO_4)_2 \cdot 10H_2O$

Uranospathite $Cu(UO_2)_2(PO_4,AsO_4)_2 \cdot 16H_2O$ (?)

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TABLE II. (cont.)

Vanadates

Carnotite	$K_2(UO_2)_2(VO_4)_2 \cdot 1-3H_2O$
Ferghanite	$(UO_2)_3(VO_4)_2 \cdot 6H_2O$ (?)
Francevillite	$(Ba,Pb)(UO_2)_2(VO_4)_2 \cdot 5H_2O$
Metatyuyamunite	$Ca(UO_2)_2(VO_4)_2 \cdot 3-5H_2O$
Rauvite	$CaO \cdot 2UO_3 \cdot 5V_2O_5 \cdot 16H_2O$
Sengierite	$Cu(UO_2)_2(VO_4)_2 \cdot 8-10H_2O$ (?)
Tyuyamunite	$Ca(UO_2)_2(VO_4)_2 \cdot 5-8H_2O$
Uvanite	$U_2V_6O_{21} \cdot 15H_2O$

Silicates

Cheralite	$(Th,Ca,Ce,U,Pb)(PO_4,SiO_4)$
Coffinite	$U(SiO_4)_{1-x}(OH)_{4x}$
Cuprosklodowskite	$Cu(UO_2)_2(SiO_3)_2(OH)_2 \cdot 5H_2O$
Gastunite	(Ca,Pb) uranyl silicate
Huttonite	$ThSiO_4$
Kasolite	$Pb(UO_2)(SiO_3)(OH)_2$
Pilbarite	$PbO \cdot ThO_2 \cdot UO_3 \cdot 2SiO_2 \cdot 4H_2O$
Sklodowskite	$Mg(UO_2)_2(SiO_3)_2(OH)_2 \cdot 6H_2O$
Soddyite	$(UO_2)_5(SiO_4)_2(OH)_2 \cdot 5H_2O$
Thorite	$(Th,U,Ce,Fe,etc.)(SiO_4)_{1-x}(OH)_{4x}$
Uranophane	$Ca(UO_2)_2(SiO_3)_2(OH)_2 \cdot 5H_2O$
Uranothorite	$(Th,U)SiO_4$
Weeksite	$K_2(Si_2O_5)(UO_2)_2 \cdot 4H_2O$

Niobates–Tantalates–Titanates (multiple oxides)

Betafite	$(U,Ca)(Nb,Ta,Ti)_3O_9 \cdot H_2O$
Brannerite	$(U,Ca,Fe,Th,Y)_3Ti_5O_6$
Davidite	$(Fe,Ce,La,Y,U,Ca,Zr,Th)(Ti,Fe,V,Cr)_3(O,OH)_7$
Delorenzite	$UO_2 \cdot Y_2O_3 \cdot 2FeO \cdot 24TiO_2$ (?)
Microlite	$(Na,Ca)_2(Nb,Ta,Ti)_2O_6(O,OH,F)_7$ Ta > Nb
Pyrochlore	$(Na,Ca)_2(Nb,Ta,Ti)_2(O,OH,F)_7$ Nb > Ta

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ferro-magnesium minerals, present to a greater or lesser extent in all ores, contribute the all-important ferric ions for the oxidation reaction.

"*Sulphides:* The behaviour of sulphides like the silicates cannot be explicitly predicted. However, in general the presence of sulphides suggests that more oxidants may be required and as a result also possible higher acid consumption. In addition, their presence may indicate that the ore may be subject to rapid weathering and bacterial leaching in stock-piles etc. In fact the presence of pyrite in low-grade ores should suggest that bacterial leaching be examined.

"*Carbonaceous constituents:* Graphite and related materials present in carbonaceous shales are generally unreactive but can lead to problems owing to the physical locking of the disseminated uranium mineral, or sometimes in the solid liquid separation processes."

Any information that the mineralogist can develop on the chemical composition, physical characteristics and variability of the feed materials will be an important contribution to the processing evaluation planning. Also, by mineralogical examination of tailings samples, reasons for unexplained low uranium extractions can often be determined. Close co-operation between the metallurgist and the mineralogist can significantly decrease the time required for process evaluation studies and also minimize the potential for unexpected surprises.

2.5. INSTRUMENTAL AND CHEMICAL ANALYSIS

It is assumed in this manual that operating analytical facilities are available for analysing the processing development samples. Therefore, details on analytical procedures and techniques will not be presented here. A few general comments, however, on analytical requirements and limitations are desirable.

Radiometric analyses: Radiometric analyses can be useful for screening head samples, but it should be remembered that uranium ore samples are not always in radiometric equilibrium. Special radiometric equipment such as beta/gamma systems may be required for reasonably accurate measurements. It should also be kept in mind that radiometric analyses are of little use for determining the uranium content of leach residues or other similar materials. Leaching with either sulphuric acid or carbonate solutions can extract the uranium but leaves nearly all of the radioactive daughter products in the residue. Therefore, the gamma and beta radioactivity of the tailings material may be nearly the same as that of the feed ore.

Chemical determinations on head samples: The following chemical analyses are desirable for nearly all head samples:

U_3O_8	Mo^{2+}	Cu^{2+}
CO_2	V_2O_5	Fe^{2+}
SO_4^{2-}	PO_4^{2-}	Fe^{3+}
S^{2-}	SiO_2	

Achieving the desired accuracy and reproducibility of uranium analyses often presents problems. Even with experienced chemists and careful procedures, the reproducibility of the uranium analyses will be in the range of 5–7%. With only slightly less careful work, the range can be considerably larger. The desirability of replicating both the experimental test work and the analytical measurements can be particularly important during the initial phases of the process development studies. The information developed can help to place the reliability of the test results and their variability in the proper perspective.

The type of analytical determination required for different uranium contents is always a question. A few laboratories have found that for materials containing less than 0.05% U_3O_8 the fluorimetric technique is often the most desirable. For materials containing in excess of 0.05% U_3O_8 colorimetric procedures have produced the best results.

3. SIZE REDUCTION

3.1. GRINDING TESTS

The purpose of grinding tests is to determine the optimum size of grind at which efficient leaching of the uranium may be achieved at an acceptable energy cost and result in a leached residue which may be thickened or filtered readily for recovery of the uranium bearing solution.

The feed for laboratory grinding tests can be a drill core or development ore crushed to −10 mesh (<1.651 mm). A standard laboratory mill, such as a 20 cm diameter by 17 cm length Paul Abbé ceramic unit, may be used.

A typical charge to the mill could be:

- Steel balls (19 mm diameter)   9 kg
- Ore (−10 mesh)   1000 g
- Water   500 mL

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Grinding times are determined by preliminary tests of, say, 20, 40 and 60 min. After grinding, each sample is filtered on a 10 cm Buchner funnel and approximately 200 g of wet cake are cut from each sample for screen analysis on a 200 mesh (74 μm) screen. The sample may be repulped and washed with water through the 200 mesh screen and then through a 325 mesh (40 μm) screen. The three fractions of +200 mesh, −200 + 325 mesh and −325 mesh are filtered, dried and weighed. The remaining filter cake can be used as leach feed for preliminary acid or carbonate extraction tests which will yield preliminary extraction efficiencies from varying grinds. Generally, increasingly fine grinds will yield higher uranium extractions until no further improvement is obtained because the uranium bearing minerals have been completely liberated. However, the finer grind must be evaluated against the increased energy cost, the possible increase in reagent consumption and the increased difficulty in recovery of the solution from the cake.

3.2. POWER CONSUMPTION

Power consumption in grinding can be predicted by an empirical formula known as the Bond work index. This work index is defined as the number of kilowatt-hours per short ton needed to reduce a material from theoretically infinite size so that 80% passes a 100 μm screen. In its practical application, the work index is a purely comparative figure obtained by grinding ores under controlled conditions in a standard laboratory mill designed by the Allis–Chalmers Manufacturing Company in the United States of America. The work index equation can be expressed as follows:

$$W = Wi \left(\frac{10}{\sqrt{P}} - \frac{10}{\sqrt{F}} \right)$$

where

W is the work required (kW·h/short ton),
Wi is the work index,
P is the screen size at which 80% of the product passes (μm),
F is the screen size at which 80% of the feed passes (μm).

Ore samples may be tested by laboratories equipped with the standard Allis–Chalmers mill but it is also possible to compare the grindability of an unknown ore if a standard reference ore sample of known grindability is available. Both samples are ground for the same time in the same mill and screened to determine the screen size at which 80% of the weight passes.

A typical calculation is given below:

Data from reference ore

$$Wi = 19.5, F = 1130, P = 133$$

Data from ore being tested

$$Wi = ?, F = 960, P = 123$$

Then

$$Wi \left(\frac{10}{\sqrt{123}} - \frac{10}{\sqrt{960}} \right) = 19.5 \left(\frac{10}{\sqrt{133}} - \frac{10}{\sqrt{1130}} \right)$$

Therefore, $Wi = 19.2$ kW·h/short ton.

The work then required to reduce an ore from, say, 80% passing 9500 μm (3/8 in) to 80% passing 105 μm will be:

$$W = 19.2 \left(\frac{10}{\sqrt{105}} - \frac{10}{\sqrt{9500}} \right)$$

$$= 16.78 \text{ kW·h/short ton}$$

If the ball mill must treat 200 short tons/d (8.3 tons/h) then the required mill power will be:

16.78 (kW·h/short ton) × 8.3 (short tons/h) × 1.34 (hp/kW) = 186 hp

3.3. SCREENING AND SIZING

Standardized testing sieves are needed to control and to check the efficiency of commercial sizing processes. The most generally accepted standard series, and the one used in mill testing worldwide, is the Tyler Standard Screen Scale. The Tyler Standard Screen Scale Testing Sieves have openings whose widths differ from the next in the series by a factor of $\sqrt{2}$ (1.414). This means that the area of each successive opening in the series is double that of the next finer or half that of the next coarser sieve. The Tyler Standard Screen Scale has 20 sieves with openings ranging from 0.0015 in (400 mesh) to 1.050 in, as shown in Table III.

There are also some intermediate sieves outside the standard series, such as the Tyler 325 mesh screen which has an opening 0.0017 in (40 μm) wide and is intermediate between the standard 270 mesh and 400 mesh screens. Microscreens with

TABLE III. TYLER STANDARD SCREEN SCALE

Mesh size	Aperture or width of opening		
	Nominal (in)	Actual (mm)	Actual (in)
	1		1.050
	0.75		0.742
	0.5		0.525
	0.375		0.371
3	0.25		0.263
4		4.699	0.185
6		3.327	0.131
8		2.362	0.093
10		1.651	0.065
14		1.168	0.046
20		0.833	0.0328
28		0.589	0.0232
35		0.417	0.0164
48		0.295	0.0116
65		0.208	0.0082
100		0.147	0.0058
150		0.104	0.0041
200		0.074	0.0029
270		0.056	0.0021
400		0.037	0.0015

openings of 0.010, 0.020 and 0.030 mm are available for extremely fine sizing determinations.

3.3.1. Preparations for screening

Screening tests can be run either wet or dry, or with a combination of both. Brass screens are commonly used for all normal testing, but stainless steel sieves

should be used for screening corrosive solids or pulps. The procedure that should be followed prior to screening is as follows:

(1)   The screens to be used should be gathered in one spot and checked to see that all the proper mesh sizes for the series are present and that they are all of the same standard series, e.g. the Tyler Standard series.

(2)   The screens should be checked for cleanness by holding them up to the light and making sure that no particles from previous screening operations are stuck in the openings. The screens should always be cleaned immediately after being used but this is sometimes overlooked. Stuck particles should be removed by carefully brushing the coarser screens with a soft brass wire brush. For the extremely fine screens, brushing can be done with a small hair brush. Cleaning is best done by brushing the underside of the wire cloth with a circular motion, taking care not to exert too much pressure against the wire cloth. Tapping the frame of the sieve gently with a wooden stick or against the bench is also helpful. Extreme care should be used in this procedure, as testing sieves are expensive.

(3)   All the screens should be checked to ascertain that there are no broken sections in the screen surface. In the case of screens of 65 mesh or finer this should be done with extreme care as it is very easy to overlook a small break. The simplest way of checking is to hold the screen up to a light source and scan the complete surface for any oversize openings or tears. A magnifying glass is helpful in this respect. The screens should then be checked by carefully examining the complete circle where the cloth is attached to the screen frame. This is one of the weakest spots on the finer screens. Any tears or oversize holes found should be repaired by maintenance personnel prior to use of the screen.

(4)   After cleaning and inspection the screens should be stacked for use in either a dry or a wet screening unit. Both units have a maximum capacity of six screens. If more screens are required, screening should be conducted in stages, starting with the coarsest size in the series. The screens should be stacked in the proper order with the coarsest screen at the top.

(5)   The sample to be screened should be truly representative of the mass of material for which the screen analysis is desired. In determining the size or weight of the sample to be taken, the type of material, its screenability and the range of particle sizes present must be considered. For example, in making a screen analysis of a material representing a feed to a screen or of a product from a crusher in which the particle range is very wide, a large sample of 500–1000 g may be required. At the other extreme, if the material to be tested is a very fine finished product, a sample of only 25–100 g may be required.

(6)   The sample weight should be limited so that no sieve in the series is over-loaded. Overloading is most likely to occur in making analyses of closely

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graded materials where the range of particle sizes is narrow. In this case, the size of the sample should be determined by the capacity, without overloading, of the sieve retaining the largest amount of the sample. Overloading results in unreliable data as blinding of the meshes occurs on a heavily loaded sieve. In addition, overloading of the finer screens will also stretch the cloth and, in severe cases, cause tearing where the cloth is attached to the frame of the sieve. If large amounts of material are to be screened, then the screening should be conducted in several smaller batches.

The general procedures for conducting standard screening tests are as follows.

3.3.2. Dry screening

The normal procedure is to use a screen shaker which gives a circular and tapping motion to the screens. The sample is normally screened for 15–20 min. Screening time depends upon the type of material being screened and upon the type of test desired. For plant control operations, a 3–5 min test on a pre-sieving material is sufficient to give the desired data. On more difficult materials, a sieving time of 15–30 min is justified. After the screening is complete, the material remaining on each screen surface is weighed and recorded against the respective mesh size. The combined weight of all the fractions should equal the initial starting weight of the sample to within a few grams per kilogram. If the difference is much greater than this, the source of error should be found and corrected, and the test repeated. When this is completed, all screens should again be very carefully checked for tears and other damage to ensure that the material held on any screen surface is of the proper specification. If tears or other damage to the screen is found, the test is not valid. The damaged screen should be repaired and the test rerun. The percentage size distribution of the material is calculated from the weights of the various screen fractions.

3.3.3. Wet screening

The dry screening of a material containing a large amount smaller than 100 mesh is generally unsatisfactory because the fine material blinds the fine screens in a few seconds. In addition, many materials would not give a proper screen analysis if dried prior to screening. For example, in pulps containing extremely fine slimes, these slimes would tend to agglomerate during drying, and if then screened dry would not give a true picture of the proper particle size distribution in the sample. In such cases, the sample should be wet screened or, if this by itself is not accurate enough, screened by a combination of wet and dry screening.

The sample is first weighed and then placed onto the finest sieve in the test series. For example, if a test of screen sizes of 65, 100, 150, 200 and finer than 200 mesh were being run, the sample would be placed on the 200 mesh sieve. This sieve is then placed in a spider which is mounted over a bucket. The sample on the screen is then washed with a small jet of water until the slimes have been almost completely washed through the screen. The appearance of the water on top of the material is a fair indication of the effectiveness of this washing. If it is clear, the sample has probably been washed enough. The sample is then washed into a pan and placed in a drying oven. If any cemented lumps are present these should be mashed with a rubber stopper, and the procedure is then followed for a dry screen test. If the −200 mesh material from the test is to be saved, the material that is washed into the bucket should be filtered, dried and combined with the dry material that is produced from the dry screening operation. The technique described is about the most accurate for screen testing where a combination of wet and dry methods is required.

After washing of the sample, the stack of up to six screens is inserted in the funnel shaped bottom of the wet screening unit, care being taken to ensure that the funnel is completely clean. A wet sample of the proper size is then carefully poured onto the top screen of the stack. The transfer container should also be washed out thoroughly to ensure that the complete sample has been transferred to the screening unit. The water distributing pan should then be inserted on top of the coarsest screen and the clamp-down unit fixed in place. A small rubber hose should be attached to the water distributor plate. The screening unit should be operated for an interval of a few seconds to about a minute to ensure that most of the liquid has passed through the stack of screens before a very light flow of wash water is applied to the water distributor plate. The liquid and fine material passing through the stack are caught in a bucket underneath the screening unit. If, after this startup practice, little or no material is found to enter the bucket, it generally indicates blinding of one of the screens. This blinding or damming action can generally be overcome by a little additional screening outside the stack before turning on the wash water or by gently tapping the sides of the screens in the stack until pulp flow is started through the screens. Once this flow has been started, a small stream of wash water can be allowed to flow through the stack and screening continued for the desired time.

As in dry screening, the time necessary for the screening is largely dependent upon the type of material being screened and upon the type of test desired. Screening should be continued sufficiently long so that the underflow from the stack of screens is not only completely clear of suspended material but also essentially free of any fine undersize. This can be checked by catching a small sample in a glass beaker and seeing whether it is free of cloudiness and undersize particles. When this point is reached, the flow of water can be stopped and the screening action discontinued. The stack of screens should then be removed from the screening unit and the material remaining on each screen carefully washed and filtered to dryness. These damp

filtered screen fractions are then dried. Each screen fraction should be marked as to size, preferably directly on the filter paper. The various sand fractions are very easy to filter and dry, and do not require any special handling. This is not normally the case with the undersize material.

If this undersize requires further screening it must be transferred to the next stack of screens in the series, the same general procedure being followed as before. The final undersize material from the last screen in the series will, for most samples, contain slimes which will make filtering extremely slow. Sometimes it is not economical to filter, dry and weigh this material. If this material is not to be used in any additional test work, a small amount of Separan can be added to this pulp to assist settling and filtration of this fraction of the sample. If the pulp then quickly settles and produces a perfectly clear effluent, this effluent can be decanted and discarded without further treatment. The remaining material, or the total material in a sample which does not settle to a clear effluent, should then be transferred to the laboratory bench filter and filtered to dryness, after which the sample is dried in an oven. With some materials it is better to dry the unfiltered pulp in a very large pan. When all of the individual size fractions have been dried, they are weighed and the net weight of material obtained in each fraction recorded. Again, as in the dry screen analysis, all screens should be checked for damage and, if any oversize holes or tears are found, these screen tests should be repeated. In the wet screen analysis, it may be difficult to check the final total dry weight of the various fractions against the original starting weight of the solids. If there is a wide divergence between the anticipated total weight and the actual total weight, the complete procedure should be reviewed and checked for a possible source of error.

A wet screening unit can also be used for combined wet and dry screening where the highest accuracy is not required. In such a case, the sample can be wet screened, as previously described, except that generally only one or two screens are necessary. The finer screen should be installed at the bottom of the stack and such other scalping screens as are desirable to alleviate the load on this screen can be placed above it. The wet screening procedure is the same as previously described, but it is not essential to screen to the same degree of completion, i.e. screening until a relatively clear underflow is obtained is normally sufficient. When this point is reached, the various sand fractions, i.e. oversize, on all screens can then be filtered together and the final undersize of the finest screen filtered and dried, as previously described. After drying, all sand fractions can be combined and then screened on a dry screening unit, it being made certain that the finest screen used during wet screening is included in the series. The undersize of the finest screen obtained from the dry screening is then added to the dried undersize obtained from the same screen during wet screening and the total weight reported as one weight for the sizing analysis. The other screen fractions are then weighed and the size distribution calculated from the obtained data.

TABLE IV. EXAMPLE OF SIZE AND URANIUM ANALYSIS OF FEED AND
LEACH TAILINGS

Mesh size	Feed		Leach tailings	
	wt%	wt% U_3O_8	wt%	wt% U_3O_8
+65	4.5	0.07	4.7	0.05
−45 + 100	9.2	0.10	9.5	0.08
−100 + 150	15.2	0.14	15.5	0.10
−150 + 200	14.7	0.17	15.1	0.10
−200	56.4	0.24	55.2	0.023

3.4. SIZE ANALYSIS

The purpose of size analysis is to determine the distribution of uranium in the
mill feed and in the leached tailings and hence the size fraction at which the maxi-
mum uranium extraction is obtained. For uranium leaching, the usual screen size
designation for a grind is the percentage −200 mesh (<74 μm) fraction. In some
ores, where the uranium mineralization is extremely fine, the size designation is
reduced to the percentage of −325 mesh (<40 μm) fraction.

For size analysis a representative sample of 200 g of dried ground ore is care-
fully taken from a 2000 g batch. The sample should be washed with water through
a −200 mesh screen and both fractions retained and dried. The +200 mesh fraction
is screened on a 65, 100 and 150 mesh stack of screens using at least a 10 min tapping
time. The new fractions are then weighed and analysed for U_3O_8 content. The
−200 mesh fraction is also weighed and analysed.

After leaching of the ore the same procedure of screening and analysis is
repeated for the size fractions. The leached tailings can then be compared with the
feed as shown in the example in Table IV. An examination of Table IV reveals that
much of the uranium is still locked in the coarser, +200 mesh fractions and that
further fine grinding would be beneficial. Consequently, further leach tests would
be carried out on finer ground material until no further significant uranium extraction
could be obtained.

If good uranium extraction is not obtained in the −200 mesh fraction, this frac-
tion may be further separated at 40, 20 and 10 μm by sub-sieve-size elutriation by
air with a Haultain Infrasizer or by water with a cyclosizer. Analysis of these frac-
tions for uranium content will then reveal at which size the uranium mineral has been
freed for leaching.

4. LEACHING

4.1. INTRODUCTION

Two main techniques are used for leaching uranium ores: acid leaching (using sulphuric acid) and alkaline leaching (using a mixture of sodium carbonate and bicarbonate). Alkaline leaching is used when the host rock contains significant amounts of acid-consuming components. The initial laboratory investigation should preferably cover both methods. In most cases it is possible to select one or the other after a few tests.

The first stage of any leaching test should include a mineralogical examination of a range of ore samples. Examination of each ore type will help in defining the variability that can be expected. Some information may already be available from identifications conducted during the phase of geological exploration. Even the simplest microscopic techniques can provide useful information, and techniques such as electron probe microanalysis, X ray diffraction and autoradiography and careful petrographic examinations can be used to identify all major mineral phases and often provide qualitative and quantitative compositions of minor and trace elements in particles down to a few micrometres in size [2, 7].

The results of these examinations will suggest suitable leaching processes and possible processing problems. For example, a high concentration of carbonate material could preclude the use of acid leaching, or the presence of refractory uranium minerals may require severe leaching conditions to achieve a satisfactory recovery of uranium. Determination of grain sizes will also be relevant to the degree of grinding required.

4.2. SAMPLE REQUIREMENTS

A complete experimental programme to evaluate leaching conditions will require 25–50 kg of ore. Individual experiments can be readily carried out using 500–1000 g samples. Such an amount provides a convenient volume of slurry for measurement of pH and oxidation potential and sampling during the experiment. There is no advantage in working on a larger scale as leaching is one operation for which direct scale-up from bench scale data is possible.

Difficulties in obtaining sufficient quantities of representative ore are often experienced in the early stages of a feasibility study. Consequently, the origin of all samples must be properly identified and a decision taken on whether a sample representative of the bulk of the ore body will be prepared or whether ores of specific types (primary, secondary, weathered, etc.) will be examined. Both point and bulk samples may be tested but the final process has to be based on the testing of bulk

samples. Care must be taken to ensure that representative samples are obtained during all stages of sampling and splitting.

4.3. LEACHING VARIABLES

The effects of the following variables must be assessed to determine optimum leaching conditions:

— Grind size
— Slurry density
— Degree of agitation
— Composition of leach liquor
— Acid concentration
— Oxidation potential
— Temperature
— Pressure (see Section 10.6)
— Leaching time.

In theory, a complete factorial study should be undertaken in which each variable is tested over a selected range. In practice, a partial factorial study with suitable levels of some variables can be chosen on the basis of previously reported mill practice. An estimate of the experimental error should also be made by conducting replicate experiments.

A brief guide to the selection of appropriate conditions is presented below. More detailed discussions are available in the literature [2, 3].

4.3.1. Grind size

Mineralogical examination should indicate the particle size required to provide satisfactory exposure of uranium mineralization to leach solution. In addition to increased grinding costs, overgrinding may result in high pulp viscosity which could adversely affect leaching rate and increase power requirements for agitation. A finer grind will also reduce filtration and settling rates in subsequent solid–liquid separation processes.

4.3.2. Slurry density

Optimum slurry density is usually the maximum possible without exceeding a viscosity which prevents satisfactory contact between the liquid and the surfaces of the solids. In some cases, the influence of slurry density on the power requirement for agitation may be a limiting factor. Operating with a high slurry density reduces the volume of the leaching circuit required to obtain a desired residence time and

reduces reagent consumptions required to achieve the desired reagent concentrations. However, slurry density is often limited by the physical properties, and sometimes by the mineralogy, of the ore. In practice, sizing operations in the grinding circuit or the underflow density obtained after thickening will determine slurry density. For ores with a high clay content, density may be restricted to 45 wt%. Normal slurry densities range from 50 to 65 wt%. In some cases, constituents dissolved from the ore may be of sufficient concentration at a high slurry density to lower the rate of uranium extraction or cause precipitation of uranium.

4.3.3. Degree of agitation

Agitation does not generally affect kinetics unless the ore is not properly suspended. Thus agitation must be sufficient to prevent settling out of coarse particles and to ensure satisfactory dispersion of reagents. In the laboratory, rapid mixing of acid and oxidant is necessary to achieve good control of leaching conditions, which is essential if the effects of small changes in conditions are to be determined. In general, the intensity of agitation (power per unit volume) will be greater than that in the plant. It is desirable to maintain the same geometrical equipment arrangement throughout the laboratory tests.

4.3.4. Acid concentration

Selection of the optimum acid concentration is one of the most important phases of laboratory leach tests. Acid consumption will also determine the lime requirement for neutralization (if needed) and affect oxidant usage. Unless very high free acid concentrations are required (> 10 g/L), suggesting that the gangue is relatively unreactive, experiments conducted under conditions of controlled pH are the most useful. Accurate control (to ± 0.1 pH unit or better) is essential as the reactivity of certain gangue minerals (e.g. chlorite) is extremely sensitive to acid concentration [8].

Control of acid concentration is of fundamental importance as it directly determines the rate of dissolution of uranium and most gangue minerals. In addition, the oxidation potential is pH dependent. This approach is preferred to the method of adding a range of selected acid quantities, for example 30, 50 and 70 kg/t ore, which results in a variable acid concentration throughout leaching. In the mill, variations in ore type will undoubtedly require a range of acid consumptions to achieve optimum extraction. The technique of adding a range of selected acid quantities can at times be useful for preliminary comparisons of samples from different parts of a deposit. This type of leach test on relatively small samples (150–200 g) can help in gaining some experience of the leaching variability that may be encountered.

In sulphate solution, the maximum dissolution rate of UO_2 occurs at pH2 [9]. For other uranium minerals, a lower pH is usually required to achieve a satisfactory

leaching rate. Depending on the type of uranium mineralization, batch laboratory experiments should be carried out over the pH range 1.0–2.2 with a pH difference of 0.2 between individual tests. For ores containing mainly uraninite, the optimum pH is typically in the range 1.5–2.0. A pH greater than 2.2 is not usually recommended as precipitation of ferric ion will result in increased oxidant demand. If a pH of less than 1 is required, addition of acid to control free acid concentration is more appropriate. Where a high residual free acid level is required, two stage leaching techniques or strong-acid pugging and curing may warrant consideration.

In mill leaching circuits, acid is often added to at least half (sometimes two thirds) of the leaching vessels to control pH or free acid, which is measured indirectly by means of conductivity. Batch laboratory tests at constant pH produce equivalent acid consumption and extraction profiles. Most of the acid is consumed during the initial phases of the leach.

4.3.5. Oxidation potential

Hexavalent uranium is readily soluble in sulphuric acid, but uranium in the tetravalent form must be oxidized for dissolution to occur. Oxidants act indirectly by converting ferrous sulphate, formed by the dissolution of iron minerals in the ore or iron introduced during grinding, into ferric sulphate. An Fe^{3+} concentration of about 1–2 g/L is usually considered adequate for effective dissolution of uraninite [10]. Ferric ion, in the form of ferric sulphate complexes, oxidizes tetravalent uranium by an electrochemical mechanism in which the concentration of Fe^{3+} ions adsorbed on the surface determines the rate of reaction [9]. The rate of dissolution is also affected by the presence of Fe^{2+} ions, but the form of the dependence is determined by the Fe^{3+}/Fe^{2+} ratio.

The ferric iron concentration in the leach liquor is controlled by adjusting the oxidation potential by addition of an oxidant. For typical leaching conditions, the relationship between oxidation potential and iron concentrations is given by the Nernst equation [11]:

$$E_c = 397 + 0.1984 \; T \; \lg([Fe^{3+}]/[Fe^{2+}])$$

where E_c is the solution potential (mV) relative to a saturated calomel electrode at 35°C, [ ] is the molar concentration and T is the temperature (K). The equation shows that at an oxidation potential of 400 mV about 50% of the iron is in the ferric state, while at 500 mV only 2% remains as ferrous ion.

The oxidation potential required to achieve complete oxidation is essentially unaffected by the type of ore (i.e. vein or sandstone) and gangue mineralogy. Consequently, a thorough investigation of the oxidation potential is not usually necessary. Satisfactory uranium extraction is obtained for most ores by maintaining an oxidation potential of 450 mV with respect to a saturated calomel electrode at 35–40°C. If the

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concentration of iron in solution is greater than 5–7 g/L after 1 h of leaching, a potential of 400 mV is adequate [12]. Leaching at potentials greater than 450 mV often increases the initial rate of extraction but the overall rate and ultimate recovery are not improved. Unnecessarily high potentials must also be avoided to minimize acid consumption by the oxidation of ferrous ion. For ores containing brannerite, recovery and extraction rates are increased significantly by leaching under more strongly oxidizing (600–650 mV) conditions [13].

The logarithmic relationship of the Nernst equation has important implications for the control of oxidation potential in both the laboratory and the mill. A variation of ±20 mV at 500 mV will not significantly affect oxidant consumption or uranium extraction, but the same fluctuation could not be tolerated at 400 mV.

Laboratory experiments should be carried out at constant oxidation potentials (±3 mV) of 425, 450 and 475 mV for the duration of leaching. For some ores, a delay in oxidant addition of 30 min after the start of leaching may be warranted to allow for completion of initial acid reactions with sulphides which would consume oxidant needlessly. From the results obtained a suitable oxidation potential can be selected for all further test work. However, it is not always necessary to maintain a constant potential throughout leaching. Typically, 425–450 mV is adequate for the first half of leaching provided that the potential remains above 400–410 mV at the completion of leaching. Additional experiments to determine a suitable oxidation potential profile are warranted as mill circuits are normally operated with oxidant addition to about half of the leaching vessels.

Saturated calomel reference electrodes are commonly used in laboratory test work whereas saturated silver chloride electrodes are almost exclusively used in the mill because of easier maintenance. At 40°C, an oxidation potential of 450 mV relative to a saturated calomel electrode is equivalent to 503 mV relative to a saturated silver chloride electrode. At 50°C, the difference is 57 mV.

Figure 5 shows the effect of oxidation potential on leaching characteristics.

4.3.6. Temperature

The effects of temperature, acid concentration and leaching time are interdependent and may need to be considered collectively. Increasing the temperature will cause an increase in the rate of leaching of uranium. However, for many ores the dissolution of gangue minerals will also be enhanced, with a consequent increase in acid consumption and a higher concentration of impurities in solution. The minimum leaching temperature for an acceptable extraction rate and ultimate recovery is typically 30–35°C.

Depending on the location of the deposit, initial laboratory experiments should be carried out at the ambient temperature of the mill site, with allowances being made for the heat input during grinding and the heat of dilution of sulphuric acid. The minimum leaching temperature could be 5–25°C above ambient conditions. In

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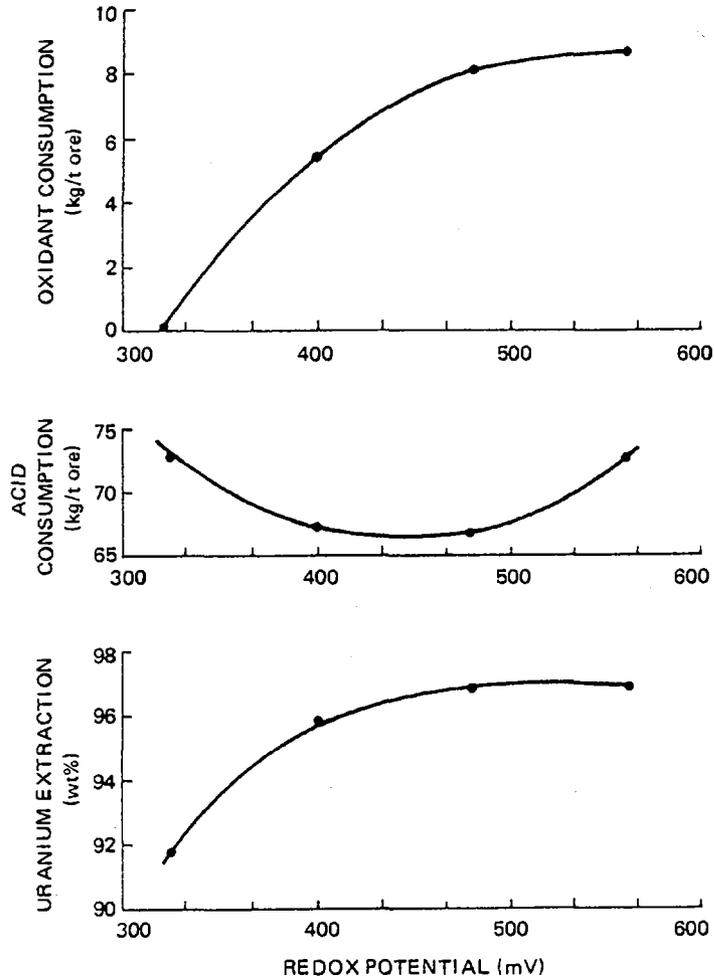
FIG. 5. *Effect of oxidation potential on leaching characteristics (pH1.5, 40°C).*

the laboratory external heating will almost certainly be required. In any case, good control of temperature (±0.5°C) must be provided to ensure equivalent leaching conditions in all test work. In particular, pH measurements are very sensitive to changes in solution temperature.

Figure 6 shows the effect of temperature on leaching characteristics.

4.3.7. Leaching time

For most ores, reagent costs are minimized by leaching under mild conditions for an extended period (10–30 h). The additional capital cost of doubling the capacity
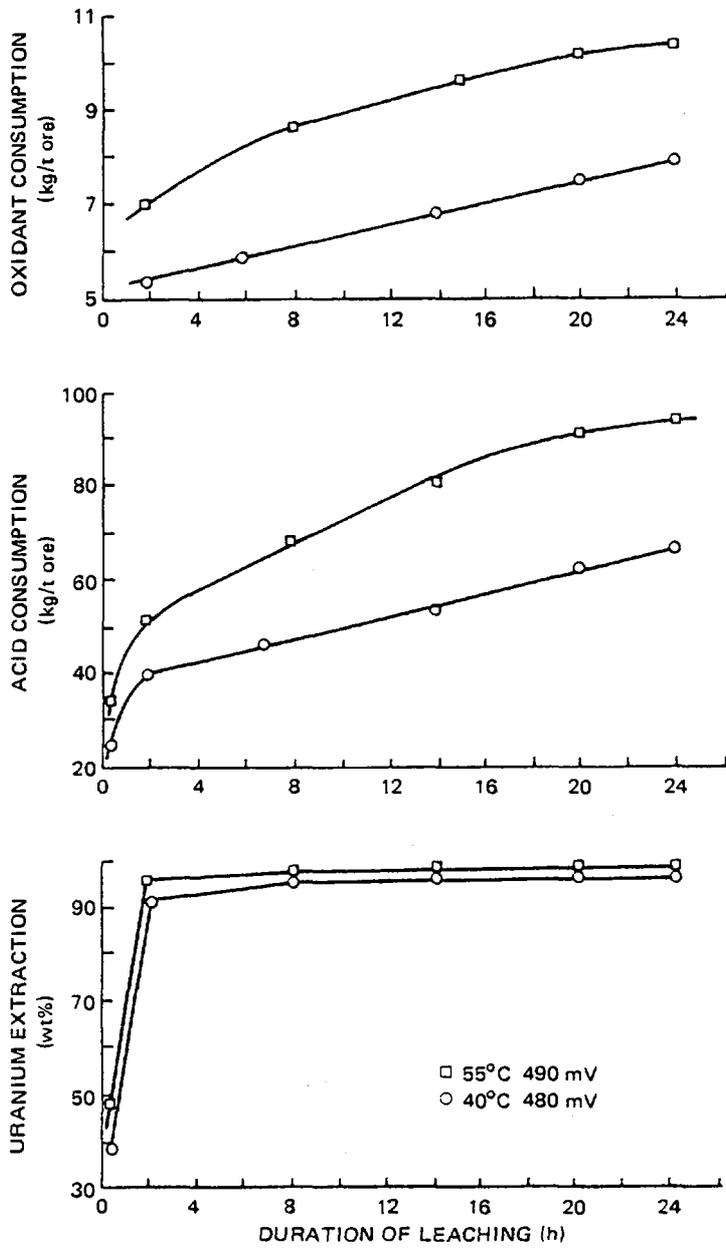
FIG. 6. *Effect of temperature on leaching characteristics (pH1.5).*

of the leaching circuit, for example, would be more than offset by a 25% saving in reagent costs over the life of a mill. For a given set of conditions, the optimum leaching time is determined by the cost of reagents required to maintain desired leaching conditions as compared with the value of the additional uranium recovered.

In the final analysis, a fine balance between leaching temperature, acid concentration and leaching time will determine the most suitable conditions. For most ores several sets of conditions can be chosen which would recover 90–95% of the uranium for the same cost. The selection of optimum conditions is often based on extraction of the remaining 3–5% of recoverable uranium.

4.4. EXPERIMENTAL TECHNIQUES FOR ACID LEACH TESTS

Laboratory test work is usually confined to batch leaching experiments. Although batch tests do not properly simulate conditions in the mill, they are the most practical means of determining preliminary extraction data and reagent consumptions. Comparative laboratory studies have shown that leaching data obtained in continuous test work are in very good agreement with batch results [14]. Under some circumstances continuous test work under optimum conditions determined from batch experiments may be warranted. In particular, the effects of the different concentration profiles of ions in solution (continuously variable in batch testing, but constant in each leach vessel) may be important. A typical batch test procedure is outlined below.

Figure 7 shows the type of laboratory leaching apparatus often used for constant pH leach tests. A 1000 mL baffled glass beaker is a suitable leach vessel for 500 g ore samples and a 2000 mL beaker is normally adequate for 1000 g samples. Some researchers prefer 1000 g test samples and others have found that tests using 500 g of feed material can produce fully satisfactory results which are readily duplicated. In general if the ore supply is adequate the 1000 g tests are preferable until some experience is gained. A variable speed electric agitator unit (0.1 hp)[5] is used to drive the impeller. Impeller diameters of 25–35 mm are normally used. The pH is measured by a combination electrode, and a platinum–calomel electrode configuration is used to determine the oxidation potential.

Combination pH electrodes should be carefully calibrated before each test in an appropriate buffer (pH1.7–2.0) at the leaching temperature. Any electrodes which have a slow response or show any signs of non-ideal behaviour should not be used. During leach tests, pH electrodes must be recalibrated every 4–6 h. With experience and proper experimental technique, a measurement accuracy (relative to buffer) of 0.02–0.03 can be achieved for a 24 h period. Reference electrodes used for oxidation

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[5] 1 hp = 745.7 W.
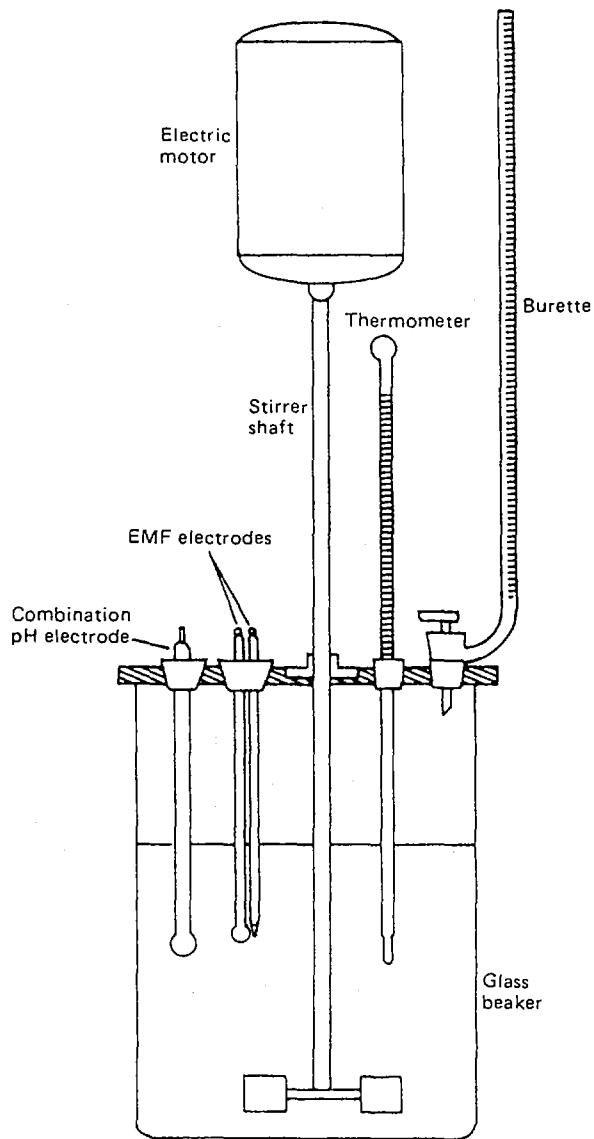
FIG. 7. Apparatus for acid leach tests.

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potential measurement are more reliable, but should be checked after every three or four leach tests.[6]

Temperatures can be measured with a standard thermometer, and various types of heating unit such as hot plates, water baths and heating mantles have been successfully used.

For a 1000 g test at 50 wt% solids, 1000 mL of water (preferably demineralized) are placed in the 2000 mL leaching vessel and the stirrer started. The ore is added and the slurry brought to the desired temperature. Temperature control to within 0.5°C is desirable.

The pH is adjusted to the target level by adding 1:1 sulphuric acid. Normally the leaching time is considered to have started when the desired pH is first reached. The pH is monitored continuously and controlled at the desired level by adding more acid. If an automatic titrator is used, the pH can be controlled to within approximately 0.02 units.

The oxidation potential is controlled by adding an oxidant such as $NaClO_3$ or MnO_2. It is often desirable to delay the first addition of oxidant for about 30 min after the start of the leaching period to allow for the initial acid reactions with any sulphides present. The $NaClO_3$ can be conveniently added as a 200 g/L solution.

The progress of the leach is followed by withdrawing 20–40 mL slurry samples at appropriate intervals. Sampling times of 0.5, 2, 6, 12, 18 and 24 h are suitable for a 24 h experiment. Immediately after each sample is taken, it is filtered and washed with five 20–40 mL volumes of acidified (approximately pH2.0) water. If the cake cracks during any given wash, the cracks should be closed with a small spatula before the next wash is added. The washed solids are dried at 105°C. During the initial tests, leaching efficiencies can be determined by assaying the dried solids for uranium and applying a small correction factor to account for ore (5–10 wt%) that dissolves during the leaching. For more definitive results, all products should be saved, measured and analysed so that a complete material balance can be made for each test. To obtain realistic results, the accuracy of the analytical method used should be ±5 wt%.

The free acid concentration and the iron concentration in the leaching solution can often be important variables. It is therefore desirable to determine the ferric and ferrous ion concentrations and the free acid concentrations[7] during at least the ini-

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[6] Performance of the Pt–calomel electrodes can be checked by immersing them in a pH4.00 buffer to which a small amount of quinhydrone has been added. At 25°C, the reading should be 218.0 mV.

[7] *Free acid determination:* Pipette 5.0 mL of undiluted filtrate into a beaker equipped with a magnetic stirrer. Add 20 mL of complexing solution made from 5 g of $K_4Fe(CN)_6$ dissolved in 100 mL of a saturated solution of KNO_3. Add 50 mL of methanol and titrate with 0.1M NaOH solution to pH5.0. Use a glass electrode for pH control. This procedure has been used successfully by a number of laboratories. Other techniques may also be applicable.

tial leaching studies. These determinations can be made on the undiluted filtrates (i.e. before washing) from the individual samples taken at different times. During both the initial tests and the optimization experiments, the final slurry should also be filtered and the undiluted filtrate analysed for U, V, Cu, Fe^{3+}, Fe^{2+}, Mo, SO_4^{2-}, Cl^-, free acid and total dissolved solids. The last quantity is determined by evaporating a sample of the filtrate at 105°C for 20 h. These determinations are required for planning subsequent solvent extraction or ion exchange investigations. Other desirable determinations include those for Al, Ca, F, K, Mg, Mn, Na, P and Zn.

It is often desirable to run several leach tests in parallel. When multiple leaching units are used, the equipment can be managed so that one set of pH and EMF electrodes can be used in succession for several leaching units. Moving the electrodes from one unit to another may require additional standardization to ensure accurate measurements.

4.5. ALKALINE LEACHING

Alkaline leaching of uranium ores is a viable alternative to acid leaching if:

(a) The ore contains significant amounts of acid consumers such as carbonates and chlorides,

(b) The uranium mineralization is in the hexavalent form or if it can be readily oxidized.

The simplified reactions are as follows:

Oxidation:

$$2UO_2 + O_2 \longrightarrow 2UO_3$$

Dissolution:

$$2UO_3 + 2Na_2CO_3 + 4NaHCO_3 \longrightarrow 2Na_4UO_2(CO_3)_3 + 2H_2O$$
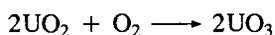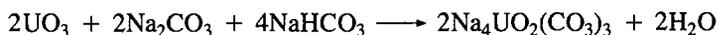
Sulphides react with sodium carbonate to form sodium sulphate as follows:

$$4FeS_2 + 15O_2 + 12Na_2CO_3 + 4H_2O$$
$$\longrightarrow 2Fe_2O_3 + 8Na_2SO_4 + 8NaHCO_3 + 4CO_2$$
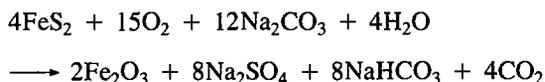
The efficiency of carbonate leaching depends upon the same factors as acid leaching (Section 4.3) except, of course, that a mixture of sodium carbonate and bicarbonate is used instead of acid. However, the leaching parameters are somewhat different, as detailed below.

Fineness of grind: Alkaline leaching requires a greater exposure of the uranium mineral for effective leaching. Consequently, a much finer grind is usually required and could be as fine as 75% −200 mesh.

Temperature: Higher leaching temperatures are required for carbonate leaching, usually from 70 to 80°C.

Retention time: Retention times are usually from 48 to 96 h.

Concentration of carbonate and bicarbonate: Concentrations of Na_2CO_3 of 35 g/L and of $NaHCO_3$ of 15 g/L are usually required for uranium leaching.

Oxidant requirement: The oxidant requirement depends upon the nature of the ore. The following values are typical: O_2: 25 cm^3/min; or air: 200 cm^3/min + 1% CO_2 (2 cm^3/min); or $KMnO_4$: 2–4 g/1000 g ore.

4.6. PRELIMINARY TESTING

A preliminary test for determining the amenability of ore to alkaline leaching may be done on as little as 100 g of sample. This test also determines the oxidation state of the uranium in the ore sample since hexavalent uranium is readily soluble in a sodium carbonate solution.

A 100 g sample of ground ore (at least 50% −200 mesh (< 74 μm)) is slurried with 200 mL of a 10% sodium carbonate solution and heated under reflux condenser at over 90°C for 30 min. The pulp is filtered and then washed with 100 mL of a 5% sodium carbonate solution and the combined solution is analysed for U_3O_8.

4.7. ALKALINE TEST PROCEDURE

The equipment used for alkaline leach tests is as follows (Fig. 8):

— 2 L Pyrex leach vessel;
— Wrap-around heating mantle (1500–2000 W) with rheostat for temperature control;
— Heated Pyrex vessel cover with top ports for stirring mechanism, reflux condenser, thermometer and gas entry;
— Variable speed mixer motor (400–600 rev./min);
— 2.5 in marine impeller and shaft.

The test procedure is as follows:

(1) Grind 1200 g of −10 mesh (< 1.651 mm) ore in a ball mill to 75% −200 mesh.
(2) Save 200 g of ore for head sample and screen analysis.
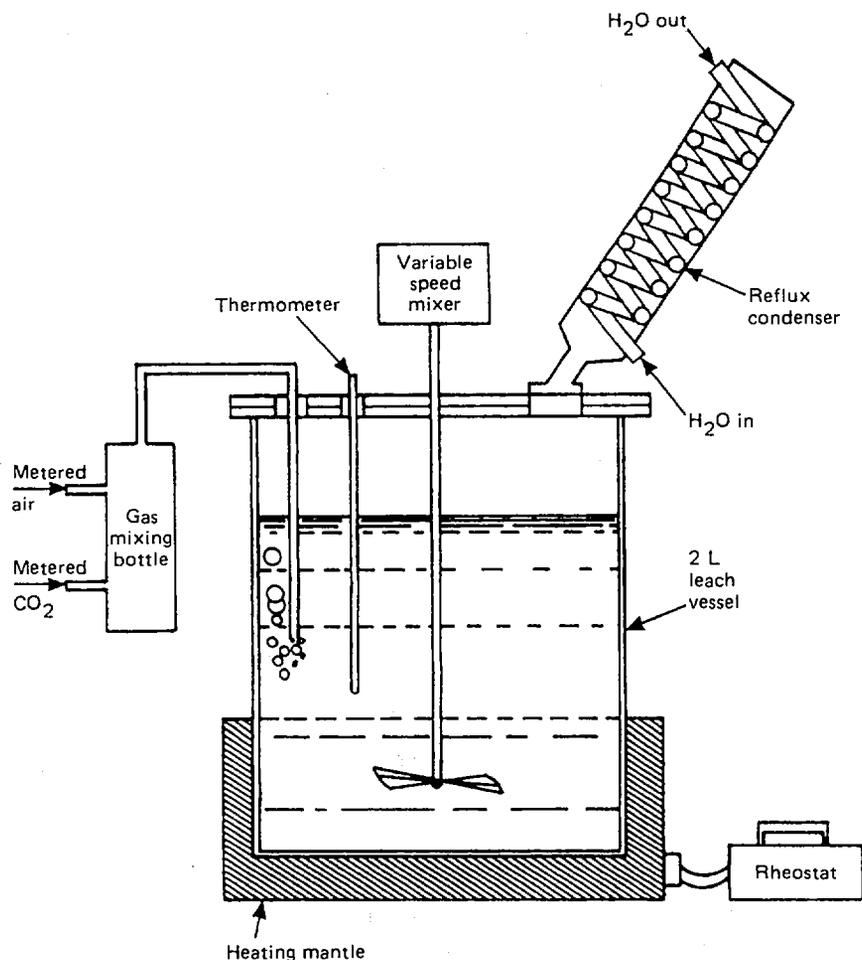
FIG. 8. Apparatus for alkaline leach tests.

(3) Repulp 1000 g of ore with 1 L of carbonate solution (Na_2CO_3: 35 g/L; $NaHCO_3$: 15 g/L) in 2 L leach vessel.

(4) Clamp cover over vessel with mixer, thermometer and gas inlet.

(5) Install reflux condenser.

(6) Place vessel in heating mantle and heat to 75°C.

(7) Sample at 24, 48, 72 and 96 h. Remove 25 cm^3 sample of pulp by pipette and filter and wash on 5 cm diameter Buchner funnel. Wash with 25 mL of 5% Na_2CO_3 solution and finally with 25 mL of water. Dry and analyse solid sample for U_3O_8.

(8) Once leaching has been completed, filter the remaining leach residue on a 25 cm diameter Buchner funnel and retain the leach liquor. Wash the residue with 1 L of 5% Na_2CO_3 solution and then with 500 mL of water. Retain all solutions and solids for U_3O_8 analysis.

(9) Draw up the metallurgical balance showing the distribution of uranium in the solution and in the residue and compare the calculated uranium content with the head analysis.

(10) Screen the residue and analyse the fractions +65 mesh (≥ 208 μm), $-65 + 100$ mesh, $-100 + 150$ mesh, $-150 + 200$ mesh, $-200 + 325$ mesh and -325 mesh to determine the recovery in each fraction.

Refractory ores containing mainly tetravalent uranium will not solubilize readily in a sodium carbonate solution. If the calculated uranium extraction by the preliminary test is not more than 30% then further alkaline leach tests are probably warranted. It must be noted that sometimes a uranium ore sample left for a long period after grinding will oxidize and alkaline leach tests will give an erroneous result as compared with that for a freshly obtained ore sample.

5. SOLID–LIQUID SEPARATION AND WASHING

Once leaching has been completed it is necessary to separate the pregnant liquor from the solid residues and to wash these residues to recover the remaining dissolved uranium. Washing must be very complete to prevent uranium losses.

Several types of equipment have been used for solid–liquid separation: conventional and high rate thickeners, filters, classifiers, cyclones and various combinations of these. Counter-current decantation (CCD) circuits with thickeners and multistage filtration with drum filters have been the two systems most widely used.

The efficiency of solid–liquid separation and washing and the type and size of equipment needed depend markedly on the nature of the ore. Metallurgical test work is therefore mandatory to provide a basis for the selection of equipment and for the estimation of capital and operating costs.

The solid–liquid separation section is usually the most expensive or the second most expensive part of the ore processing plant. The proper choice of type and size of equipment is therefore of prime importance for a successful operation.

Solid–liquid separation and washing are also performed after precipitation of a uranium concentrate. Although the size of equipment and process parameters are different, the basic principles are the same and the test procedures described in this section may be used with minor modifications.

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5.1. THICKENING

5.1.1. Conventional thickeners

Pulp thickening is used to eliminate water from a slurry or to recover the uranium bearing solution from a leached pulp. Conventional thickeners are equipped with rakes in the bottom of settling tanks to rake the settled solids to the tank centre for discharge from the tank bottom. The most important factors influencing the settling rate of a slurry are:

— The initial feed dilution
— The size and shape of the particles
— The specific gravity of the solids
— The specific gravity of the liquid
— The viscosity of the liquid
— The temperature of the liquid
— The type and quantity of flocculants added.

The thickener area required depends on the free settling rate of the solids, the initial liquid to solid weight ratio of the feed and the final liquid to solid weight ratio of the discharge. The depth of the thickener is dependent on the total residence time required to achieve the desired pulp density in compression with a minimum free settling depth and a maximum depth in compression based on empirical engineering data.

Laboratory tests for thickener sizing can be done with a 1 L graduated cylinder and by applying the Coe and Clevenger equation [15]:

$$A = \frac{1.33 \ (F - D)}{RS}$$

where

A is the unit area (ft^2) per ton of dry solids per 24 h,[8]
R is the free settling rate (ft/h),
S is the specific gravity of the liquid,
F is the initial liquid to solid weight ratio,
D is the final liquid to solid weight ratio.

The 1 L cylinder is filled with pulp at the initial density expected (F is, say, 3 parts liquid to 1 part solid). The free settling rate R is observed by measuring the drop in the settling pulp at short intervals (say every 5 min). Let R be 2 ft/h in this

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[8] 1 ft^2 = 0.0929 m^2.

example. The pulp is allowed to settle in compression until the final density is reached (D is, say, 1 part liquid to 2 parts solid). Therefore,

$$A = \frac{1.33\ (3\ -\ 0.5)}{2\ \times\ 1} = 1.663 \text{ ft}^2 \text{ per ton of dry solids per 24 h}$$

If the tonnage expected is 500 tons per day then the total thickener area required is:

$$\frac{500}{24} \times 1.663 = 34.6 \text{ ft}^2$$

This test is repeated at various increments of F (the initial liquid to solid weight ratio) to simulate compression area requirements, and the final maximum area A is used for design purposes.

Thickener depths are based on the total retention time required to reach final density but a minimum of 6 ft of free settling depth is required for slow settling solids and 1–2 ft for fast settling solids. As in all engineering design, a safety factor of above 50% should be added to allow for variations in the ore characteristics as mining progresses.

Uranium bearing ores often require flocculants to increase the settling rates. The most commonly used are high density non-ionic polyacrylamides usually added as a 0.1% solution in water at rates of from 0.01 to 0.1 lb of flocculant per ton of ore.

5.1.2. High rate thickeners

High rate thickeners have recently been developed particularly for fine, slow settling ores. Some have inclined plates which allow solids to settle earlier and slide down the plates to the thickener discharge area. In others the feed enters near the bottom or middle of the thickener tank and the flocculant is dispersed through a series of launders spread evenly over the pulp surface. Testing of these units is best carried out by the manufacturers since they have the latest designed units and the techniques required to test the ore.

5.2. FILTRATION

5.2.1. Introduction

The two most used types of filter are discussed here: rotary drum vacuum filters and horizontal belt vacuum filters. In applications where good washing is

essential horizontal belt filters are better than drum filters. When washing is not required (e.g. in filtration of neutralized effluents), drum filters may be a better choice because of their relative simplicity and compact design. It should be noted that drum filters may differ significantly in the type of device for cake discharge. Two types are used, knife discharge and filter belt discharge. The first type uses little or no cloth wash water. Belt discharge, however, requires good washing because of sticky filter cakes. This wash water consumption has great influence on the overall water balance.

Filtration is a complex operation and its effectiveness depends on many parameters. The following section describes the basic parameters which should be evaluated during testing. The description of the test procedures is given in Section 5.2.3.

5.2.2. Filtration parameters

The basic filtration parameters to be considered are:

(a) Type and amount of flocculant
(b) Cake formation rate
(c) Cake moisture content
(d) Cake washing rate
(e) Cake thickness
(f) Wash solution consumption and washing efficiency
(g) Type of filter cloth.

Other parameters, such as air flow through the filter cake and the vacuum applied, may also be important. These parameters usually are fixed during testing and are based on industrial experience and/or equipment performance. The granulometric composition of the solids to be filtered, the composition and properties of the liquor and the temperature of the slurry significantly influence filtration and should be considered in the overall evaluation of the process. The same is true of parameters such as the amount of filtrate and the properties of the cake, e.g. its thixotropic behaviour and its tendency to adhere to the filter cloth.

5.2.2.1. Type and amount of flocculant

Flocculants are macromolecular compounds which in the solution combine small solid particles to be filtered into bigger aggregates, or flocs. These flocs form more porous filter cakes, thus allowing faster filtration and washing and consequently reducing the size of equipment needed.

Flocculants are usually polysaccharides and polyacrylamides with or without ionic character. Other products such as polyethylene oxides are also used. Flocculants may be of natural or synthetic origin. They are effective in small additions

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of the order of 100 ppm with respect to the quantity of slurry. They are, however, expensive and their proper choice is important.

The type of flocculant to be used may be chosen partly on the basis of slurry composition but usually different products have to be tested in the preliminary screening test. The decision of which flocculant to use is based on economic considerations.

5.2.2.2. Cake formation rate

The cake is formed by solid particles collecting on the filter cloth through the action of the liquid flow against and through the filter cake itself and the filter cloth. The driving force is the pressure difference between the two sides of the cloth (e.g. under vacuum). The particles may be picked up from the filter boot in the case of a rotary drum filter; the cake is then formed against the force of gravity which tends to discharge the collected solids. In horizontal belt filters the force of gravity helps in the formation of the cake. In drum filtration the fines collect at the surface of the slurry in the filter boot and are picked up first. In this way the cloth becomes clogged. This problem may be overcome by not starting the vacuum pick-up before the bottom dead position is reached; thereby some of the filter cycle time is lost. In belt filtration the segregation of bigger particles first is desirable because these particles then form a drainage layer which protects the cloth from being clogged and facilitates later cake discharge.

The time for cake formation is measured from the moment when the vacuum is applied on the immersed filter leaf or on the filled Buchner funnel to the time of disappearance of the liquid from the surface of the cake. From the amount of dry solids picked up or filtered in the measured time the cake formation rate can be calculated; this is expressed as the weight of dry solids formed in unit time per unit area.

5.2.2.3. Cake moisture content

The water content in the wet cake expressed in weight per cent is the cake moisture content. The amount of wash solution needed for adequate washing is proportional to the moisture content. A distinction has to be made between capillary moisture and water that is chemically bound with the solids, e.g. in hydrated calcium sulphate, and which is also measured when the cake is dried at the usual temperature (105°C).

5.2.2.4. Cake washing rate

The cake washing rate is the rate with which the wash liquor passes through the formed cake and is expressed as the volume or weight of wash solution per unit area of cake and per unit time of washing. Washing may follow the formation of the

cake immediately after disappearance of the mother liquor from the surface of the cake or after a short period (a few seconds) for the 'first dry' which simulates the operation of the drum filter. From data for the washing rate and the amount of wash water required for adequate washing, the washing time can be calculated.

The cycle time, which is the time for one revolution of the filter drum or the time in which the belt moves from the feed to the cake discharge, is the sum of the time required for the formation of the cake, the drying time after formation of the cake (if required), the washing time, the final drying time and some additional time required for operation of the filter (time for washing the filter cloth, time for drum to reach the bottom dead position, etc.).

5.2.2.5. Cake thickness

In industrial practice the thickness of the cake is usually 10–15 mm. A cake of suitable thickness will discharge by gravity, leaving only traces of solids on the cloth. To initiate cake discharge the filters are provided with devices to blow off the cake (drum filters) or with cracking rolls (belt filters) which allow bending of the cloth and cracking of the cake, which then discharges by gravity. Different types of knife are applied to facilitate the discharge.

Thin cakes are more difficult to discharge, but the capacity of the filter is bigger when operating with thinner cakes. Consequently, during tests an attempt is normally made to form the thinnest cake which discharges without difficulty. Normally some millimetres of thickness are added as a safety margin. In this way during the operation of the filter there will be some allowance for a quicker run of the filter with higher capacity and with thinner cake.

5.2.2.6. Wash solution consumption and washing efficiency

The wash solution which is sucked through the cake displaces the mother liquor in the cake and washes the dissolved uranium out from the cake. The displacement is not ideal and a quantity of wash solution has to be used which is several times greater than the amount of mother liquor retained by the filter cake (moisture content). In spite of this it is not possible to wash out 100% of the dissolved uranium. In the series of filtration tests a determination should be made of how much of the uranium dissolved during leaching is washed out using different amounts of wash solution. The wash solution requirements are normally expressed as the number of cake moisture displacements or as the consumption of wash solution in tons per ton of dry solids in the washed cake.

Washing efficiency is expressed as the fraction of the uranium dissolved during leaching which is collected in the filtrate after filtration and after the washing of the cake. Typical losses of dissolved uranium because of incomplete washing are only some tenths of a per cent and typical consumption of wash solution is about 1 ton

per ton of dry solids. Typical moisture contents being about 20 wt%, it is evident that for good washing the moisture of the cake has to be displaced several times (usually two to four times).

5.2.2.7. Type of filter cloth

The filter cloth should retain essentially all the solids and yield filtrates as clear as possible. However, it should not exert significant resistance to the passage of liquid as compared with the filter cake itself. Fine particles in the slurry may clog fine filter cloths or filter papers and cause poor filtration rates. It is recommended, therefore, that industrial filter cloths be used for laboratory testing too. Coarse cloths may give cloudy filtrates which may have to be clarified separately for further use but this is normal procedure in industrial practice.

The cost of filter cloth may contribute significantly to operating costs. For this reason proper selection is essential. Adequate testing of the cloth requires long term tests in the laboratory or at the pilot scale. To prevent uncontrolled influence of the filter cloth on the filter test results it is recommended that monofilament industrial cloths be used for testing and be washed periodically.

5.2.3. Filtration test procedures

5.2.3.1. Slurry samples

The amount of slurry required for filtration tests is different from that for drum and belt filtration simulation. For drum filtration tests the filter leaf is completely immersed in the slurry, so some 2 L are required as a minimum for each series of tests (e.g. for each flocculant dose). It should be noted that sometimes the density of the slurry tends to increase because the filter leaf removes more liquid than solids from the slurry; this may completely change the results. In simulating belt filtration all the slurry is filtered, thus the quantity required for each test is smaller. Systematic tests require at least 10 kg of the ore sample leached under optimal conditions, but preliminary tests may be performed with a few kilograms of ore.

Filtration tests should be performed only on freshly leached slurries. Ageing (say overnight) even without heating and agitation may significantly change the properties of the slurry, especially with respect to granulometric composition. Old slurries may be used in preliminary tests if enough fresh ore sample is not available, e.g. for flocculation tests, rough estimation of flocculant consumption and filter cloth selection.

When fresh slurries are leached for filtration tests the ore should preferably be leached in a number of small portions, rather than being leached in a big quantity and the samples split before filtration. In this way, serious problems are overcome

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which may occur if segregation of bigger or heavier particles takes place in the slurry. If thief samples have to be taken, very good agitation during sampling or sample splitting must be ensured.

The samples should have the same temperature as that expected on the basis of leaching conditions.

5.2.3.2. Apparatus

The apparatus for filtration tests is basically the same for drum and belt filter simulation, though there are some differences and these will be specified later. The apparatus is shown in Figs 9 and 10. Its essential parts are:

— Safety flask (1 L) to collect all overflows which may occur (either oil from the vacuum pump or the filtrate) and to hold the vacuum;
— Air bleed valve for vacuum regulation;
— Vacuum gauge, preferably separated from the system by a stopcock, and having a range of ± 1 bar (10^5 Pa) with respect to ambient pressure;
— Rotameter for air, with a range of 1–10 $m^3 \cdot h^{-1} \cdot m^{-2}$ at ambient pressure;
— Receptacle for the filtrate, with a volume of 500 mL;
— Buchner funnel for belt filter simulation, with an area of about 1 dm^2;
— Filter leaf (Fig. 10) for drum filter simulation, with an area of about 1 dm^2;
— Slurry beaker with hand agitator, with a volume of about 2 L;
— Graduated beaker for the wash solution;
— Rubber pipes for flexible connections of all parts;
— Stop-watch.

All glassware and pipes must be able to withstand the vacuum.

5.2.3.3. Flocculation tests

Stock solutions (100 mL each) of different flocculant samples should be prepared using magnetic stirrers. The concentrations as recommended by producers should be used. Normally the stock solutions contain 3–10 g of dissolved flocculant. Flocculant should be added slowly (grain by grain) into the agitated water and agitation continued for 6 h until a homogeneous solution is obtained. Stock solutions should preferably be prepared a day before use. Stock solutions which are very viscous may be stored for longer periods (weeks) without deterioration of their properties. However, some natural products tend to alter quickly, especially in hot climates. The manufacturers' instructions should be followed. Just before use the flocculant should be diluted to the final concentration, which is usually 1 g/L.

For a flocculation test small increments of flocculant solution are added slowly to the slurry with gentle agitation until visible flocs are formed in clear liquor. All the slurry should be homogeneously mixed with the flocculant. The point of optimal

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FIG. 9. Apparatus for filtration tests.

Flexible connection

Filter leaf

Metallic ring

Cloth

Drainage groove

Supporting rib

Metallic ring

Filtrate outlet

Bolt

Cloth

(a)

(b)

Slurry

Cake

Wash
solution

Blow

Cake formation   First dry   Washing   Drying   Discharge
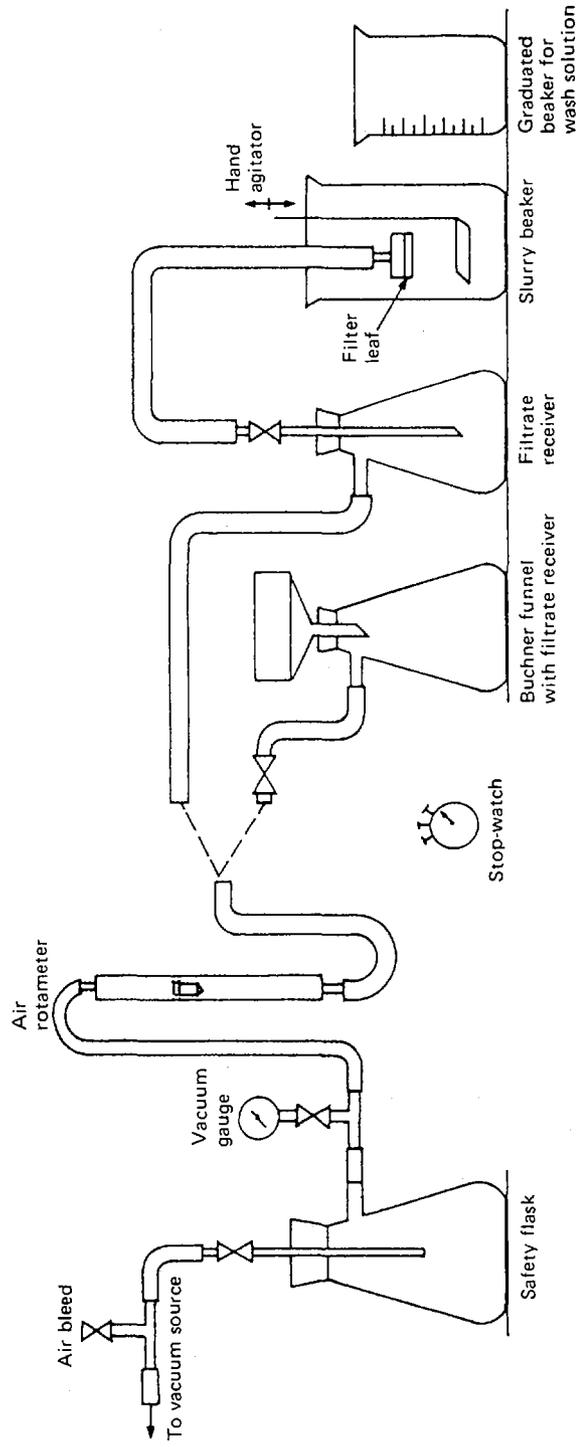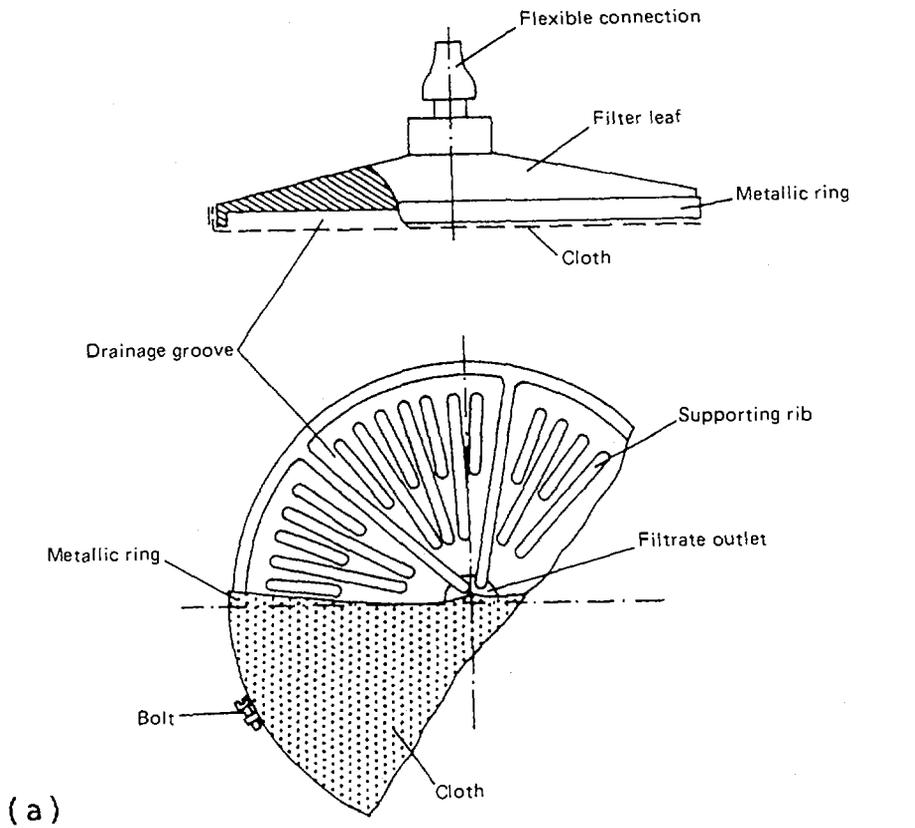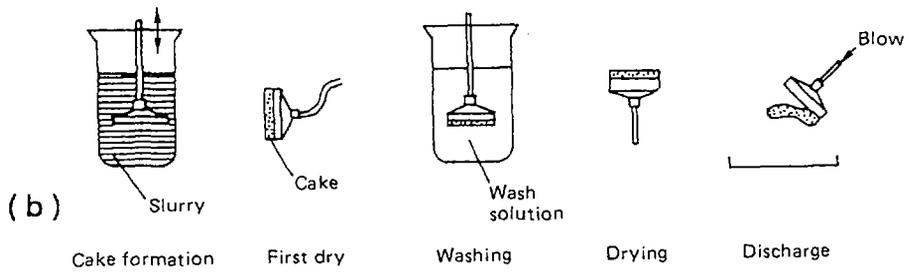
FIG. 10.   (a) Filter leaf; (b) filter leaf positions during testing.

flocculation is not easy to see. The filter cakes formed must be homogeneous and no segregation of the particles should be observed. Flocculant additions range from less than 100 g up to several hundred grams of flocculant (dry basis) per ton of ore (dry basis).

Flocs are sensitive to excessive agitation and they tend to break if agitated for prolonged times. After addition of the flocculant the slurry should be agitated in all tests for the same time (say 2 min) and in the same manner in order to obtain reproducible results.

5.2.3.4. Drum filtration

The slurry should be prepared and flocculated with one of the chosen flocculants at the chosen flocculant rate of addition. Cakes should be formed after different times using the filter leaf with a proper filter cloth. The vacuum must be at a proper level all the time (about 400–500 mmHg)[9]. The cakes are discharged by blowing into the filtrate pipe (which is disconnected first) and weighed wet and dry. The cakes are usually dried in a laboratory drier at 105°C to constant weight. The relationship between formation time and cake formation rate is determined. Cake thickness and moisture content are determined. The cake thickness which allows smooth cake discharge should also be determined.

These tests should be repeated with other flocculant addition rates and with other flocculant types.

Once the flocculant has been chosen and flocculant consumption, formation time and cake thickness have been determined, the washing curve should be determined. The cakes should be formed under optimal form conditions and, once formed, immersed after, say, 5 s of first dry (no cracks should occur) into the wash solution. The wash solution is acidified water; the acid added prevents any loss of uranium by hydrolysis (pH1–2). Each cake should be washed for a different time and the consumed volume of wash solution recorded. These volumes have to correspond to one to four cake moisture displacements. The cakes are then dried until cracks occur. The cakes are cut in half, and one half weighed wet and the other dry. The wet half is transferred to a beaker, repulped with acidified water and filtered over a clean filter paper (avoiding any contamination with uranium). The filtrate is collected and its volume measured and is then analysed for uranium content. Then all the cake is discharged and weighed both wet and dry.

5.2.3.5. Belt filtration

Flocculant selection and filter cloth selection are done by the same procedures as described before. The vacuum should be about 600 mmHg.

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[9] 1 mmHg = 133.3 Pa.

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Date:

Filter
Area:
Cloth:

Flocculant
Type:
Concentration:

Slurry
Temperature:
Density:
Solid content:
Specific gravity of liquid:

Wash solution
Temperature:
pH:

Test No.	Vacuum				Filtering time						Cake cracks		Wash volume			Cake thickness	Weight of cake		Filtrate volume	Repulped cake				Cake moisture	Flocculant consumption
	Form	1st dry	Wash	Dry	Form	1st dry	1st wash	2nd wash	3rd wash	Dry	Yes	No	1st wash	2nd wash	3rd wash		Wet	Dry		Weight		Filtrate			
																				Wet	Dry	Vol.	Uranium concn		

Remarks:

FIG. 11. Data sheet for vacuum filtration test.

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The quantity of slurry which will give the appropriate cake thickness on the Buchner funnel should be determined first and then a series of leaches made on the determined amount of the ore. Formation times for different flocculant additions should be determined.

Once cake formation conditions have been determined, the cakes have to be formed under optimal conditions and then washed with different amounts of wash solution corresponding to the different displacements of the moisture content (see Section 5.2.3.4). The wash solution has to be added in three separate portions, the
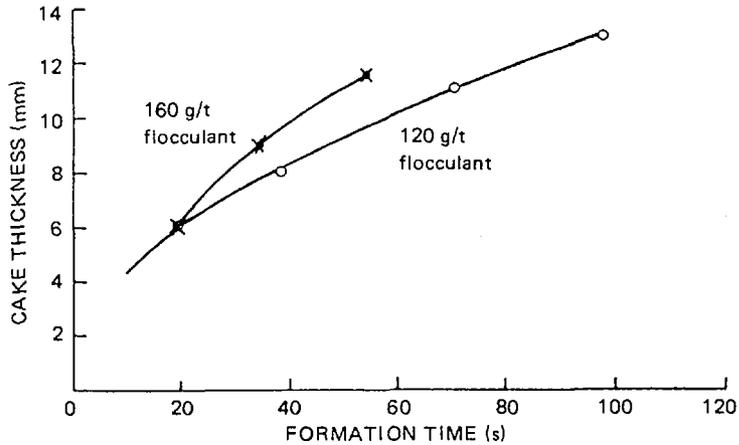
FIG. 12. Preliminary testing: cake thickness versus formation time for two rates of flocculant addition.
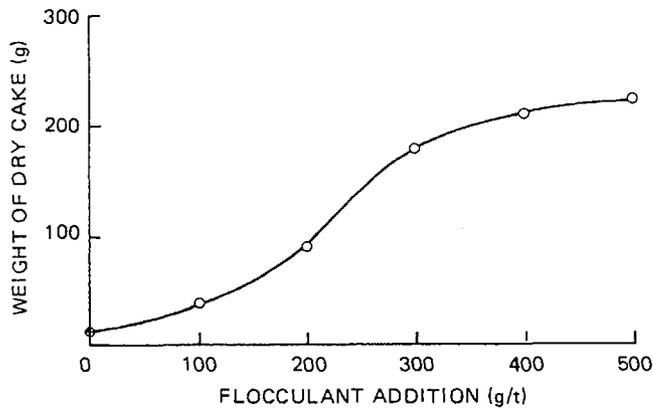
FIG. 13. Drum filtration simulation: cake weight versus flocculant addition (formation time 20 s).

60
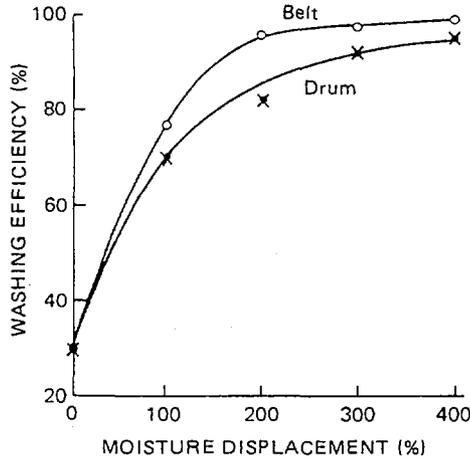
FIG. 14. Washing curve.

subsequent portion just after disappearance of the wash water from the surface of the cake. In this way contamination of the subsequent wash portion with uranium from the previous one is avoided.

5.2.3.6. Test data recording

Filtration is a complex operation and many data must be recorded in a short time. It is therefore very helpful if prepared data sheets are used. A sample data sheet is shown in Fig. 11.

Some examples of typical results for cake thickness versus formation time, cake weight versus flocculant addition and washing efficiency are given in Figs 12, 13 and 14, respectively.

6. URANIUM RECOVERY
FROM LEACH SOLUTIONS

The solutions obtained from leaching of the ore contain a complex mixture of cations and anions. The composition of such a solution is influenced by both the mineralogy of the ore and the leaching parameters employed. In some cases a uranium concentrate may be precipitated directly from a leach liquor, but as a general practice leach liquors are concentrated and partly purified before precipitation. There are two well established techniques for this purpose: ion exchange and solvent extraction.

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6.1. SOLUTION CHARACTERIZATION

A detailed chemical analysis of the leach solution provides reasonably reliable information about the applicability of the concentration process to be used and about any special problems that may be encountered in the application of such a process.

The possibility of recovering any by-product may also be indicated by the analysis. The main constituents to be analysed in acid leach liquors are: U, Fe (both Fe^{3+} and Fe^{2+}), Al, Ca, Cu, Mn, Zn, K, Mg, Na, Pb, V, Mo, Th, rare earths, Cl^-, F^-, SO_4^{2-}, PO_4^{3-}, SiO_2 and organic matter. The pH and oxidation potential of the liquor should also be determined. In the case of carbonate solutions many metals such as Fe, Ca, Mn and rare earths would be practically absent but concentrations of CO_3^{2-} and HCO_3^- must be determined.

To obtain a reasonable assessment of the range of variation of leach liquor composition it is advisable to analyse as many leach liquor samples as possible. Samples from 500–1000 g batch leach tests may be used.

As a general guide, treatment of leach liquors with a low uranium content, say less than 0.5 g/L, by ion exchange is preferred. For solutions with higher uranium content ($>$1 g/L) or with a pH higher than 1.2, or with high concentrations of impurities such as Fe^{3+}, solvent extraction is likely to be a better choice.

6.2. DIRECT PRECIPITATION

If alkaline leaching is used, a more or less selective dissolution of uranium takes place. If the uranium concentration is more than 3 g U_3O_8/L, uranium can be precipitated directly with sodium hydroxide, producing a sodium diuranate concentrate:

$$2Na_4UO_2(CO_3)_3 + 6NaOH \longrightarrow Na_2U_2O_7 + 6Na_2CO_3 + 3H_2O$$

sodium uranyl   sodium
tricarbonate   diuranate
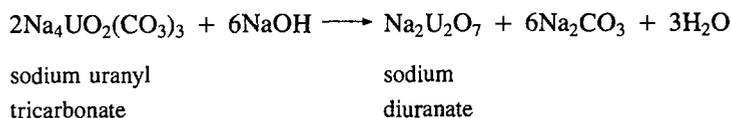
Uranium can also be precipitated with magnesia or ammonia if the excess carbonate is decomposed by first acidifying the solution. In the case of more dilute solutions ($<$3 g U_3O_8/L) reagent consumption would make direct precipitation uneconomical. In such a case preconcentration of uranium is required. This can usually be achieved by ion exchange. Solutions from the acid leach process invariably contain many impurities such as Al, Fe, Mn, Mg, Ti, V, Cu, Ni and SiO_2. Some of them, e.g. Fe, may be in a much higher concentration than uranium. Any direct precipitation technique would lead to an impure product.

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6.3. ION EXCHANGE

Ion exchange is a well established technique for the concentration and purification of uranium from sulphuric acid leach solutions. The standard process usually involves adsorption of uranium on anion exchange resins contained in columns. Several types of resin are available: there are weak base resins and strong base resins in the form of either fine or coarse beads. Quaternary ammonium groups implanted in the resin matrix are responsible for the exchange behaviour. The associated mobile, exchangeable anion can be represented as X^- in $R_4N^+X^-$ where X^- is NO_3^-, Cl^-, SO_4^{2-} or OH^-. A typical resin used in the uranium industry is obtained by copolymerizing styrene and divinylbenzene, chloromethylating the product and reacting it with trimethylamine:
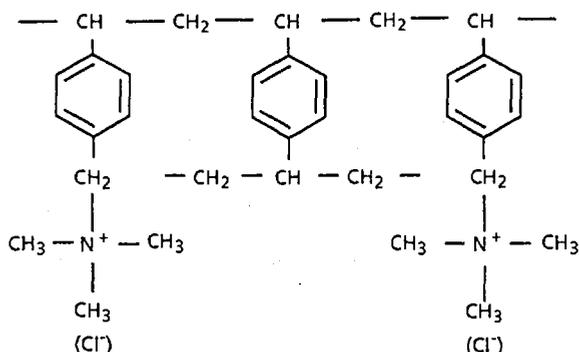
Most of the manufacturers market the resins under trade names such as Amberlite IRA-400, DeAcidite FF-530, DOWEX 1, IONAC and ZEOCARB.

In sulphuric acid leach solutions the following species of hexavalent uranium are present in dynamic equilibrium:

$$UO_2^{2+} \rightleftharpoons UO_2(SO_4) \rightleftharpoons [UO_2(SO_4)_2]^{2-} \rightleftharpoons [UO_2(SO_4)_3]^{4-}$$

However, in process solutions with a normal range of U concentration, pH and SO_4^{2-} concentration the dominant species may be $[UO_2(SO_4)_3]^{4-}$. Uranium uptake by a quaternary amine resin in the chloride form can be represented by the equilibrium reaction:

$$4R_4NCl + [UO_2(SO_4)_3]^{4-} \rightleftharpoons (R_4N)_4UO_2(SO_4)_3 + 4Cl^-$$
resin   solution   resin   solution

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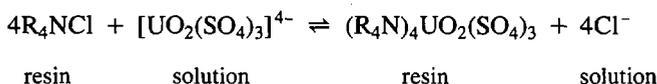
Uranium adsorbed on the resin can be displaced by contacting it with a solution of sodium chloride, causing the reaction shown above to proceed from right to left. A concentrated and relatively pure uranium solution is thereby obtained by adsorption followed by elution.

In carbonate leach liquors uranium is present as the anionic complex $[UO_2(CO_3)_3]^{4-}$. Its adsorption and elution are similar to the processes for sulphate complex. In this case, however, most of the barren effluent carrying carbonate and bicarbonate is recycled to the leaching circuit.

6.3.1. Characteristics of resins used for uranium recovery

Type: Weak and strong base anion exchange resins having quaternary amine functional groups (strong bases are the most commonly used).

Particle shape and size: Spherical beads, the majority of which are 0.3–0.9 mm in diameter (+50 to −16 mesh).

Density: Apparent densities are 600–750 kg/m³. The apparent density is defined as the product of the true density of the wet beads and (1 minus the fraction of void space in the packed bed). For commercial resins the void space is approximately 40% of the packed volume.

Exchange capacity: The inherent capacity of an ion exchange resin is governed by the number of fixed ion exchange sites available. This can be ascertained by measuring the maximum amount of mobile ions that may be adsorbed onto or desorbed from a given weight or volume of resin. Resins used for uranium recovery have exchange capacities in the range 3.5–5.0 equivalents per dry kilogram or 1.2–1.8 equivalents per litre of wet, settled resin.

Moisture content: The equilibrium moisture content of a resin varies with the nature of the mobile ion present. For the chloride form in which the resin is usually supplied it is in the range 40–60%.

6.3.2. Factors affecting uranium adsorption capacity

The uranium adsorption capacity of a given resin varies with the composition of the feed solution. Some of the important factors that affect the capacity are given below.

Effect of pH: Uranium capacity increases with decreasing acidity (increasing pH), as shown in Table V. Adsorption is poor below pH1.2. In practice there is also an upper limit. Above pH1.8 ferric iron, which is invariably present in all leach liquors, is increasingly taken up by the resin. Moreover, in the presence of anions such as PO_4^{3-} uranium may be precipitated in solution or in the resin phase.

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TABLE V. EFFECT OF ACIDITY ON URANIUM CAPACITY

(resin: Amberlite IRA-400; feed solution: 1.2 g U_3O_8/L, 30.0 g SO_4^{2-}/L, excess sulphate added as $MgSO_4 \cdot 7H_2O$)

pH	Free acidity (g H_2SO_4/L)	U capacity (g U_3O_8/L wsr)[a]
1.35	12.0	59
1.50	4.5	73
1.85	2.0	77.5

[a] L wsr: litre of wet, settled resin.

TABLE VI. EFFECT OF URANIUM CONCENTRATION ON URANIUM CAPACITY

(resin: Amberlite IRA-400; feed solution: pH1.45, 30.0 g SO_4^{2-}/L, excess sulphate added as $MgSO_4 \cdot 7H_2O$)

U concn (g U_3O_8/L)	U capacity (g U_3O_8/L wsr)[a]
0.20	42
0.75	65
1.2	70
3.0	90

[a] L wsr: litre of wet, settled resin.

Effect of uranium concentration: Other conditions remaining the same, an increase in uranium concentration increases uranium capacity (Table VI).

Effect of sulphate concentration: An increase in total sulphate concentration leads to a gradual decrease in uranium capacity (Table VII). This is essentially due to an increase in bisulphate (HSO_4^-) concentration, this ion being a strong competitor for resin sites.

TABLE VII. EFFECT OF SULPHATE
CONCENTRATION ON URANIUM CAPACITY
*(resin: Amberlite IRA-400; feed solution: 1.2 g U_3O_8/L,
5 g free H_2SO_4/L, excess sulphate added as
$MgSO_4 \cdot 7H_2O$)*

Sulphate concn (g SO_4^{2-}/L)	U capacity (g U_3O_8/L wsr)[a]
20	75
30	73.5
50	69

[a] L wsr: litre of wet, settled resin.

Effect of phosphate and arsenate: Phosphate (PO_4^{3-}) and arsenate ($As_2O_4^{3-}$), if present in the solution, form anionic complexes with uranium which are adsorbed. If their combined concentration is above 0.5 g/L uranium tends to be precipitated in the resin phase, particularly when the solution is low in free acid, as in elution. The elution of uranium is therefore slowed. The use of an eluant more acidic than usual (0.075M H_2SO_4) may ease this situation but the final uranium product would be contaminated with these ions.

Effect of ferric iron: Ferrous iron is not adsorbed on the resin to any significant extent. However, ferric iron forms complexes of the types $[Fe(SO_4)_2]^-$ and $[Fe(OH)(SO_4)_2]^{2-}$ which can be adsorbed and thus compete with uranium for resin sites. The decrease of uranium capacity depends on the concentration of Fe^{3+}, the Fe/U mole ratio and the pH of the solution. This effect is significant at a pH of 1.5 with a ferric ion concentration greater than 5 g/L and an Fe/U mole ratio greater than 2. As in most of the leach liquors with Fe^{3+}/U $\gg$ 1, at the start of the adsorption the resin becomes saturated with iron. During the rest of the adsorption cycle uranium replaces most of the iron in the column.

Effect of other constituents: There are no fixed rules for estimating how a particular combination of ions will behave in the ion exchange process or to what extent it will affect the purity of the product. Only general guidelines can be given and the true behaviour of the leach liquor has to be assessed by experiments.

In general, elements forming high valency ions and complex anions are suspect. Components such as Si and Ti which tend to undergo irreversible hydrolysis and polymerization are possible causes of problems. In addition, if V, Mo and Co are present, investigation for possible poisoning of the resin (irreversible adsorption causing accumulation of impurities on the resin) or contamination of the final product

should be made. Considerable information is available in the literature on the behaviour of specific resin poisons and procedures for regeneration of resin affected by such constituents.

6.4. SOLVENT EXTRACTION

Solvent extraction is the second technique used for the concentration and purification of uranium from dilute leach liquors. It can be readily applied to sulphuric acid leach solutions but it cannot be used with carbonate leach solutions because there are as yet no extractants capable of extracting uranium at a high pH and in the presence of a high salt content.

Solvent extraction consists essentially of two steps. The first is 'extraction', where the uranium bearing leach solution is thoroughly mixed with an organic solvent mixture and the uranium is selectively transferred to the organic phase. The organic solvent mixture is made by dissolving an organic reagent, called the 'extractant', and a 'modifier' (optional) in a hydrocarbon-like solvent. The extractant forms a complex with uranium which has a high miscibility in the hydrocarbon phase. The second step, called 'stripping', consists of re-extracting the uranium from the organic phase into the aqueous phase. This is achieved by contacting the extract with an aqueous solution of a suitable reagent.

It is possible to obtain reasonably reliable data from laboratory batch and continuous extraction tests to assess the applicability of amine extraction for the leach solution in hand. Many of the parameters required for operating a large scale system can also be confidently determined by such tests.

6.4.1. Uranium extraction by amines

For the extraction of uranium from sulphate solutions mostly tertiary amines (R_3N) are used. Secondary amines (R_2NH), however, find limited applications. Important factors influencing metal extraction by amines are the structure and length of the carbon chain. The amines which find industrial application are the alkylamines with molecular weights of about 250 (tertiary) and 600 (secondary).

Two types of extractant are useful for recovering uranium from sulphate solutions. The first is acidic: the dialkylphosphoric acids, such as di(2-ethylhexyl)phosphoric acid (DEHPA):

$$
\begin{array}{cc}
\text{OR} & \text{C}_2\text{H}_5 \\
| & | \\
\text{O} = \text{P} - \text{OH} \quad \text{where R is} & -\text{CH}_2 - \text{CH} - (\text{CH}_2)_3 - \text{CH}_3 \\
| & \\
\text{OR} & \\
\text{DEHPA} & \text{2-ethylhexyl group}
\end{array}
$$
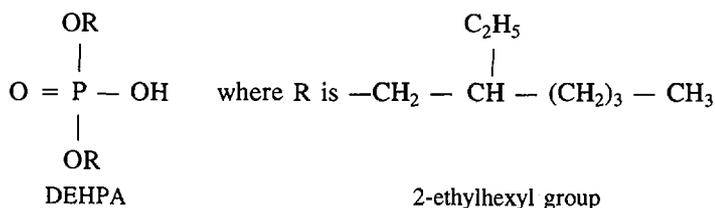
DEHPA extracts uranium in the cationic form. It has the inherent ability to extract any cation. Though uranium is preferentially extracted, at the acidity of most of the leach liquors ferric iron is more strongly extracted. Iron can first be reduced to the ferrous form by addition of powdered iron, but even so DEHPA is not sufficiently selective for uranium to find general application in processing leach liquors.

The second type of extractant is basic. High molecular weight alkylamines are the most important reagents of this type. They exhibit a high degree of selectivity for uranium. Moreover, the extracted uranium can be readily stripped using aqueous solutions of sodium chloride or sodium carbonate. The alkylamines therefore find ready application in uranium hydrometallurgy. To obtain a high degree of uranium recovery and a high degree of separation from associated impurities the extraction has to be repeated several times. This is usually achieved in a multistage counter-current extraction and stripping system. The most common type of contacting equipment used for this purpose on an industrial scale is the mixer–settler assembly.

Those alkylamines with lower molecular weights tend to be soluble in the aqueous phase, which is undesirable. A number of amines suitable for uranium extraction are marketed under different trade names: for example, Amberlite LA-1 and Amberlite LA-2 are secondary amines with 21–24 carbon atoms; Alamine-336, Adogen-364 and Hostarex A-327 are tertiary amines with 24–30 carbon atoms. These amines are generally used as 3–5 vol.% solutions in a hydrocarbon diluent, such as kerosene, along with 2–3 vol.% aliphatic primary alcohol, such as decanol, added as a modifier.

6.4.2. Mechanism of extraction and stripping

The amine, on contact with a sulphuric acid solution, is converted into the sulphate or bisulphate salt:

$$2R_3N + H_2SO_4 \rightleftharpoons (R_3NH)_2(SO_4)$$

organic   aqueous   organic

$$R_3N + H_2SO_4 \rightleftharpoons (R_3NH)(HSO_4)$$
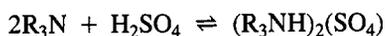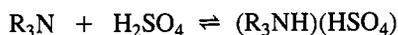
The actual extraction reactions are complex. However, in a simplified way, the extraction can be depicted as an anion exchange where the anionic uranium complex from the aqueous solution replaces the sulphate or bisulphate ion in the organic phase:

$$(R_3NH)_2(SO_4) + [UO_2(SO_4)_2]^{2-} \rightleftharpoons (R_3NH)_2[UO_2(SO_4)_2] + SO_4^{2-}$$

organic   aqueous   organic   aqueous

$$2(R_3NH)_2(SO_4) + [UO_2(SO_4)_2]^{2-} \rightleftharpoons (R_3NH)_2[UO_2(SO_4)_2] + 2HSO_4^-$$

68
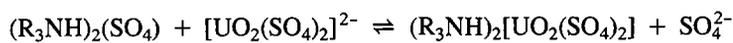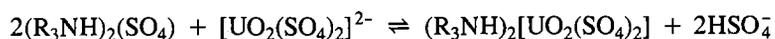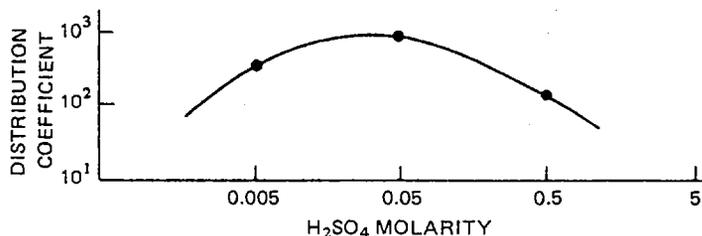
FIG. 15. *Effect of sulphuric acid concentration on uranium extraction with a tertiary amine.*

The reactions indicate that uranium extraction would decrease with increasing sulphate or acid (bisulphate) concentration. The effect of sulphuric acid concentration on uranium extraction with a tertiary amine is shown in Fig. 15.

When the loaded solvent is contacted with a stripping agent, e.g. 1.0M NaCl containing 0.05M H_2SO_4, the chloride ion displaces the uranyl sulphate complex from the organic phase to the aqueous phase:

$$(R_3NH)_2[UO_2(SO_4)_2] + 2NaCl \rightleftharpoons 2R_3NHCl + [UO_2(SO_4)_2]^{2-} + 2Na^+$$

  organic   aqueous   organic   aqueous
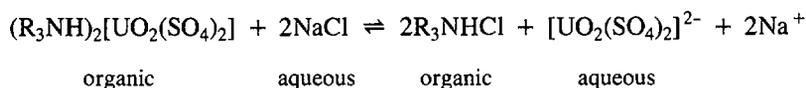
A typical solvent extraction flow sheet for uranium using amine as extractant is shown in Fig. 16.

7. URANIUM RECOVERY
BY ION EXCHANGE

7.1. AIMS OF LABORATORY TESTS

Ion exchange laboratory tests have the following purposes:

(a)   To determine the uranium loading capacity of the resin, using leach liquors from the ore being tested. In some cases the pH of the leach liquor may have to be adjusted to obtain adequate uranium loading. This will be an added cost in the full scale process.

(b)   To prepare loading and elution curves for the resin, liquor (after pH adjustment if needed) and eluant being tested.

(c)   To study the loading and elution behaviour of the resin after repeated cycling. The cumulative effects of some constituents of the leach solution can then be ascertained.
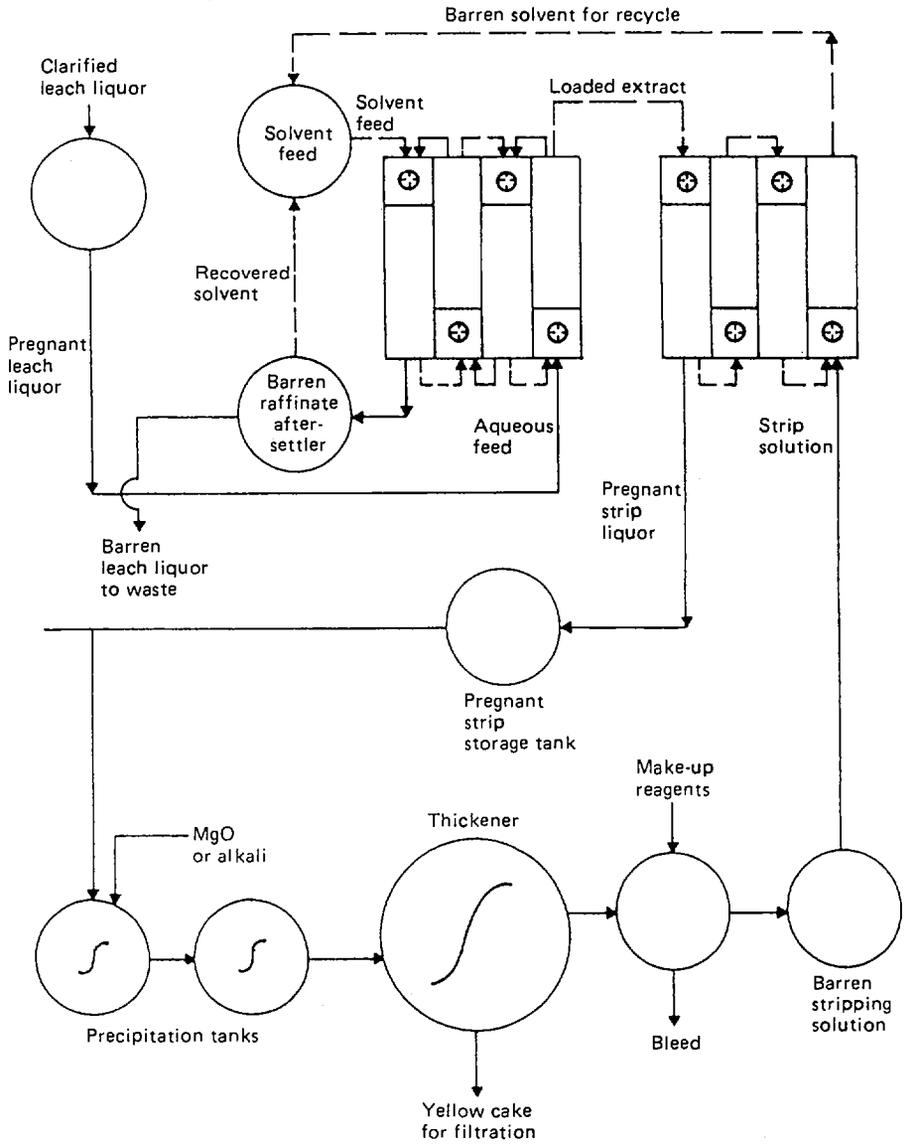
FIG. 16. Typical flow sheet for recovery of uranium from sulphate leach liquors by solvent extraction with amines.

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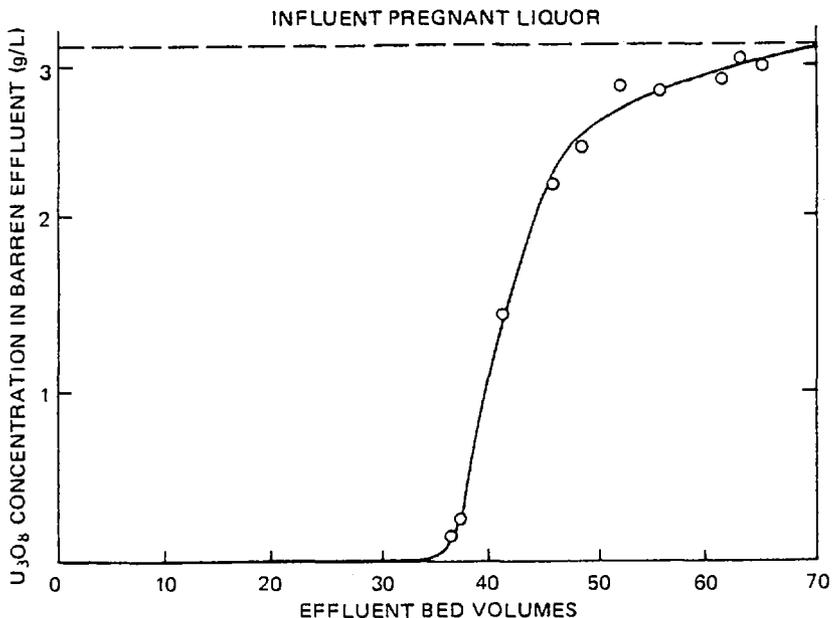
FIG. 17. Typical loading curve.

(d)   To determine the retention times for adsorption and elution and to determine the size and number of columns to be used in the full scale process.

(e)   To accumulate sufficient uranium rich eluate for precipitation tests, and from these to determine the purity of the uranium concentrate and to verify that it meets specifications.

All this information can be obtained from tests with a single column or with multiple columns (usually three) operated for 25 to 50 cycles.

7.2.   CHANGES IN EFFLUENT COMPOSITION IN THE ADSORPTION–ELUTION CYCLE

Changes in effluent composition through the adsorption and elution stages can be outlined as follows, considering a synthetic feed solution (Fig. 17):

Adsorption: In this example 100 mL of a resin in the chloride form are packed in a column of 2.5 cm diameter and 40–50 cm height. The resin would be about 20 cm deep. A pregnant solution containing 1.0 g U_3O_8/L, with 25 g SO_4^{2-}/L added as $MgSO_4 \cdot 7H_2O$, and adjusted to pH1.5 with H_2SO_4 is passed through the bed at a regulated rate.

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Bed volume (BV): 100 mL.

Void volume in the resin bed: 40% of *BV* = 40 mL.

If the solution is passed through the column at 20 mL/min, then:

$$\text{retention time } (RT) = \frac{\text{void volume}}{\text{flow rate}} = 2 \text{ min}$$

If the effluent is collected in 100 mL (1 BV) fractions and chemically analysed the following is normally found:

(1)   The first bed volume essentially displaces the water in the column.

(2)   The next few bed volumes carry the chloride displaced from the resin by the sulphate and bisulphate present in the feed solution. Practically no uranium will be detected.

(3)   Up to 35–40 bed volumes, the effluent is barren, containing mainly sulphate and bisulphate ions but no uranium.

(4)   While the concentration of sulphate and bisulphate remains nearly constant in subsequent fractions, that of uranium gradually increases and in the next 40–45 bed volumes it will reach the same level as in the influent solution (1.0 g U_3O_8/L).

(5)   When the uranium concentration in the effluent is about 1 % of the influent concentration (0.01 g U_3O_8/L), *breakthrough* is said to occur. The volume of solution passed up to that stage is called the breakthrough volume and the total uranium adsorbed, expressed in g U_3O_8/L wsr,[10] is called the breakthrough capacity. This can be calculated as follows:

$$\text{breakthrough capacity (g } U_3O_8\text{/L wsr)} = \frac{C(V - v)}{BV}$$

where

C is the concentration of uranium in the influent solution (g U_3O_8/L),
V is the volume of effluent collected up to the breakthrough point (L),
v is the void volume of the resin bed (L).

In the present example, C = 1.0 g U_3O_8/L, V = 4.0 L, v = 0.04 L and BV = 0.1 L and hence:

$$\text{breakthrough capacity} = 1 \times \frac{(4 - 0.04)}{0.1} = 39.6 \text{ g } U_3O_8\text{/L wsr}$$

When the concentration of uranium in the effluent reaches the same level as in the influent (1.0 g U_3O_8/L) the column has reached saturation.

---

[10]   L wsr: litre of wet, settled resin.
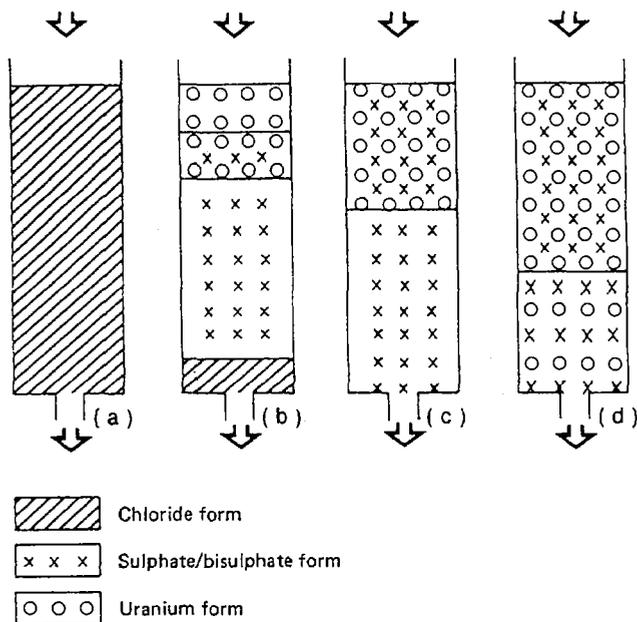
FIG. 18. Changes taking place in resin column during loading.

7.3. CHANGES IN RESIN COMPOSITION IN THE ADSORPTION–ELUTION CYCLE

The changes taking place in the resin column during the adsorption cycle are represented in Fig. 18:

(a)   The resin is saturated with chloride.

(b)   The top portion of the resin is saturated with uranium, while the middle portion is only partly loaded with uranium, establishing a concentration gradient; chloride is more or less displaced by SO_4^{2-} and HSO_4^- except in the end portion.

(c)   Chloride is completely displaced from the column. Uranium is loaded in the upper half of the column, the lower portion containing only SO_4^{2-} and HSO_4^-.

(d)   While uranium saturation is reached in the upper part of the column there is a gradient in its concentration almost up to the end. The effluent shows traces of uranium, corresponding to the breakthrough point.

(e)   (not shown in Fig. 18) The column reaches equilibrium with respect to U, SO_4^{2-} and HSO_4^- distribution and the effluent shows the same uranium concentration as the influent, signalling saturation of the column.

Elution: When the column reaches saturation the flow of pregnant solution is cut off and a backwash is given with water to displace the feed solution from the

column and to loosen and expand the bed to wash off accumulated fine solids. The resin is allowed to settle and an eluant is passed down the column to displace the uranium. The most commonly used eluant consists of 1.0% sodium chloride with 0.05–0.075M sulphuric acid. The retention time provided for elution is in the range 8–12 min. For a retention time of 8 min the flow rate can be calculated as follows:

$$\text{elution flow rate} = \frac{\text{void volume}}{RT} = \frac{40}{8} = 5 \text{ mL/min}$$

A typical elution curve is shown in Fig. 19. In this example, for near complete displacement of uranium 20–25 bed volumes of eluant have to be passed. To estimate the capacity of the resin at saturation, for a given feed solution, the total uranium (g U_3O_8) in the eluate must be determined; then:

$$\text{saturation capacity} = \text{total } U_3O_8 \text{ in eluate} \times \frac{1000}{100} \text{ g } U_3O_8/L \text{ wsr}$$

From Fig. 19 it can be seen that most of the uranium is eluted with relatively few bed volumes of eluant but complete elution requires passage of many more volumes. Moreover, when the bed is rinsed after elution the initial effluent contains mostly the eluant. To avoid dilution of the uranium solution sent for precipitation, while achieving at the same time a high degree of uranium recovery, different fractions of eluate are collected separately. The leaner fractions are recycled for elution.

After flushing with water to displace the eluant the column is ready for another cycle of adsorption and elution.

7.4. ION EXCHANGE TESTS WITH A SINGLE COLUMN

7.4.1. Resin loading tests

Laboratory testing of the ion exchange process can be started soon after the leaching parameters have been at least approximately determined. For preliminary tests leach liquor can be accumulated from the 500–1000 g leach tests.

Clarified solutions free from detectable suspended solids are to be used for the tests. Detailed chemical analysis that includes minor constituents can give a fairly reliable indication of the behaviour of the solution during the process. Preliminary tests can be conducted using single columns containing 50–100 mL of wet, settled resin. Adsorption isotherms (see example in Fig. 17) can be constructed for different retention times and pH levels of feed solution.

The retention time can be 2–6 min and the pH from that of the original leach liquor to 1.8. The pH can be adjusted by addition of small amounts of reactive magnesia. The amount of MgO consumed per litre of solution, the composition of any precipitate formed during neutralization and the extent of uranium loss in such

74
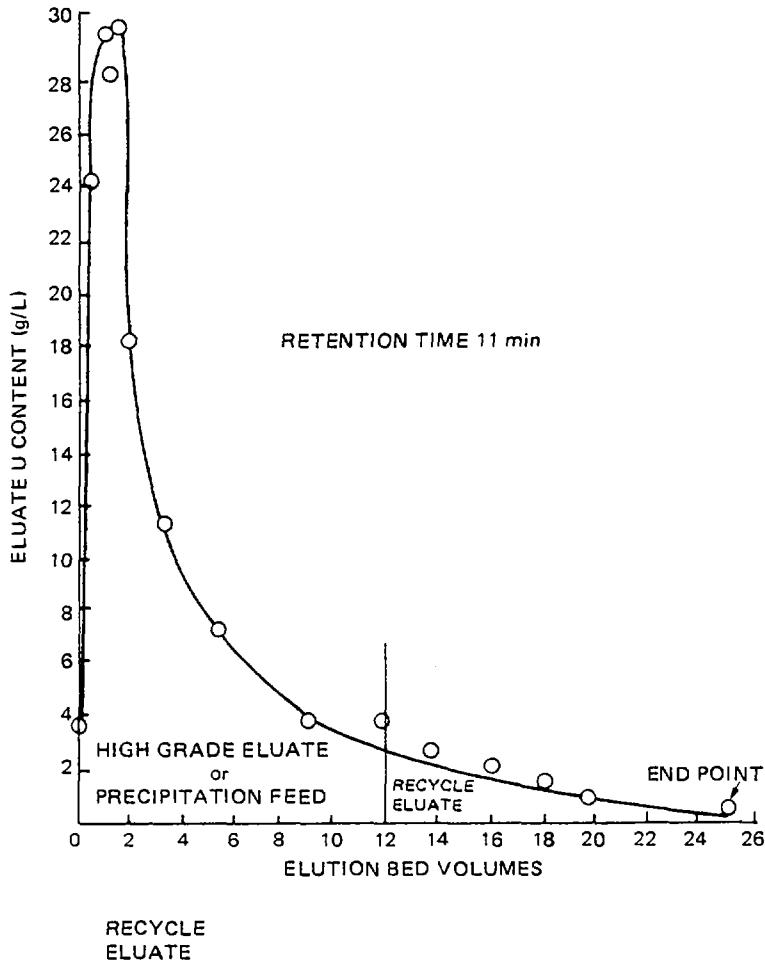
FIG. 19. Typical elution curve.

a precipitate should be recorded. The breakthrough volume, breakthrough capacity and saturation capacity for uranium are recorded each time a test is conducted. If a feed solution with 1.0 g U_3O_8/L and a 100 mL resin column are used, 8–10 L of feed solution are needed for a single test. The exact volume required depends on the uranium concentration.

A simple manually operated system for these tests is shown in Fig. 20. By locating storage vessels of pregnant solution, wash water and eluant at a suitable height and connecting them to the column through flow meters and two way stopcocks the flow of solution can be controlled.
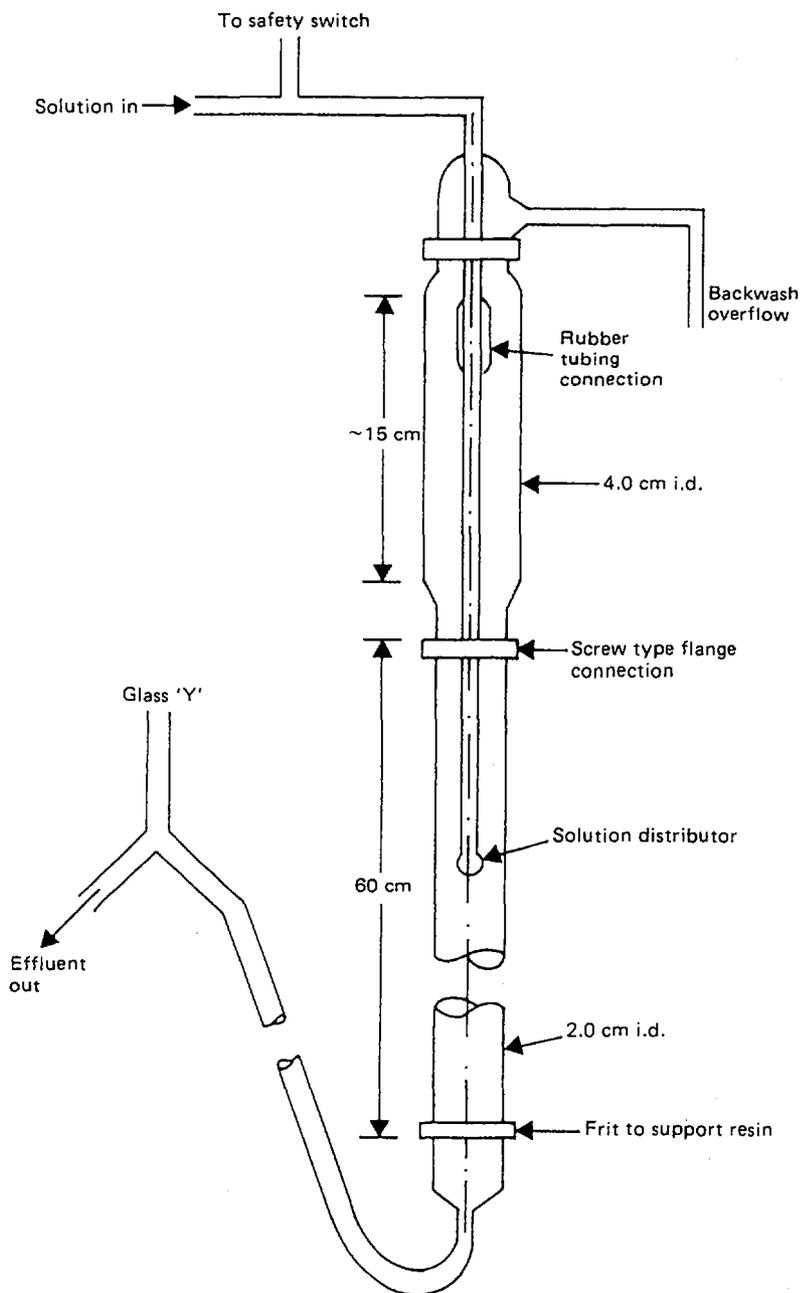
FIG. 20. Single column apparatus for ion exchange tests.

The following example shows the type of data that can be collected in such a test run:

(1) Pregnant solution composition:

U_3O_8	1.3 g/L
Free acid	0.3 g H_2SO_4/L
Total Fe	2.4 g/L
Fe^{3+}	1.9 g/L
SO_4^{2-}	12.5 g/L
$Na_2S_4O_6$ (tetrathionate)	0.008 g/L
As + P_2O_5	0.08 g/L
SiO_2	0.30 g/L
V_2O_5	0.005 g/L
EMF	450 mV
pH	1.7–1.85

(2) Volume of wet, settled resin (Cl^- form) after backwash: 100 mL

(3) Average feed flow rate: 12.5 mL/min

(4) Average retention time calculated: 3.5 min

(5) The breakthrough volume is defined as the sum of the bed volume fractions up to the middle of the effluent fraction collected in which the average uranium concentration is 0.02 g U_3O_8/L.

(6) Total volume of barren solution collected: 3.55 L

(7) Result of U_3O_8 assay of composite barren solution: 0.008 g/L

(8) Breakthrough capacity calculated: 46.2 g U_3O_8/L
(If corrected for U in the barren solution the breakthrough capacity is 45.9 g U_3O_8/L.)

(9) After breakthrough the passage of pregnant solution is continued until three successive fractions of the effluent contain about 1.3 g U_3O_8/L. At this stage the column has reached saturation.

(10) The passage of pregnant solution is interrupted and the column is flushed with 1 bed volume of water (acidified to pH1.5 with H_2SO_4). The total U_3O_8 in the wash water is determined — say 0.15 g.

(11) The column is backwashed with 2 bed volumes of water at about 100 mL/min to expand the bed to about 200 mL, but ensuring that no resin passes out of the column. The resin is then allowed to settle.

(12) The loaded column is eluted using 1.0M NaCl + 0.05M H_2SO_4 at a rate of 4 mL/min, corresponding to a retention time of 10 min. The eluate is collected in 1 BV fractions and the uranium assayed in each fraction. The elution is continued until the U_3O_8 content in a fraction is 0.2 g/L or less.

(13) Total volume of eluate collected: 20 bed volumes = 2.0 L

(14) Total U_3O_8 in eluate (by analysis): 6.5 g

(15) Saturation capacity of the resin (calculated): 65 g U_3O_8/L wsr

(16) The tests are repeated with different retention times for adsorption and elution.

7.4.2. Resin poisoning tests

It is a normal experience that uranium loading obtained with a fresh resin declines rapidly in the first few cycles of operation of the column and then declines more slowly. This is partly due to the behaviour of the resin and partly due to the irreversible adsorption of some impurities from the feed solution. The latter is called resin poisoning. The nature and extent of such poisoning can be assessed by running 20–50 adsorption–elution cycles using a single column of resin. A set-up of the type shown in Fig. 20 can be used for this purpose.

In some cases a larger number of cycles may be needed. Since such studies are time consuming, automatic and semiautomatic equipment has been used in some laboratories. A block diagram of one such unit is shown in Fig. 21. The sequential timer is an electrically operated device which turns the solenoid valves on and off in sequence to cause loading, backwashing, elution and washing of the resin in that order. This cycle is repeated automatically. The metering pump is used to pump the various liquids through the column at preset rates. The ion exchange column and the sequence of operations remain more or less the same as in the manually operated system.

After the predetermined number of cycles of operation (say 25) using a synthetic feed solution of a fixed composition, samples of resin are withdrawn from the column and tested for parameters such as strong base capacity, total chloride capacity, moisture, sulphated ash and composition of that ash, silica, polythionates and saturated uranium loading capacity. The values of these parameters are compared with those obtained with a sample of unused resin.

For comparison two typical sets of results are shown in Tables VIII and IX. In the first case, after 50 cycles the resin has not shown any significant deterioration but in the second case serious poisoning due to polythionates is noted.

7.5. MULTICOLUMN ION EXCHANGE OPERATION

The overall uranium recovery operation by ion exchange consists of the following stages:

— Adsorption (loading)
— Flushing
— Backwashing and settling
— Elution
— Rinsing.

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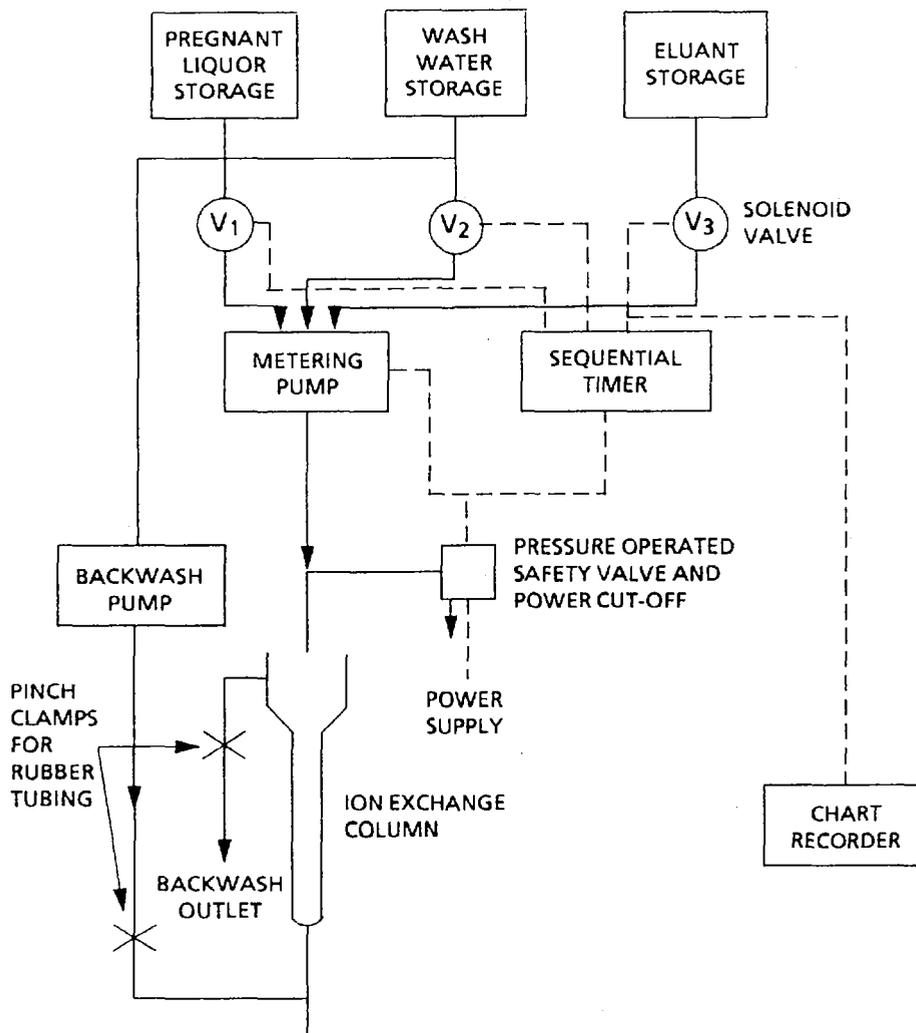
FIG. 21. Automatic unit for resin life tests.

Elution costs per unit volume of resin are essentially the same regardless of uranium loading. Therefore, considerations of economy dictate that the resin should be saturated with uranium before it is eluted. Moreover, saturation with uranium minimizes the loading of impurities such as Fe^{3+} on the column. The nature of the adsorption curve (Fig. 17), on the other hand, indicates that it is impossible to achieve complete removal of uranium and at the same time achieve saturation of the resin in a single column operation. This problem is solved by using two (usually) or three columns in series and passing the feed solution at such a rate that when

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TABLE VIII. RESULTS OF RESIN ANALYSIS AFTER REPEATED USE, SHOWING NO SIGNIFICANT POISONING

Property tested	Unused resin	Resin after 50 cycles
Strong base capacity (meq/g)	3.25	3.10
Total chloride capacity (meq/g)	3.60	3.55
Moisture (wt%)	45	40
Sulphated ash (wt%)	Low	0.5
SiO$_2$ (wt%)	Low	0.1
Polythionate sulphur (%S)	Nil	0.1

TABLE IX. RESULTS OF RESIN ANALYSIS AFTER REPEATED USE, SHOWING POLYTHIONATE POISONING

Property tested	Unused resin	Resin after 50 cycles
Strong base capacity (meq/g)	3.0	0.9
Total chloride capacity (meq/g)	4.0	2.8
Moisture (wt%)	45	42
Sulphated ash (wt%)	0.3	0.4
SiO$_2$ (wt%)	0.2	0.25
Polythionate sulphur (%S)	Nil	4.5

breakthrough occurs on the second (or the third) column the first column is already nearly saturated. This column can then be cut off from the adsorption circuit and taken for backwash and elution. A simplified layout of a three column set-up is shown in Fig. 22. The changes taking place on the columns in one cycle are shown in Fig. 23.

7.5.1. Description of set-up

The ion exchange columns are made of glass tubing with 3 cm i.d. and a length of 150 cm. About 500 mL of wet, settled resin are contained in each column, representing about half the column volume.
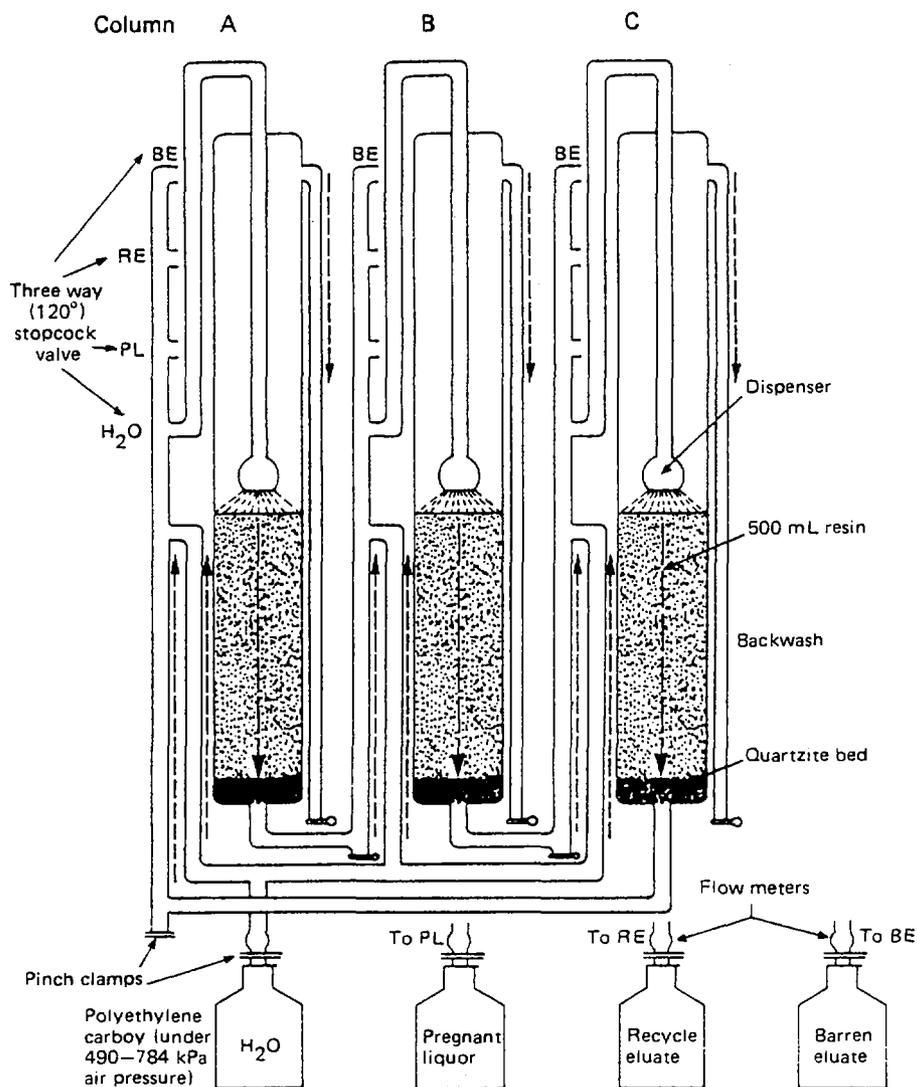
FIG. 22. Three column ion exchange set-up. BE: barren eluate; RE: recycle eluate; PL: pregnant liquor.

Pregnant liquor, recycle and barren eluates and water are stored in polyethylene carboys which can be pressurized to 490–784 kPa to send the liquids in the right sequence to the columns. These solutions are fed to the resin bed through flow meters. Flow rates are controlled by pinch clamps and three way (120°) glass stopcocks.
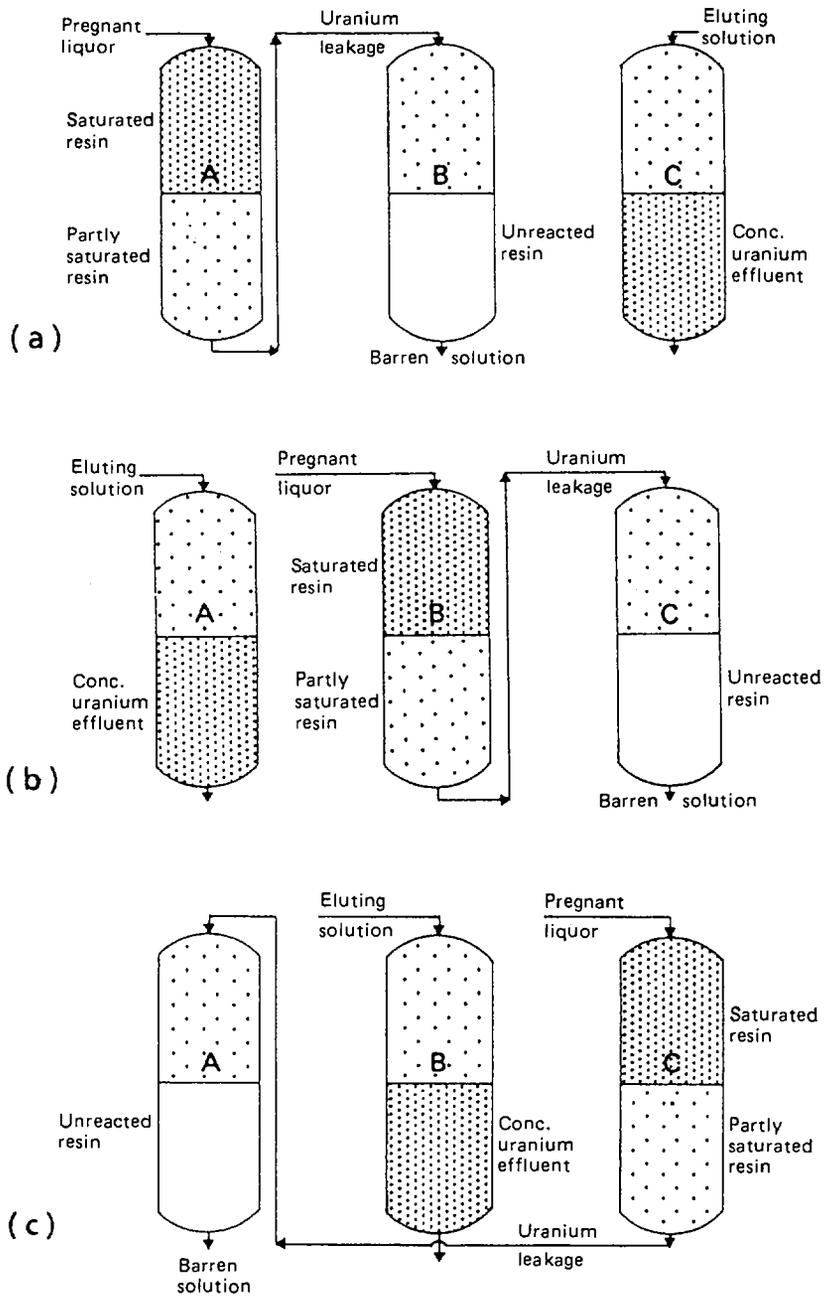
FIG. 23. Operation of a three column ion exchange set-up: (a) columns A and B: adsorption; column C: elution; (b) columns B and C: adsorption; column A: elution; (c) columns C and A: adsorption; column B: elution.

Recycle eluate (about 11 bed volumes) is used on a freshly loaded column, followed by another 11 bed volumes of neutral barren eluate adjusted to 1% NaCl and 0.05–0.075M H_2SO_4. The high grade eluate obtained in the first half of the elution step may be used for precipitation studies to recover uranium concentrate by two stage or single stage precipitation.

7.5.2. Sequence of three column operation

(1) Two columns, A and B, are connected in series and loaded with pregnant liquor. Barren effluent flowing out of column B is collected. The first bed volume of displaced water from column B is mixed with backwash.

(2) The flow of pregnant liquor to column A is stopped when breakthrough (about 1% of influent U_3O_8 level) occurs on column B.

(3) One bed volume of water is passed through column A to displace the residual liquor to column B.

(4) Column A is removed from the series flow, column C is connected in series to column B and pregnant liquor is fed to column B.

(5) Column A is backwashed with 2.5–3.0 bed volumes of water (pH1.5). The effluent is separately collected and analysed for uranium. After the resin has been allowed to settle, 1 bed volume of recycle eluate is passed through column A to displace water and the effluent is combined with backwash.

(6) Approximately 11 bed volumes of influent recycle eluate are collected as high grade eluate. The actual volume depends on the elution conditions and the nature of the resin, and is determined by single column elution tests (Fig. 19).

(7) One bed volume of feed NaCl + H_2SO_4 eluant is fed to displace the recycle eluate to the high grade eluate already collected, i.e. a total of 11 + 1 bed volumes are collected as high grade eluate to be taken for precipitation of uranium.

(8) Eleven bed volumes of barren eluate are collected as recycle eluate.

(9) One bed volume of water is used to displace the remaining recycle eluate from the column. Thus a total of 11 + 1 bed volumes of recycle eluate are available for the first half of the uranium elution of fully loaded column B. One more bed volume of wash water through column A is collected separately and analysed for uranium.

At this stage column A is ready to be connected as the end column on a subsequent adsorption (order C → A). Thus the cycle A → B, B → C, C → A, A → B continues.

The retention times for adsorption and elution derived from the operation of single columns can be used for the operation of the three column set-up. For smooth operation the total loading time (lead column + scavenger column) should be about 25% more than the time required for rinsing, backwashing, elution and rinsing.

TABLE X. LOADING AND ELUTION DATA

Loading data

Cycle No.	Columns	Total time (h)	Actual retention time (min)	Feed solution pH	Feed solution g U_3O_8/L	Last fraction (g U_3O_8/L) Lead column	Last fraction (g U_3O_8/L) End column	Barren effluent Vol. (L)	Barren effluent g U_3O_8/L	U_3O_8 recovery (%)	Resin loading (g U_3O_8/L wsr)	Remarks
1	A B	15	3.5	1.5	2.5	1.0	0.25	50	0.001	99.8	69	

Elution data

Cycle No.	Column	Total time (h)	Actual retention time (min)	Recycle eluate entering Vol. (L)	Recycle eluate entering g U_3O_8/L	High grade eluate g U_3O_8/L	High grade eluate Vol. (L)	End elution (BV)	Recycle eluate leaving Vol. (L)	Recycle eluate leaving g U_3O_8/L	Remarks
1	A	7.5	9	5.5	—	9.0	5.5	19	5.5	4.25	

A uranium concentrate can be precipitated from the strong eluate in one or two stages. After precipitation, the uranium free liquor can be reused as barren eluate for the next loaded column after adjusting the pH and the concentrations of Cl^- and SO_4^{2-}.

The loading and elution data can be recorded in many ways. One possibility is shown in Table X.

8. URANIUM RECOVERY
BY SOLVENT EXTRACTION

8.1. LABORATORY TESTS

8.1.1. Scope of tests

Reliable information can be obtained on many aspects of the solvent extraction process by batch shake-out tests. These tests essentially consist of shaking known volumes of an organic solvent and the aqueous feed solution in a separating funnel. After a few minutes of vigorous shaking the two phases are allowed to separate and one or both phases are analysed for uranium. The distribution of uranium is studied under different conditions of solution composition, organic phase composition, phase ratio and equilibration time. Visual observations are made while carrying out these tests and a record is kept of the rate of phase separation, formation of a third phase, formation of insoluble compounds, etc. This information is useful in planning and carrying out the next phase of the investigation. The aqueous feed solution required for these tests should be clear and free from visible turbidity or suspended solids. The solution can be composited from different batch leach tests. The chemical composition, pH and oxidation potential of the solution must be measured and recorded for each batch of solution used for the tests. Between 10 and 20 L of solution are required to carry out the preliminary batch tests.

8.1.2. Preparation of solvent and determination of loading capacity

The solvent is prepared by mixing 40–50 mL of a tertiary amine (Alamine-336 or equivalent) with 30–40 mL of isodecanol and making the mixture up to 1 L with kerosene. The solvent is equilibrated with 2 L of 0.1M sulphuric acid (to convert the amine into the sulphate form), and is then separated from the aqueous phase and stored.

Glass separating funnels of appropriate capacity can be used for all batch extraction tests.

8.1.2.1. Determination of maximum loading capacity

A 50 mL aliquot of the solvent is equilibrated with 50 mL of a standard uranium solution containing 2.5 g U_3O_8/L and 20–25 g/L of total sulphate at a pH of 1.5. The two phases are allowed to separate and the aqueous phase is rejected. A fresh aliquot of uranium solution is added to the partly loaded organic solvent and the extraction process repeated. This is done two more times. The loaded solvent is now stripped of uranium by contacting with 25 mL of 1M ammonium nitrate containing 0.1M nitric acid. The strip solution is collected and the stripping repeated twice with fresh aliquots of ammonium nitrate. Uranium is determined in the combined strip solutions. The uranium loading obtained in this manner, for a 5% amine solvent, should be 6–6.5 g U_3O_8/L. This test can be used to check the capacity of a solvent which has been recycled several times in extraction tests, to make sure that a marked deterioration of the extractant has not taken place.

8.1.2.2. Determination of effective maximum loading capacity

The effective maximum loading capacity of a solvent for a particular leach liquor depends on its chemical composition, particularly the uranium concentration, sulphate concentration, pH and the presence of anions such as chloride or nitrate. The loading capacity can be determined by the procedure used for the standard uranium solution except that the leach liquor is substituted for the standard solution. If different samples of leach liquor are available from preliminary leach tests, it is necessary in each case to record their chemical composition and the loading capacity of the solvent. Final tests are done on composited leach liquor pooled from different leach tests or on a solution available from a pilot scale leach test.

8.1.3. Rate of extraction

An important factor to be determined is the minimum time of contact required for effective transfer of uranium from the aqueous to the organic phase.

A 50 mL aliquot of leach liquor is shaken with an equal volume of solvent for 15 s in a 150 mL separating funnel. The two layers are allowed to separate. The aqueous phase is analysed for uranium. The equilibration is repeated for times of 30, 45, 60, 90, 120, 150 and 180 s using fresh aliquots of aqueous solution and solvent for each equilibration time. The equilibrium uranium concentration in the aqueous phase is plotted against equilibration time. The minimum time required to obtain the minimum uranium concentration in the raffinate is then found. Generally, a contact time of 30–90 s is sufficient when using a tertiary amine. An illustrative plot is shown in Fig. 24.
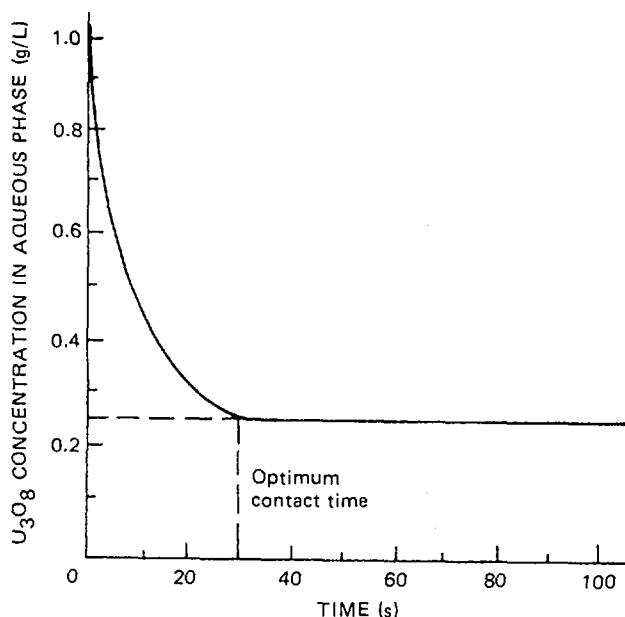
FIG. 24. *Determination of optimum contact time for uranium extraction.*

Another technique for obtaining the same information consists of agitating the aqueous and organic phases (250 mL each) together, withdrawing samples at regular time intervals between 30 and 180 s and analysing the separated aqueous phase for uranium. By drawing a plot of the uranium concentration in the aqueous phase as a function of agitation time it is possible to determine the mixing time required to reach equilibrium. The speed with which equilibrium is reached depends on the type of agitator used and the turbulence of agitation. By using a set-up of the type shown in Fig. 25 some of these parameters can be tested.

8.1.4. Extraction isotherm

A graphic representation of the variation of metal concentration in the organic phase with metal concentration in the aqueous phase is generally referred to as the extraction isotherm. For obtaining the requisite data 100 mL aliquots of the leach liquor are placed in eight separating funnels of suitable size, to which are added 10, 20, 30, 50, 100, 250, 500 and 1000 mL of solvent, respectively.

The two phases are thoroughly mixed for 2–3 min and then the two layers allowed to separate clearly. Aliquots of both layers from each funnel are analysed for uranium. The equilibrium aqueous and organic concentrations are plotted and joined by a smooth curve. A typical curve for uranium is shown in Fig. 26.
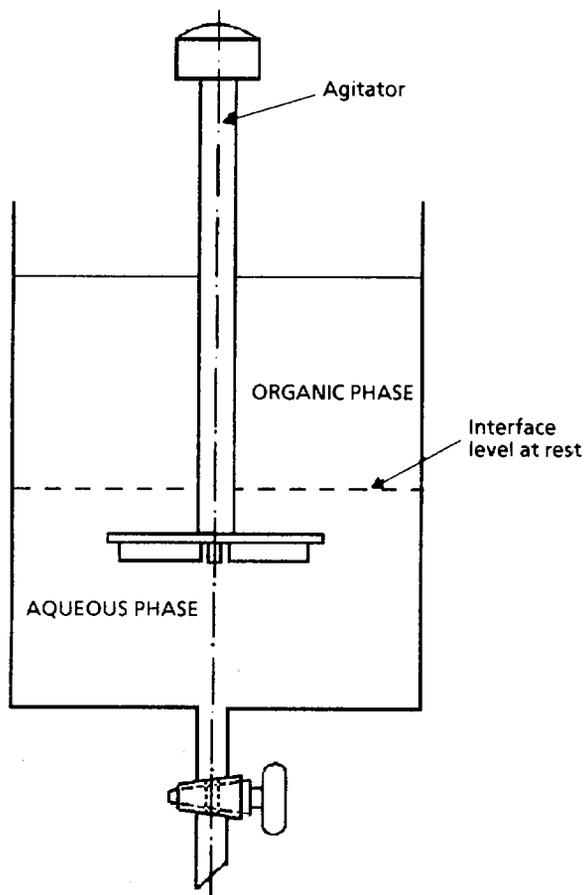
FIG. 25. *Apparatus for determination of contact time.*

Another approach is as follows. Two separating funnels (250 mL capacity) are marked A and B. In funnel A 100 mL of leach liquor are contacted with 100 mL of solvent by vigorous agitation for 2 min. The two layers are allowed to separate. The aqueous phase is transferred to funnel B. A 5 mL aliquot from each phase is withdrawn and analysed for uranium. To funnel A are added 95 mL (original volume minus aliquot withdrawn) of leach liquor and the extraction is repeated. Aliquots from the equilibrated organic and aqueous phases are again analysed. The aqueous layer is discarded and a fresh 90 mL portion of leach liquor is added and the process repeated. This sequence is repeated until the concentration of uranium in the equilibrated aqueous phase is almost the same as in the leach liquor. Meanwhile, in funnel B 95 mL of fresh organic phase are added and the extraction and analysis of the two layers carried out as for funnel A. Here, the sequence is repeated until the
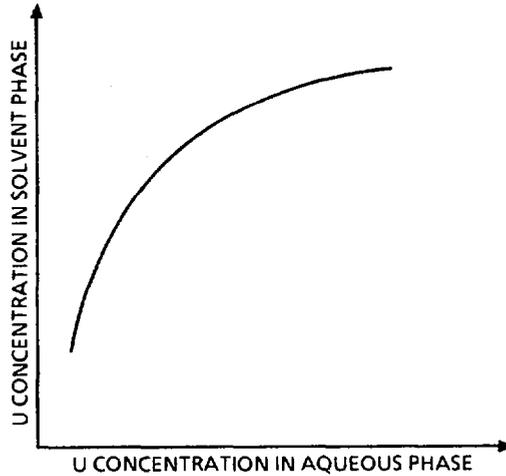
FIG. 26. Typical extraction isotherm for uranium using a 0.1M tertiary amine dissolved in kerosene.

uranium concentration in the equilibrated aqueous phase is 1–2% of the original feed concentration. Each pair of concentrations obtained in the experiment would provide data for one point for the equilibrium isotherm.

8.1.5. Stage calculations (McCabe–Thiele diagram)

The extraction isotherm can be used to prepare a McCabe–Thiele diagram for predicting the number of counter-current stages required to achieve the desired degree of extraction of uranium.

The extraction isotherm is first drawn. A second line called the 'operating line' is drawn on the same graph. The latter is a straight line drawn from the origin of the graph (as a first approximation) with a slope numerically equal to the volume ratio of the aqueous to the organic phase.

The operating line is a graphic representation of the uranium mass balance of the extraction system. A vertical line is then drawn starting from the feed uranium concentration. Where this line intersects the operating line a horizontal line is drawn to intersect the extraction isotherm. From this point of intersection a vertical line is drawn down to the operating line. This procedure of drawing lines to intersect the extraction isotherm and the operating line is continued until the desired concentration of uranium in the raffinate (about 1% of the original feed concentration) is reached. The number of stages required for achieving this can be counted. An example of the McCabe–Thiele diagram for uranium extraction using Alamine-336 is shown in

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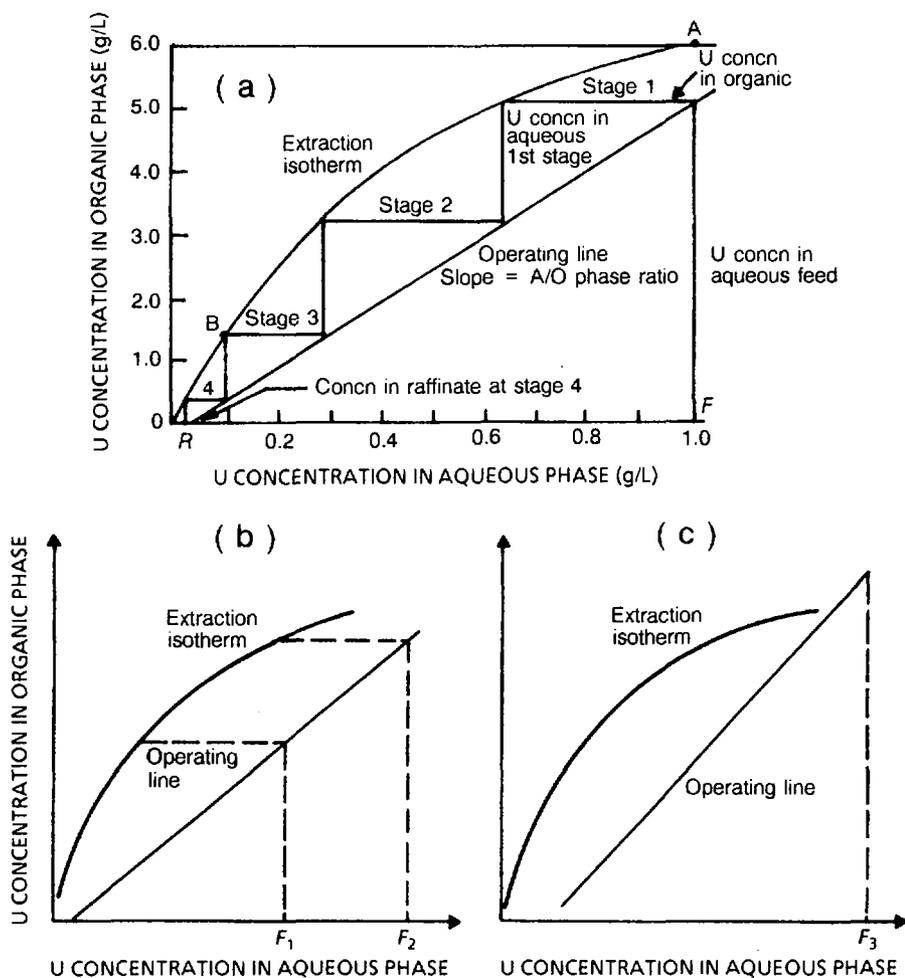
FIG. 27. (a) Typical McCabe-Thiele diagram for a four stage counter-current extraction system using Alamine-336 as extractant; (b) and (c) show operating lines for effective A/O phase ratio variations.

Fig. 27. From the diagram it can be seen that theoretically four stages of counter-current extraction are required to reach a raffinate with 0.01 g U_3O_8/L.

An adjustment must be made in the starting point of the operating line in those cases where the solvent entering the bottom stage is not completely barren. With the expected raffinate uranium value and the feed concentration known, the efficiency of the extraction can be calculated as follows: if F is the feed uranium concentration (g U_3O_8/L) and R is the raffinate uranium concentration (g U_3O_8/L), then the percentage uranium extraction is $[(F-R)/F] \times 100$.

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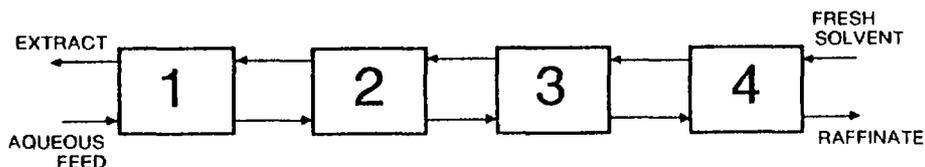
FIG. 28. Flow of aqueous and organic streams in a four stage solvent extraction system.

For different aqueous to organic phase ratios (A/O) the number of stages required for $\geq 99\%$ extraction can be calculated by drawing suitable operating lines. It is obvious that the number of stages decreases as the A/O ratio becomes smaller. However, for efficient and economical operation the extract should carry at least 80–85% of the maximum possible uranium for a given leach liquor. For example, in Fig. 27 the maximum loading capacity of the solvent, for the given feed solution, is given by A on the extraction isotherm (6 g U_3O_8/L). With A/O = 3 and a three stage operation the extract can have a uranium concentration represented by B. In this case B is a little less than 10% of A. In general, two to four stages are required for extraction. A reasonable compromise can be reached keeping in mind the number of stages required and the effective loading in the extract. The concentration of amine in the organic phase has an important bearing on these two factors. For example, if the feed concentration is much lower than F_2, say F_1, the solvent would always be underloaded (Fig. 27(b)) compared with its capacity (given by A). It is then necessary to work with an amine concentration lower than 5 vol.% in the organic phase.

The vertical line from the feed uranium concentration should intersect the operating line a little below the extraction isotherm (Fig. 27(b)) but not above it (Fig. 27(c)). In the latter case the extraction will not proceed effectively and raffinate uranium cannot be reduced to the level required. This sets an operating limit on the phase ratio A/O.

8.2. BATCH COUNTER-CURRENT EXTRACTION TESTS

8.2.1. Extraction

After determining the number of stages required for $\geq 99\%$ extraction the result can be verified by running a batch counter-current extraction using separating funnels. The system is based on the stage to stage counter-current flow of aqueous and organic streams as described below for a four stage contacting system (Fig. 28):

— In each stage (separating funnel) the aqueous and organic phases are equilibrated for a predetermined period. The two layers are allowed to separate. The separating funnel acts as a mixer as well as a settler.

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Operation 1. · Keep four empty separating funnels in positions 1,2,3,4 on a stand.

· Put a predetermined volume of organic in each funnel.

· Put a predetermined volume of aqueous in each beaker below the funnel.

· Pour the aqueous into the funnel above it.

· Mix the two layers vigorously for 2—3 min.

· Allow the two layers to separate.

· The situation is represented by (a):

(a)
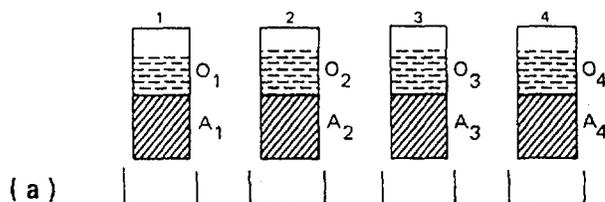
Operation 2. · Run each aqueous layer into the beaker below the funnel.

· Move each beaker one step to the right.

· The new situation is represented by (b):

(b)
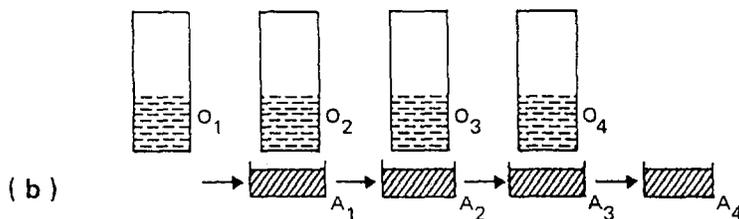
Operation 3. · Collect A_4 as raffinate and place an empty beaker under the funnel in position 1.

· Add fresh aqueous to this beaker.

· The new situation is represented by (c):

(c)

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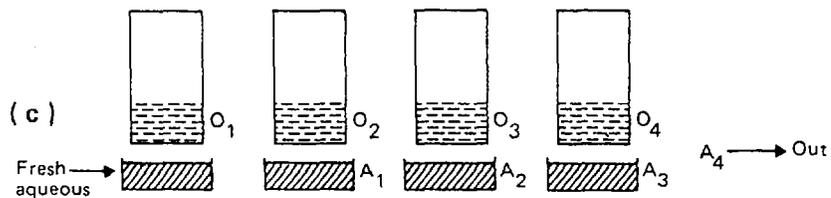
Operation 4. · Move each funnel one step to the left, keeping the beakers in the same positions as in (c).

· The new situation is represented by (d):

(d)

Fresh ⟶ aqueous

O_1 O_2 O_3 O_4

A_1 A_2 A_3
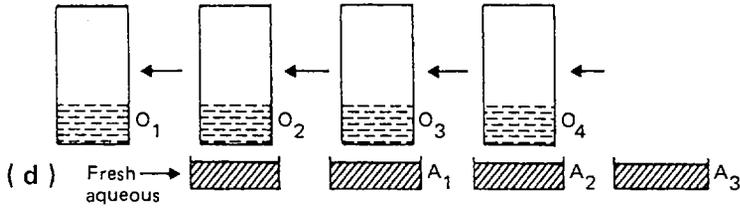
Operation 5. · Collect organic O_1 as extract and place empty funnel in position 4.

· Add fresh organic to this funnel.

· The new situation is represented by (e):

Out ⟵ O_1

(e)

O_2 O_3 O_4

Fresh ⟵ organic

Fresh ⟶ aqueous

A_1 A_2 A_3
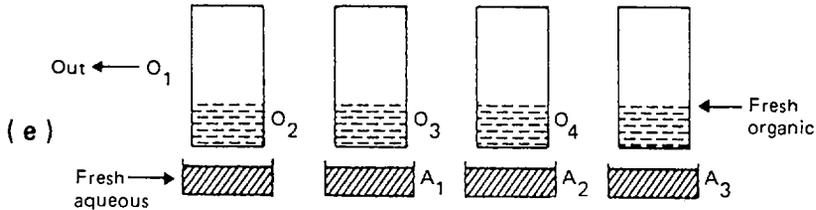
Operation 6. · Pour the aqueous from each beaker into the funnel above it.

· Mix the two layers vigorously for 2—3 min.

· Allow the two layers to separate.

· The situation is similar to that in (a).

· Repeat the cycle of operations 2—6 at least twice.

FIG. 29. Simulation of a four stage counter-current extraction.

— The aqueous layer is then moved a stage to the right (say from 2 to 3) while the organic moves a stage to the left (say from 4 to 3). Therefore, in any given stage (say 3) the aqueous from the left (say 2) and the organic from the right (say 4) are received and equilibrated.

— Fresh aqueous phase enters stage 1 while fresh solvent enters stage 4. The loaded solvent leaves stage 1 and the raffinate leaves stage 4.

There are several ways of simulating the counter-current flow in a batch system of this type. A simple method for laboratory operation is shown in Fig. 29.

The phase ratio A/O and the number of stages to be used in this test are determined by the procedure described in Section 8.1.5.

In the beginning the raffinates and extracts collected in the counter-current extraction system show a variation in uranium concentration. However, after several cycles of operation equilibrium is attained and the raffinate as well as the extract concentration stabilizes and tends to become more or less constant. From that stage the extraction can be run endlessly. By combining a sufficiently large number of extracts a composite is made that can be used to study the stripping behaviour and determine the phase ratio and number of stages required for effective stripping.

8.2.2. Stripping of uranium from loaded solvent

The stripping process is the opposite of extraction in the sense that uranium is now brought back to the aqueous phase using a suitable chemical reagent. The strip solution obtained is suitable for recovery of uranium concentrate (yellow cake) by direct precipitation.

Several reagents can be used to strip uranium from the loaded solvent. These are NaCl, Na_2CO_3, $(NH_4)_2CO_3$, $(NH_4)_2SO_4$ + NH_4OH at controlled pH, NH_4NO_3, H_2SO_4 and MgO slurry.

The most common stripping agent is 1.0M NaCl + 0.05M H_2SO_4. Using a composite loaded solvent obtained from batch counter-current extraction tests, the parameters for stripping can be fixed by a procedure similar to that employed for extraction. The steps are:

(a) *Determination of the time required for equilibration:* in the case of tertiary amines it is usually in the range of 60–90 s.
(b) *Stripping isotherm:* this can be constructed in the same way as the extraction isotherm. A typical graph is shown in Fig. 30.
(c) *Determination of the number of stages:* this is done using the McCabe–Thiele diagram.

If coupled with batch counter-current extraction the stripping system (consisting usually of three stages) would yield a strong uranium strip solution. The combined system can be represented as shown in Fig. 31.

Under favourable conditions starting with a leach liquor containing 1–1.5 g U_3O_8/L it is possible to obtain a final product solution with 20–30 g U_3O_8/L.

8.3. CONTINUOUS EXTRACTION TESTS

In the uranium industry it is a general practice to use mixer–settler assemblies for continuous counter-current extraction. Though multistage operation of this type can be simulated in the laboratory using separating funnels, it is generally a time con-
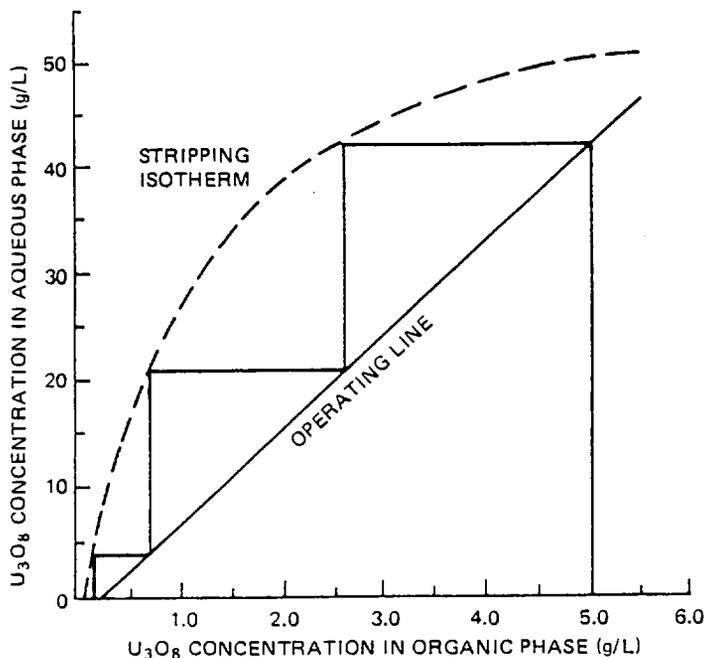
FIG. 30. *Typical McCabe-Thiele diagram for stripping uranium in three stages from loaded organic.*

suming operation. However, it has the advantage of simplicity and can be done without special equipment. After carrying out such tests it is advisable to procure or fabricate a bank of small scale mixer–settlers. This unit can be used to obtain a regular supply of strong uranium strip solution for precipitation tests. By recycling the solvent several times, it is possible to obtain data on solvent performance, including phase separation and solvent degradation, buildup of metals, such as molybdenum, and the required frequency of solvent regeneration.

A typical mixer–settler unit is shown in Fig. 32 and consists of a compartment for mixing the leach liquor and the extractant for a desired duration of time, and a settling compartment wherein the mixed emulsion phase is allowed to separate completely prior to the movement further of the two phases in counter-current flow. The optimum duration of mixing would have been determined experimentally. A separate experimental determination would also have been made of the time required for phase separation. The phase separation takes place in two steps. The first is the 'primary break', where the bulk of the mixed phase separates by a process of coalescence and settles into two phases. The primary break determines the actual size of the settling compartment. This is followed by the gradual disappearance of fine droplets, which is the 'secondary break'. If the secondary break is not complete, the

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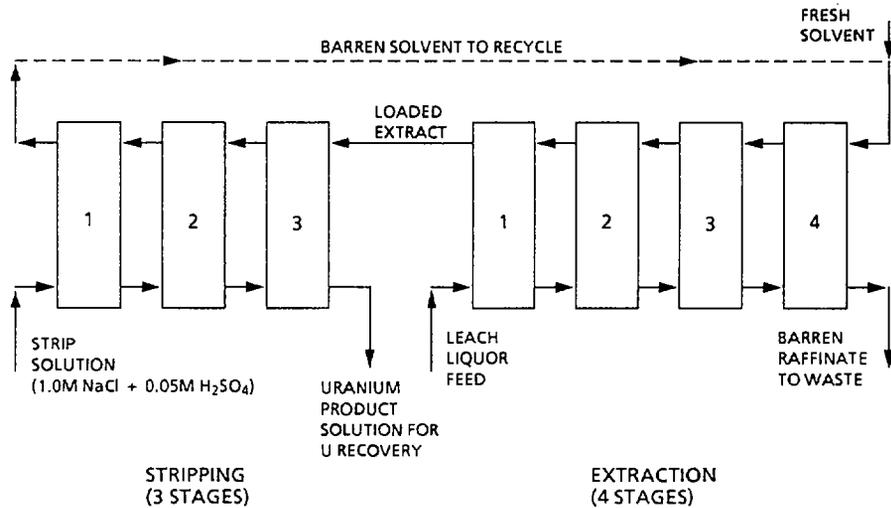
FIG. 31. Combined extraction and stripping system.
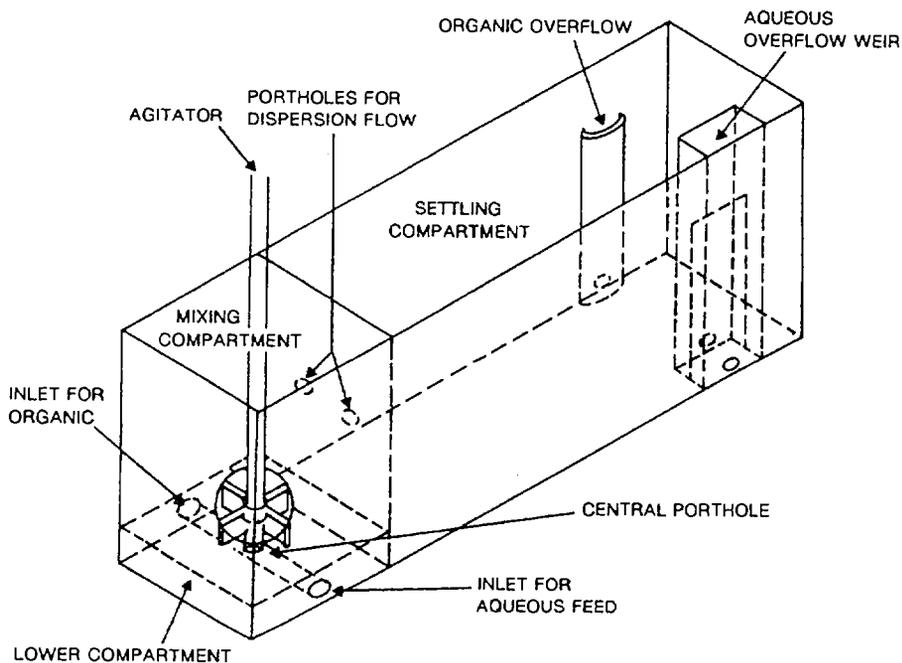
FIG. 32. Typical box type mixer–settler unit.

fine aqueous/solvent drops will be carried to the next stage. Though this would not affect the uranium extraction appreciably, the solvent droplets leaving the system along with the raffinate would cause high solvent loss. Hence, in industry it is a general practice to provide large tanks, called 'after-settlers', to recover this solvent loss.

8.3.1. Determination of coalescence rate and settler area

8.3.1.1. Static method

In the static method, 300 mL of leach liquor and 60 mL of amine solution are thoroughly mixed for 2 min in a 500 mL stoppered graduated cylinder. The phases are allowed to separate. The heights of the top and bottom edges of the dispersion band are noted at various times and the values plotted as shown in Fig. 33. The point where the two curves meet the time axis indicates the time required for the dispersion to separate completely. With the surface area of the cylinder known, the flow rate of dispersion which can be tolerated per unit area of the settler can be calculated as shown in Fig. 33.

8.3.1.2. Dynamic method

The dynamic method employs a small single stage mixer–settler unit with facilities for continuous feed of solvent and leach liquor into the mixing compartment. A typical arrangement is shown in Fig. 34.

The dispersion band is measured at different rates of flow of the two phases and the thickness of the band is plotted as a function of flow rate. Generally, the thickness of the dispersion band varies with the logarithm of the volume of the dispersed phase. For practical purposes, the flow which results in a band thickness of 10–12 cm is taken as the design capacity of the settler. It should be remembered that several parameters such as the degree of mixing in the mixer vessel, the pH of the solution and the phase ratio influence the settling characteristics of the dispersion. For uranium extraction from leach liquors with amines, the generally accepted requirement is about 50 L/min per square metre of settler area.

8.3.2. Characterization of dispersion

As mentioned earlier, the phase separation behaviour of the dispersion depends on the quality of mixing in the mixer compartment. There are two types of dispersion: the dispersion of aqueous droplets in the solvent phase and the dispersion of organic droplets in the aqueous phase. The first is known as 'organic continuous' and the second as 'aqueous continuous' dispersion. The organic continuous dispersion

The graph shows:
- Y-axis (top): HEIGHT OF SEPARATED ORGANIC PHASE (cm), with values 0, 0.5, 1.0, 1.5, 2.0, 2.5, 3.0, 3.5, 4.0
- Y-axis (bottom): HEIGHT OF SEPARATED AQUEOUS PHASE (cm), with values 0.5, 1.0, 1.5, 2.0, 2.5
- Left axis label: Dispersion band
- X-axis: TIME (s), with values 10, 20, 30, 40, 50, 60, 70
- Active interphase
- t
- h

Surface area of settler $= a$ cm^2

Height of separated phase $= h$ cm

Volume of dispersion settled in t seconds $= ha$ cm^3

Volume of dispersion which can settle per minute per a cm^2 $= \dfrac{ha \times 60}{t} cm^3$

Settling rate $= \dfrac{ha \times 60 \times 10^4}{ta \times 10^3} L \cdot min^{-1} \cdot m^{-2}$

$= 600 \dfrac{h}{t} L \cdot min^{-1} \cdot m^{-2}$

FIG. 33. Determination of coalescence rate by static method.

facilitates quick separation and is preferred for reducing the solvent losses in the raffinate. If the mixing compartment contains a proportion of solvent that is equal to or greater than that of the aqueous solution, this would help in obtaining such a dispersion. But in practice, to upgrade uranium concentration the solvent phase is always smaller (A/O $\geq$ 4). Hence, to obtain a higher solvent volume, part of the solvent phase which has separated in the settler is recycled to the mixing compart-
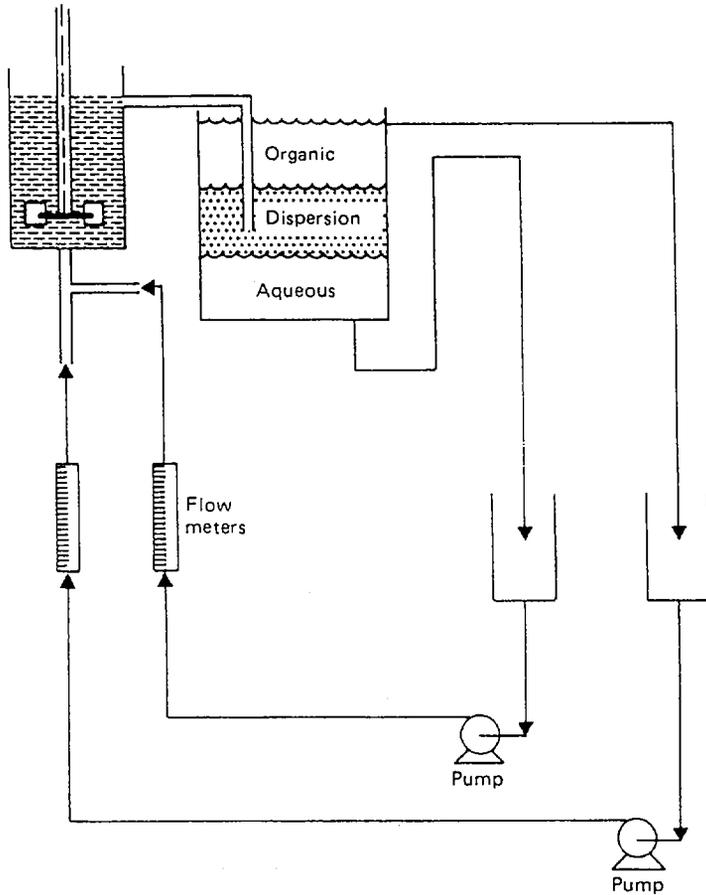
FIG. 34. Single stage mixer–settler assembly for phase separation tests.

ment to maintain a phase ratio of A/O = 1. Though this adds to the load of the agitator and the settler, as long as it does not exceed 50% of the design capacity it does not cause a problem.

The type of dispersion formed can be identified in the laboratory by a simple device involving the measurement of conductivity. A milliammeter is connected to a flashlight battery such that when the two terminal probes are immersed in the mixing compartment the circuit is completed. If the dispersion is organic continuous it will be non-conducting and the bulb will not glow, whereas if it is aqueous continuous the current will flow and the bulb will light up.

8.3.3. Solvent loss

The raffinate leaving the final settler would carry entrained as well as dissolved organic phase. The amines normally used in the uranium industry dissolve to an extent of 10–20 ppm. Entrainment losses depend on equipment performance and extraction parameters and are variable.

To determine the total solvent loss (dissolved and entrained) 0.5 L of the barren raffinate flowing out of the extraction system is collected and contacted in a separating funnel with 50 mL of a solvent such as heptane for about 5 min. The phases are allowed to separate completely. The aqueous phase is discarded and the solvent phase, which would now contain all the entrained amine solution, is utilized for estimating the amine content by a suitable standard analytical procedure.

For determining the dissolved amine content, the raffinate is filtered through a Whatman No. 1 PS phase separation paper and the clear aqueous solution is utilized for amine estimation by a spectrophotometric method.

9. PRECIPITATION

9.1. SOLUTION CHARACTERIZATION

The final uranium solutions may be obtained by different methods, in most cases by the selective ion exchange or solvent extraction processes. In spite of the high selectivity of these processes some impurities may be coextracted with the uranium. The majority of methods for the precipitation of the final product are relatively non-selective but they are very efficient procedures with high recovery (more than 99%). Thus, on the basis of the chemical analysis of the final solution of uranium the quality of the final product may be predicted. The solution should be analysed for uranium, major constituents such as chloride and sulphate and impurities, e.g. iron, vanadium, molybdenum, phosphate, fluoride and thorium, as expected from the ore composition and process selected. If the relative concentrations of the solution components are close to the standards for concentrate purity (Table XI) the concentrate will probably also meet the standards. In such a case precipitation with ammonia or magnesium oxide may be considered as the appropriate method. If vanadium and molybdenum in the final solution exceed the standards the precipitation of uranium peroxide may be considered as a more selective procedure.

Basically, in the test procedures for different ore treatment steps some tens of kilograms of the ore are treated. The amount of uranium which eventually may be precipitated is insufficient to allow adequate testing for the determination of thickening rates and filterability.

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TABLE XI. TYPICAL SPECIFICATIONS FOR URANIUM CONCENTRATES

Minimum U_3O_8 content: 65 wt%
Maximum impurities permissible (wt% based on U_3O_8 content):

Na	7.5
H_2O	5.0
SO_4	3.5
K	3.0
Th	2.0
Fe	1.0
Ca	1.0
Si	1.0
CO_3	0.5
Mg	0.5
Zr	0.5
PO_4	0.35
Halogens (Cl, Br, I)	0.25
V_2O_5	0.23
Rare earths	0.2
P	0.15
Mo	0.15
As	0.1
B	0.1
Extractable organic matter	0.1
Insoluble uranium	0.1
Ti	0.05
Ra-226	20 000 pCi/g[a]
Particle size	0.25 in (6.35 mm)

Note: Only natural uranium concentrates (non-irradiated material) containing 0.711% of the isotope U-235 are acceptable.

[a] 1 Ci = 37 GBq.

Even when a significant amount of the final solution of uranium (say 1 L) is prepared it is very difficult to simulate in small equipment the results which are expected in a continuous commercial operation. Consequently, the precipitation of uranium from final solutions obtained by laboratory ore testing may only give preliminary results. In particular, the physical properties of the precipitates obtained in the laboratory may be significantly different from those of precipitates in the full scale plant.

9.2. PRECIPITATION FROM ACIDIC SOLUTIONS

9.2.1. Polyuranates

The most common method of precipitation of uranium from purified acidic solutions is neutralization by ammonia to pH7–8. Other neutralizing agents may be used too, e.g. NaOH or MgO. The major advantages of ammonia are its easy handling and the fact that the process can be regulated by mixing another gas with ammonia. There is the further advantage that calcination of the product removes ammonia, resulting in a higher grade product. Generally, the product obtained is a mixture of hydrated oxides, polyuranates and basic uranyl sulphates.

The precipitation is done by adding a mixture of air or nitrogen and ammonia gas to a stirred beaker of uranium solution. This avoids high ammonia concentration gradients in the solution and also prevents the solution from being sucked into the gas inlet pipe, valve, etc., when the flow of ammonia is stopped. If gaseous ammonia is not available, a dilute ammonium hydroxide solution may be used (1–5 wt% NH_3 in water). The solution should be added slowly (dropwise) from a burette. Ammonia should be added until a pH between 7 and 8 has been reached. The temperature range of precipitation is from 40 to 60°C. The product is then filtered, washed and dried at 105 to 200°C. The apparatus is shown in Fig. 35.

9.2.2. Uranium peroxide

Uranium may be precipitated from acidic solutions by means of hydrogen peroxide as shown by the following equation:

$$UO_2^{2+} + H_2O_2 + 2H_2O \longrightarrow UO_4 \cdot 2H_2O + 2H^+$$
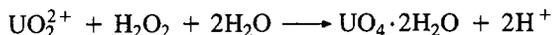
This method is more selective than neutralization but requires more attention.

The apparatus is the same as shown in Fig. 35. Ammonia is still required for the neutralization of the acid formed (see equation). Hydrogen peroxide (30 volumes) should be added in excess (35 to 100%) over stoichiometric. The temperature should be lower than for neutralization and is kept at 25°C approximately.

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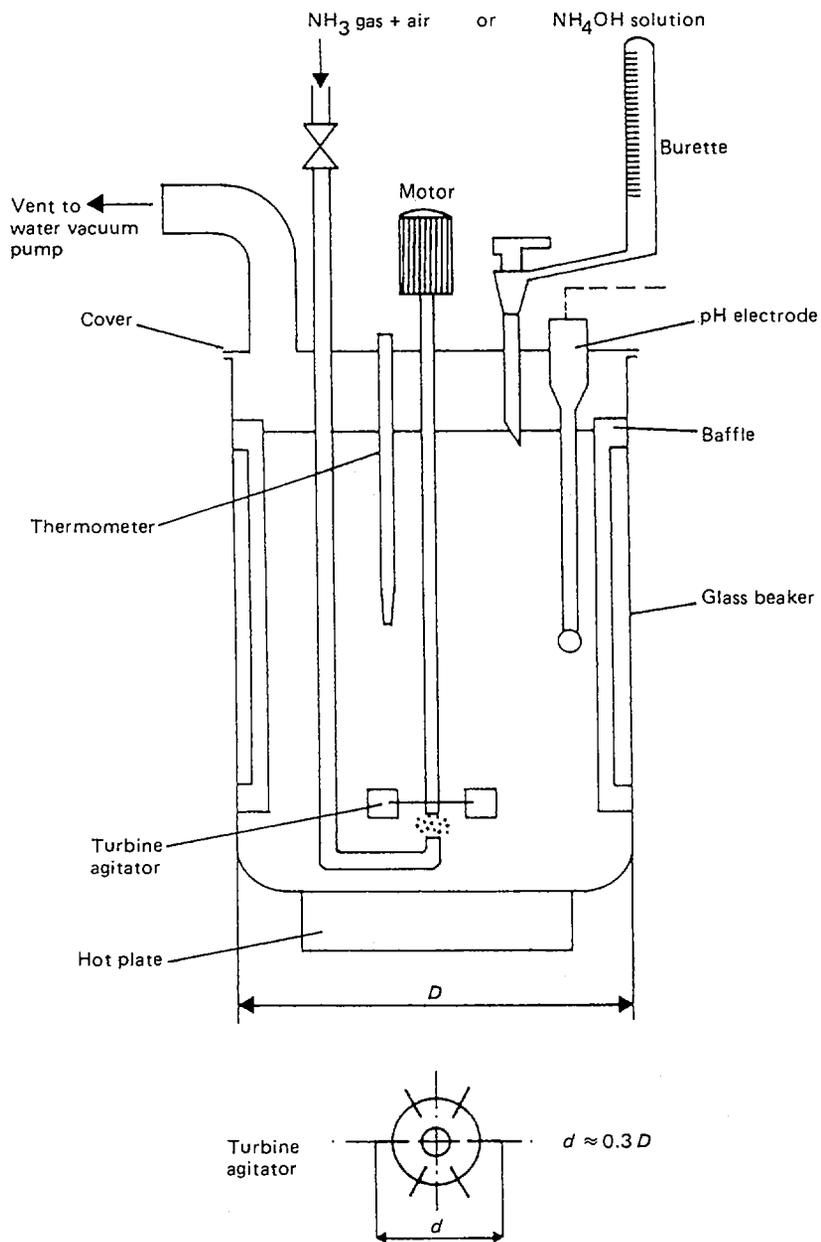
FIG. 35. Apparatus for uranium precipitation.

TABLE XII. SODIUM DIURANATE PRECIPITATION:
OPERATING PRACTICE

	Direct precipitation from alkaline leach solution	Precipitation from carbonate strip solution
Temperature (°C)	50–80	20–30
Digestion time (h)	6–12	2–4
Free NaOH (g/L)	4–6	4–6
NaOH addition (kg NaOH/kg U_3O_8)	5–6	3–4

Dilute solutions of uranium do not precipitate completely and may require a longer induction period before the precipitation occurs (sometimes many hours). Excessive concentrations of sulphate ion lower the efficiency of the precipitation.

9.2.3. Other methods

Other methods for the precipitation of tetravalent uranium include electrolytic or SO_2 reduction followed by the precipitation of $UF_4 \cdot xH_2O$ with HF. This procedure is very seldom used and is rather complicated for laboratory work.

9.3. PRECIPITATION FROM CARBONATE SOLUTIONS

The practices for precipitating sodium diuranate from alkaline leach liquors and sodium carbonate strip solutions differ greatly, as shown in Table XII. Precipitation from leach liquors is performed at elevated temperatures (50–80°C) for fairly extended periods (6–12 h). Much of the precipitate is recycled to maximize the surface area for precipitate growth. Precipitation from strip solutions is done at ambient temperature for fairly short retention times (2–4 h). The terminating precipitation conditions are similar for both procedures. The primary control is the sodium hydroxide excess (4–6 g NaOH/L). Because the leach liquors are more dilute, precipitation from these solutions requires more sodium hydroxide (5–6 kg NaOH/kg U_3O_8) than does precipitation from carbonate strip solutions (3–4 kg NaOH/kg U_3O_8).

In sodium carbonate leach systems, it is more important to conserve the reagent for recycle than to achieve almost complete precipitation; therefore, the preferred method is to precipitate with the addition of excess sodium hydroxide.

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Shown below are the caustic reactions for uranium precipitation in an alkaline circuit and then for regenerating the solutions for recycle:

$$NaHCO_3 + NaOH \rightleftharpoons Na_2CO_3 + H_2O$$

$$2Na_4UO_2(CO_3)_3 + 6NaOH \rightleftharpoons Na_2U_2O_7 + 6Na_2CO_3 + 3H_2O$$

$$2NaOH + CO_2 \rightleftharpoons Na_2CO_3 + H_2O$$

$$Na_2CO_3 + CO_2 + H_2O \rightleftharpoons 2NaHCO_3$$

$$6NaHCO_3 + Na_2U_2O_7 \rightleftharpoons 2Na_4UO_2(CO_3)_3 + 3H_2O$$
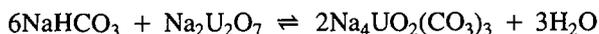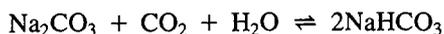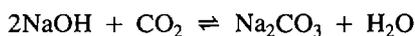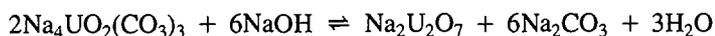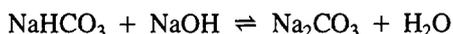
Caustic addition, as indicated in the first two equations, first converts any bicarbonate present to carbonate and then brings about a reaction with sodium uranyl tricarbonate to precipitate the uranium as sodium diuranate or more probably as polyuranates.

After uranium precipitation, the solutions are regenerated by contacting them in packed or tray type towers with CO_2 gas, as shown in the third and fourth equations. Excess sodium hydroxide is converted to carbonate or carbonate to bicarbonate by this method. Carbonation is carried out only to the extent necessary to produce the desired ratio of sodium carbonate to bicarbonate in solution. The final equation shows how bicarbonate will react to reconvert any residual sodium diuranate back to the uranyl tricarbonate complex before the regenerated solution is recycled for use as wash solution in the solid–liquid separation step.

When treating sodium carbonate strip solutions it is important to obtain almost complete uranium recoveries. In this case the solutions are acidified, boiled to expel CO_2 and then neutralized to precipitate the uranium.

10. ALTERNATIVE TECHNIQUES

Several alternative techniques can be used to supplement or replace the conventional hydrometallurgical processes briefly described in Section 1 when the latter do not yield adequate efficiencies or are uneconomical. The main alternative techniques are physical beneficiation, roasting, static bed leaching, bacterially assisted leaching, acid pugging and acidic pressure leaching.

10.1. PHYSICAL BENEFICIATION

The purpose of physical beneficiation of uranium bearing ores can be either to concentrate the uranium mineral in a smaller weight fraction or to eliminate

reagent consuming minerals such as sulphides prior to carbonate leaching. Three methods which have been investigated for physical beneficiation are gravity concentration, flotation and radiometric sorting. Generally, the objective of physical beneficiation is to eliminate the greatest weight of run of mine ore with the lowest uranium content.

10.1.1. Gravity concentration

Sink–float tests are used to determine whether an ore is amenable to gravity concentration. This method uses heavy liquids such as tetrabromoethane (specific gravity 4.5) mixed with carbon tetrachloride (specific gravity 0.89) to give the desired specific gravity. For example, if the gangue rock has a specific gravity of 2.65, then the heavy liquid is adjusted (usually by adding carbon tetrachloride to tetrabromoethane) to a specific gravity of 2.85 and then to 2.75. It is important to note that these two organic liquids are very toxic and all work should be carried out in a properly ventilated fume hood. The float fraction of the first heavy liquid test (at specific gravity 2.85) is removed usually with a perforated scoop and then is retreated at a specific gravity of 2.75. The fractions are washed (usually with carbon tetrachloride to remove the more expensive tetrabromoethane) and dried on a hot plate in a ventilated fume hood.

Heavy liquid tests are normally done on the $-8 + 35$ mesh fraction of a 3000 g ore sample crushed to -0.5 in $(-12.7$ mm). The -35 mesh fraction may be further screened to 65, 100 and 200 mesh. These fractions are usually treated on a shaking table or panner to yield a gravity concentrate, middling product and a reject.

A typical sink–float test result is shown in Table XIII. Thus, 92.8% of the uranium can be recovered at a specific gravity of 2.75 while 72.7 wt% of the fraction is rejected as waste. An example of the results of a shaking table or panner test on a finer fraction of the ore (-35 mesh) is shown in Table XIV. In this case 54% of the uranium is recovered in less than 2% of the weight while over 98 wt% of the fraction is rejected, but with 46% of the contained uranium.

TABLE XIII. TYPICAL SINK–FLOAT TEST RESULT

Product	wt% of feed	wt% U_3O_8	U_3O_8 distribution (%)
Sink at SG 2.85	16.5	0.80	87.1
Sink at SG 2.75	10.8	0.08	5.7
Float	72.7	0.015	7.2

TABLE XIV. EXAMPLE OF A SHAKING TABLE OR PANNER TEST ON A FINER FRACTION OF THE ORE (−35 MESH)

Product	wt% of feed	wt% U_3O_8	U_3O_8 distribution (%)
Gravity concentrate	0.4	50.6	45.1
Middling product	1.3	3.0	8.9
Reject	98.3	0.2	46.0

The general objective of gravity concentration is to reject at least 50 wt% of the ore fraction but lose less than 10% of the uranium.

10.1.2. Flotation

Flotation of uranium ores is normally employed to eliminate gangue minerals which consume leaching reagents. For example, in alkaline leaching with sodium carbonate, sulphides are the main consumer of the leaching reagent. Sulphides may be floated with various xanthates and frothers, as in the base metal industry. Carbonates are the main consumers of acid in acid leaching. It is possible to float carbonates from uranium ores with combinations of oleic acid and frother. In general, the reagent consuming minerals which are removed contain significant amounts of uranium mineral and, therefore, some method must be adopted to recover the uranium from these fractions.

10.1.3. Radiometric sorting

Radiometric sorting of uranium ore is based on the detection of gamma radioactivity from individual pieces of ore and waste. Sorters operate by conveying coarse ore (usually +1.5 in (38.1 mm)) over a sensor which allows the uranium content to be calculated rapidly and then causes a fine high pressure air stream to impel the rock as it discharges over the belt to the sorter bins below (Fig. 36).

Large amounts of ore sample are usually required for radiometric sorting tests. A 10 t sample crushed to −6 in (−152.4 mm) is screened to +3 in (76.2 mm), −3 + 1.5 in (38.1 mm) and −1.5 in. Each fraction must be washed with water to remove slimes which could coat the waste rock and give erroneous radioactivity readings.

Each fraction is passed through the ore sorter and the sensitivity of the detector set to reject particles containing less than 10% of the head assay. If the ore is estimated to be of grade 0.2% U then the sorter is adjusted to reject as waste any

Lead
shielding

Video unit

Blast
manifold

Gamma
detector

Splitter
plate

Ore   Waste

Blast
valves

High pressure
air source

Logic
unit

Feed bin and
vibrating feeders

Belt

External
displays
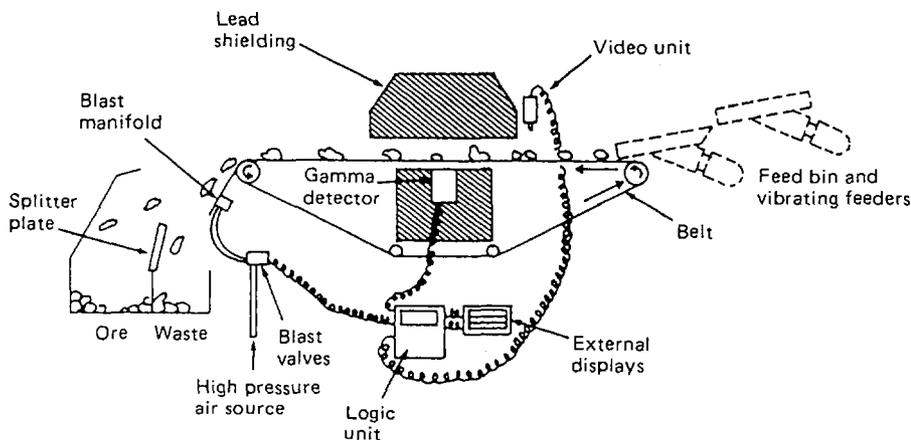
FIG. 36. Radiometric sorter.

material below 0.02 % U. Each fraction is weighed, sampled and analysed. A typical promising material balance is shown in Table XV. A preliminary test result as shown in Table XV would indicate that further improvements could be made, particularly on the -1.5 in to $+1.5$ in fraction. Manufacturers of radiometric sorting equipment have made many improvements to these units and should be contacted for the latest information.

10.2. ROASTING

Pre-leach roasting techniques have been used to improve the chemical processing characteristics of uranium ores and also to improve the settling and filtering characteristics of some ores. During the late 1950s and the 1960s, hydrometallurgical techniques were developed that achieved the same objectives, and these procedures have almost entirely replaced the roasting procedures. Both Merritt [3, pp. 103–108] and Clegg and Foley [16] discuss the roasting techniques that have been used on uranium ores. Roasting procedures have been used primarily for the following purposes:

(a) *To form soluble vanadium and uranium compounds by salt-roasting carnotite ores*. This approach has largely been replaced by hydrometallurgical processing in which the uranium is extracted first by a relatively mild acid leach and then the vanadium by a more severe, hot acid leach. The uranium is recovered from solution by either ion exchange or solvent extraction and the vanadium is then recovered by solvent extraction.

(b) *To remove carbonaceous matter from ores before leaching*. Uranium is commonly associated with carbonaceous materials; notable examples are

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TABLE XV. MATERIAL BALANCE

Size range (in)	Feed		Reject		Concentrate		Recovery (%)
	wt%	wt% U_3O_8	wt%	wt% U_3O_8	wt%	wt% U_3O_8	
+3	36.8	0.154	42.8	0.015	57.2	0.258	95.8
−3 + 1.5	11.7	0.197	39.1	0.036	60.9	0.300	92.8
−1.5 (not treated)	51.5	0.276	—	—	—	—	—
Overall	100.0	0.206	20.3	0.020	79.7	0.254	98.1

shales, lignites and asphaltic sandstone ores. Roasting these materials in the 500–600°C temperature range usually both increases the uranium extraction and reduces reagent requirements. This temperature range is usually quite critical; at temperatures both above and below this range, the uranium extraction decreases significantly. Even though roasting is a relatively expensive operation and is not practised at present, it is advisable to include at least a few roasting tests in an ore evaluation and process development programme. This would be particularly appropriate for higher grade ores.

(c) *To improve settling and filtration characteristics.* Roasting in the 300–600°C temperature range has been found to improve significantly the settling and filtering characteristics of ores containing finely divided clay minerals. This advantage, however, has been offset by the development of effective flocculants and resin in pulp ion exchange systems, which have lower capital and operating costs. Even though the overall economic potential for improving physical characteristics is small, a few exploratory tests are recommended.

The most logical application for roasting would probably be in the case of a high grade ore or concentrate containing an appreciable amount of carbonaceous uranium bearing material. Even under these circumstances, however, careful attention should be paid to the potential environmental problems associated with a roasting operation.

Laboratory roasting tests are usually carried out in shallow rectangular fireclay roasting dishes. If the bed depth is greater than 10–15 mm, rabbling of the charge during the roast period is often necessary to ensure complete oxidation. This is particularly true if the material contains more than 10 wt% organic carbon. It is usually best to place the charge in the furnace when the furnace temperature is at least 200–300°C below the desired roasting temperature and then to bring the furnace to temperature. This helps to simulate the heat-up profile that would occur in

a kiln or multiple hearth roaster. After roasting, both air cooling and quenching of the hot calcine into the leaching solution should be investigated. Quenching into the leaching solution can sometimes significantly improve the leach extraction.

10.3. STATIC BED LEACHING

A static bed leaching system has been defined as any leaching system in which the leaching solutions are passed through a stationary bed of ore. Terms such as in situ leaching, in-place leaching, heap leaching and percolation leaching have been used to denote various modifications of the static bed leaching practice. To some degree, these terms have been used interchangeably. In the 1983 OECD/NEA–IAEA joint report on uranium extraction technology [1, pp. 62, 63] the following definitions were proposed for these terms:

(a) *In situ leaching:* The ore is not moved from its geological setting. The leaching solutions are forced through the bed, usually in a horizontal direction. Most unconsolidated sandstones can be leached in situ, 'as is', without fragmentation. For other materials, various types of pre-leach fracturing have been considered.

(b) *In-place leaching:* The ore is broken by blasting but left in underground stopes. The leaching solutions may be passed through the static ore bed in a downward direction or the stope may be at least partially filled with the leaching solution. The leaching solutions are recovered by a collection system below the bed of broken ore.

(c) *Heap leaching:* The ore is mined and then piled over a collection system. The leaching solutions are distributed over the top surface of the heap and pass downwards through the bed of ore. The bed may be run of mine ore or coarse crushed material.

(d) *Percolation leaching:* The mined ore is finely crushed (normally to about −1 in (−25.4 mm) or less) and then bedded into tanks or vats. The leaching solutions are passed through the static ore in either an upward or a downward direction. Various types of pre-leach treatment, such as pelletizing, have also been practised.

Many variables can affect the results obtained from a static bed leaching operation, but the most important variable is the solution distribution within the ore bed. More difficulties have probably been encountered because of inadequate solution distribution than from any other single factor.

Some types of static bed leaching have been practised in the hydrometallurgical industry for many years, but in general the techniques achieved only scattered use in the uranium industry. Recently, however, a combination of economic and

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environmental factors have produced a revived interest. The complex subject of simulating static bed leaching in the laboratory is beyond the scope of this manual; the reader is referred to the background information presented in the Bibliography.

10.4. BACTERIALLY ASSISTED LEACHING

If a uranium ore contains sulphide minerals, bacterially assisted leaching may be possible. Under the proper conditions, autotrophic bacteria such as *Thiobacillus ferrooxidans* attack the sulphide minerals to produce an acidic ferric sulphate lixiviant capable of extracting reduced uranium minerals. The complex microbial and chemical reactions that take place in such a system where pyrite is present include the following:

(a)   Bacterial oxidation of pyrite, producing ferric sulphate and sulphuric acid:

$$4FeS_2 + 2H_2O + 15O_2 \longrightarrow 2Fe_2(SO_4)_3 + 2H_2SO_4$$

(b)   Chemical oxidation of pyrite by ferric sulphate:

$$FeS_2 + 7Fe_2(SO_4)_3 + 8H_2O \longrightarrow 15FeSO_4 + 8H_2SO_4$$
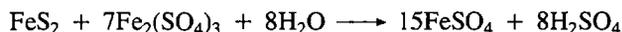
(c)   Chemical oxidation of uranium from the tetravalent to the hexavalent state by ferric sulphate:

$$UO_2 + Fe_2(SO_4)_3 \longrightarrow UO_2SO_4 + 2FeSO_4$$
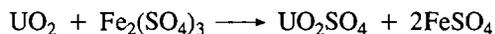
(d)   Bacterial oxidation of ferrous to ferric sulphate:

$$4FeSO_4 + 2H_2SO_4 + O_2 \longrightarrow 2Fe_2(SO_4)_3 + 2H_2O$$
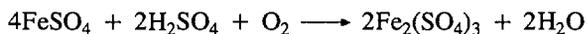
One of the major difficulties is establishing conditions for carrying out these reactions simultaneously at their optimal rates. Under almost all conditions, there is normally a lag of at least 72 h after the bacteria are introduced before oxidation commences. Researchers have also found that small amounts of various inorganic salts must be available as nutrients. The optimal concentrations of these nutrients (NH_4^-, Ca^{2+}, K^+, Mg^{2+}, SO_4^{2-}, PO_4^{3-}, etc.) may be different for each leaching system.

Bacterially assisted leaching is difficult to simulate in laboratory scale test work; most successful studies have been in the small to medium scale pilot plant category. For additional information on the complex subject of bacterially assisted leaching the reader is referred to the Bibliography.

10.5. ACID PUGGING

Acid pugging is the term most often used to designate the following sequence of operations:

(1) Dampened ore is pugged with concentrated sulphuric acid and an oxidant if required.
(2) The pugged mixture is cured by keeping it at constant temperature (often 90 to 100°C) for at least 2 h.
(3) The cured mixture is then leached with water. Agitation leaching is most often used and the contact time is usually less than 2 h.

Acid pugging should be considered if high terminal free acid concentrations (>10 g/L) are required for satisfactory uranium extractions. This situation can occur with refractory uranium minerals and also when the uranium is associated with organic materials or even some types of clay. The SOMAIR and COMINAK operations in Niger are the principal examples of acid pugging mill circuits [17].

In general the primary variables that must be considered for acid pugging are:

— *Particle size:* The grind at the operations in Niger is about 95% -630 μm, but each ore will require individual optimization studies.
— *Moisture content of feed ore:* Moisture contents in the range of 8–10% are often optimal.
— *Quantity of concentrated acid added:* The SOMAIR operation is reported to use about 90 kg/t, but every ore will require individual optimization studies.
— *Oxidant requirements:* $NaClO_3$ and HNO_3 are both used as oxidants in the Niger operations.
— *Curing time and temperature:* These variables are usually interdependent.

Exploratory acid curing tests can often be carried out by placing 200–300 g of dry ore in a heavy duty beaker and adjusting the moisture content to the desired level. The ore and the water should be thoroughly blended. The desired amount of concentrated sulphuric acid is then slowly added while the ore–acid mixture is continuously pugged with a 316 SS spatula or similar implement. A watch glass is then placed over the beaker and the beaker and its contents are placed in an oven preheated to the desired test temperature. After the curing period, the beaker is removed from the oven and cooled to about 60°C, and water is then carefully added to produce a slurry with about 50% solids. This slurry is leached under agitation for several hours and the residue is then filtered and washed. Often the cured cake will break up readily and agitation leaching is no problem. If a hard cake forms during the curing operation, it may be desirable to transfer the pugged mixture to a heavy Pyrex baking dish before the curing operation. The cured product can then be broken up if necessary before the agitation leach. Test results are compared with those obtained by standard agitation leach procedures.

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10.6. ACIDIC PRESSURE LEACHING

Acidic pressure leaching is most often considered for solubilizing refractory uranium ores that require relatively stringent oxidation. The leaching temperatures are normally, but not always, higher than the normal atmospheric boiling point of the leach solution. If sulphide minerals are present, sulphuric acid may be generated in situ by the oxidation of the sulphides. This technique is costly and should only be considered when conventional leaching at atmospheric pressure appears inadequate.

The optimal oxygen partial pressures for a given ore must be determined experimentally and may range from 100 kPa to 700 kPa or more. Temperatures between 70 and 200°C have been reported, but optimal temperatures are often near 150°C. As would be expected the reactions proceed more rapidly as the temperature is increased and leaching is often complete in 4 h or less.

Laboratory pressure leaching tests have most often been carried out in 1 or 2 L stirred autoclaves equipped with a gas inlet line which can also often be used to take thief samples of the pulp. The autoclaves also have a gas bleed valve and a heavy walled well to house the temperature measuring device. The autoclave should be equipped with a rupture disc to provide pressure relief in the event of an unanticipated pressure excursion.

Since pH cannot normally be monitored and acid additions cannot be made during bench scale pressure leach tests, acid variations are most often investigated by varying the amount of acid introduced into the slurry at the start of the test. After the test is completed and the reaction vessel cooled, the leach products are handled in the same way as those from atmospheric leach tests. Some reaction vessels are equipped with internal cooling coils that permit rapid cooling at the completion of a test. With sulphate solutions at temperatures up to about 160°C, type 316 stainless steel is normally a satisfactory construction material for laboratory autoclaves. For tests at higher temperatures or if Cl^- or similar ions are present, metals such as titanium may be required. Manufacturers' recommendations should be carefully studied and followed. As in all pressure leaching work, when working with new samples which have unknown characteristics, relatively dilute slurries and smaller reagent additions should be used during the initial test work.

11. FLOW SHEETS AND MATERIAL BALANCES

11.1. FLOW SHEETS

During laboratory testing the ore sample passes through different steps in which the conditions of the treatment of an ore in full scale equipment are simulated. At each test parameters are measured whose values must be known in order that two basic types of information required for economic evaluation can be developed:

(a)   Type and size of equipment required
(b)   Consumption of reagents and energy.

It may be said that the basic objective of laboratory testing is to select the unit operations which require the simplest and smallest equipment and the lowest consumption of reagents and energy. In each test procedure the results are compared with the information available from prior experience or with data available from processing similar ores. In some cases further testing may be unnecessary if the results are evidently unreasonable, e.g. leaching efficiencies as low as 50% or filtration times as long as 1 h. In principle, however, the selection of alternative processing steps should not be based on individual tests. The final economic evaluation of the project should be done on the basis of the complete process, from the ore to the concentrate. To make possible an economic assessment of the entire process, single unit operations should be integrated into a complete process in the form of a flow sheet. The test results may lead to a single flow sheet or to several alternative flow sheets. A final selection can be arrived at by further testing, possibly including pilot plant tests, and more refined economic evaluations.

Depending on the level of testing (e.g. preliminary, initial or detailed), the flow sheets show different degrees of complexity. The flow sheet may be relatively simple in an early stage of the development of the process but may become very complex and may include a great number of different data when the development of the project proceeds.

The flow sheet together with material balances are in fact the product of complex information developed from the test results and serve for further technical and economic evaluations performed by different experts outside the testing laboratory. The transformation of direct test results or measurements into a form which may be used by other engineers and economists is a rather complicated procedure which requires much experience of laboratory testing, pilot plant testing and industrial practice. However, once a probable process route has emerged from mineralogical studies and hydrometallurgical tests, the selection of the equipment type may be done and equipment size selected or estimated to accommodate the required tonnage of the ore to be treated.

In Table XVI examples are given of laboratory tests, the test results and the design data which are developed from these results for flow sheet development.

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TABLE XVI. EQUIPMENT DESIGN DATA DEVELOPED FROM LABORATORY TEST RESULTS

Laboratory test	Test results	Design data
Grinding Laboratory batch grinding test	Bond work index	Mill power consumption; mill size and type
Leaching Leaching tests (acid and alkaline, static and agitated leaching)	Uranium recovery, reagent consumption and leach liquor compositions for different temperatures, leaching times, reagent concentrations, grind size, slurry densities, pressures	Type of leaching (acid or alkaline, atmospheric or pressure, agitated or static), number and volume of leaching vessels, reagent and energy requirements, uranium losses, leach liquor composition
Solid–liquid separation Settling and filtration tests	Thickener areas, slurry densities, flocculant requirements, filtration and washing rates	Number and size of thickener in CCD circuit, filter type (drum or belt), filter size, number of filtration stages, wash water requirements, washing efficiencies, flocculant and energy requirements. Composition of pregnant liquor, vacuum pump size
Uranium recovery Ion exchange, solvent extraction	Type of resin, type of strip solution, breakthrough curves, elution curves, resin capacity, eluate composition, resin poisoning	Size of ion exchange columns, number of stages, resin inventory, reagent composition, uranium losses, etc.
	Types of extractant, solvent and modifier, extraction and stripping isotherms, phase ratios, extraction kinetics, settling area, stripping solution, organic load, raffinate composition, pH, entrainment loss	Size and number of mixer–settler units, type of extractant, strip reagent system, reagent consumption, entrainment loss, uranium losses, etc.

TABLE XVI. (cont.)

Laboratory test	Test results	Design data
Uranium precipitation		
	Reagent type, retention times, filterability, washing rates, settling rate, chemical composition of precipitate and filtrate, reagent requirements	Number and size of precipitation tanks, precipitation reagent type, type and size of solid–liquid separation equipment, cake moisture, drying requirements, uranium losses, etc.
Waste management Neutralization of raffinate, decontamination with $BaCl_2$, etc.	Reagent consumption, retention times, effluent quality, settling rate, filterability, permeability of solids, angle of repose, etc.	Size of neutralization vessels, reagent consumption, type and size of solid–liquid separation equipment, energy consumption, surface area, etc.

In the transformation of test results into design data some safety margins must be included which will allow the designed capacity to be maintained in the case of ore variability, decrease of equipment efficiency, resin poisoning, etc. As mentioned before, experience is required for this work and assistance can generally be obtained from engineering companies and equipment vendors.

When the type of equipment is chosen and its size estimated, with allowance for sufficient safety margins, the cost may be estimated in two ways:

(a) Equipment design data (along with the original test results preferably) may be sent to equipment manufacturers, who will give approximate costs.

(b) Costs may be estimated on the basis of published information for similar equipment (in type and size).

Both methods of cost estimation yield only approximate results. In estimating investment cost, inexperienced persons may very easily underestimate the total investment costs, which include the costs of auxiliary equipment, civil works and spare parts, transportation costs, erection costs, taxes, etc.

Figures 37–39 show flow sheets of different degrees of complexity representing the acid leaching process.
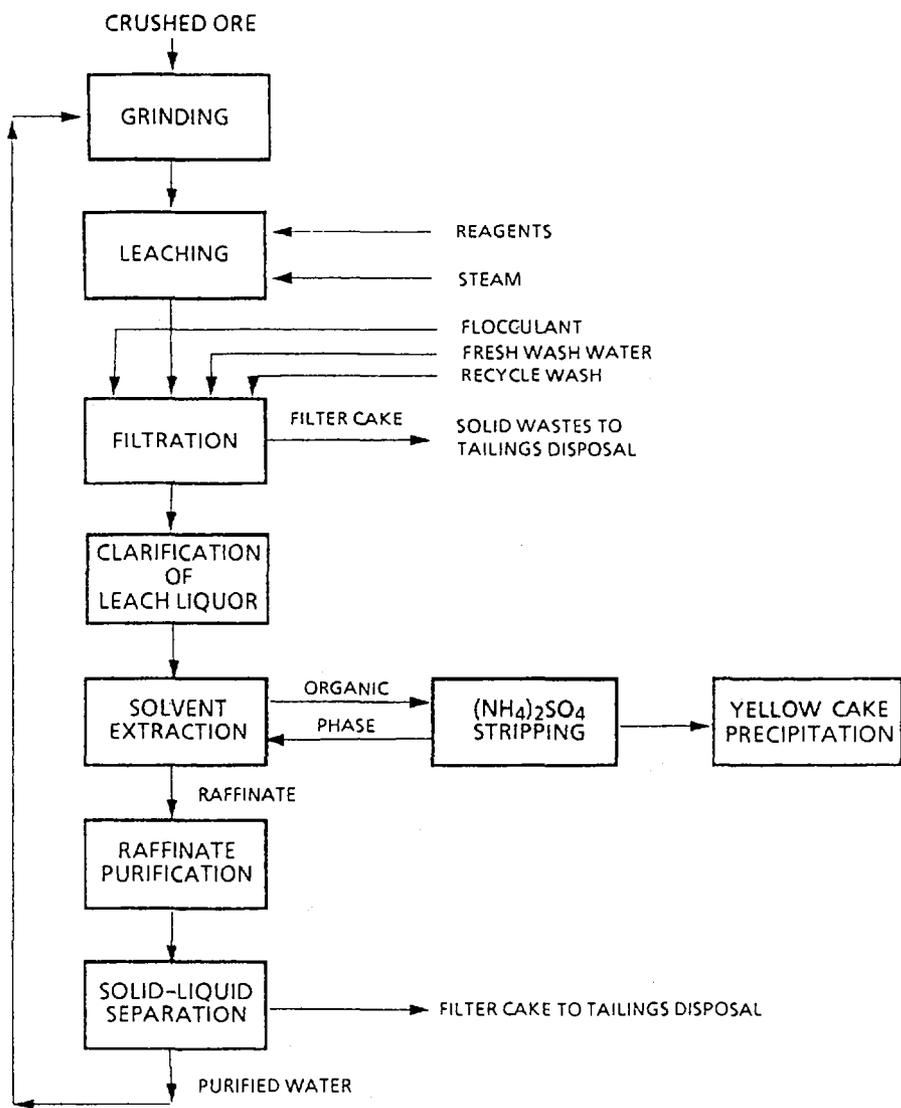
FIG. 37. Flow sheet for acid treatment of uranium ore.

11.2. MATERIAL BALANCES

The most basic set of data which describes a particular treatment process are data on the flows of solids and liquids (and gases) through the process. All flows have to be quantified and represent the material balance of inputs, outputs and intermediate quantities.

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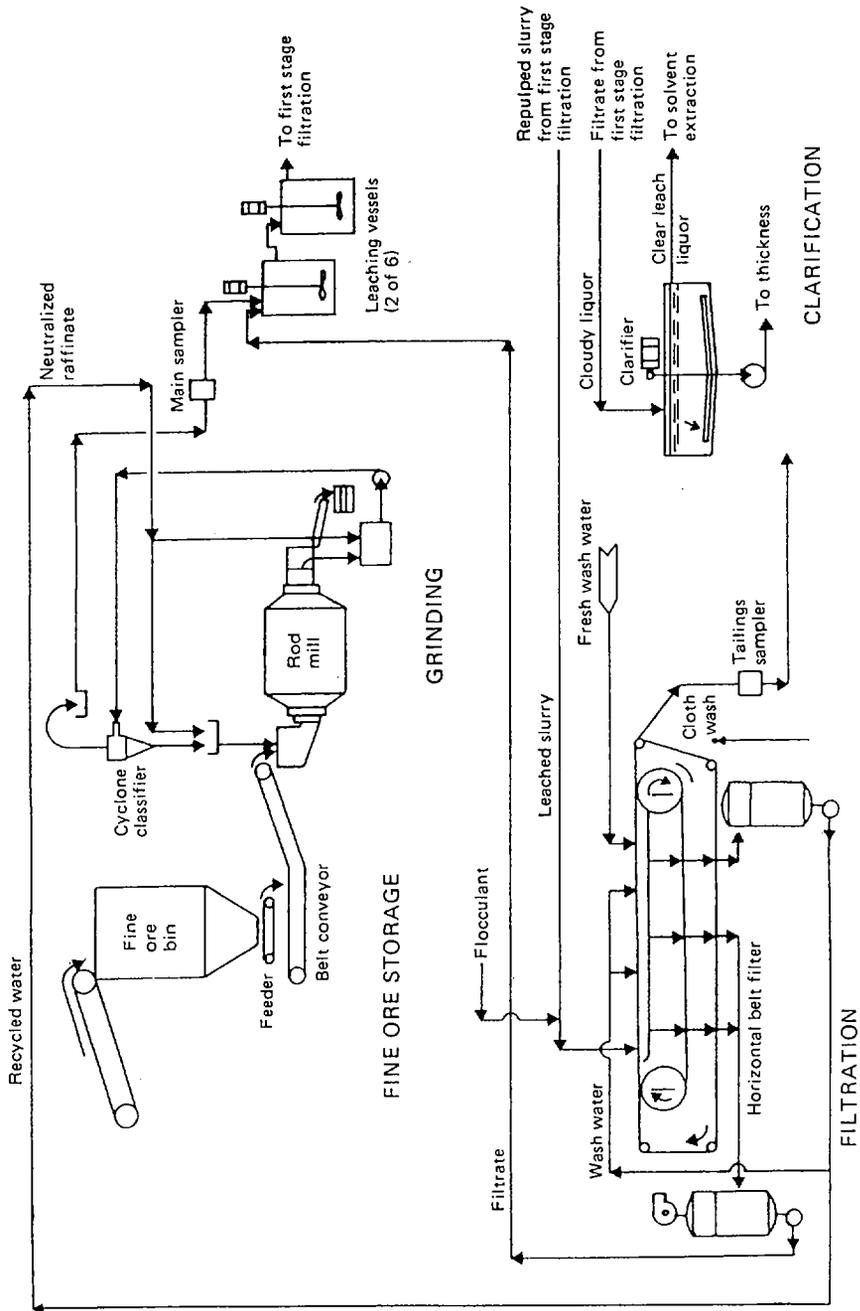
FIG. 38. Partial flow sheet for acid treatment of uranium ore: fine ore storage, grinding, filtration and clarification.

FIG. 39. Flow sheet for acid treatment of uranium ore: grinding circuit.

Material balances should include the flows of:

— Solids, e.g. the ore, leached residues and washed tailings, uranium concentrate and chemicals;

— Liquids, e.g. process solutions, wash waters, auxiliary waters, liquid chemicals and organic extractants;

— Gases, e.g. gaseous chemicals, steam, ventilation air and off-gases such as CO_2.

The material balance of the main solids is usually a simple one: the moisture in the ore must be known and the weight change during leaching has to be measured. The material balance of the liquids, or 'water balance', may be very complicated because solutions are often used several times and are recirculated in the internal and external loops. The material balance should include also the tailings disposal area. For the water balance, data on the specific gravity of the different liquids should be obtained. Of major importance is the 'metal balance', which shows the distribution of uranium in different streams: liquid effluents, washed residues, other wastes and products. The sum of the portions of the uranium in these streams must be balanced with the uranium entering the process with the feed.

The material balances usually are mass balances, but volumes also have to be given for liquids (clear liquids and slurries). The data for different materials are referred to the unit weight of dry feed ore, such as:

— The consumption of chemicals and materials per tonne of ore treated, e.g. steel consumption during grinding, acid consumption, flocculant consumption, consumption of neutralizing agents and consumption of filter medium;
— The consumption of water per tonne of ore treated, e.g. water for grinding, wash waters for filter cakes and CCD, wash water for equipment, floor wash water and spills.

Consumptions which are directly proportional to the quantity of ore treated and are in fact independent of the grade of the ore are referred to the unit weight of dry feed ore.

The consumption of some reagents is proportional to the quantity of uranium treated, e.g. ammonia for precipitation. In this case the consumptions are referred to the unit weight of uranium or U_3O_8 in the product. With the data on the grade and overall efficiency of the treatment, these consumptions may be converted to consumptions referred to the unit weight of dry feed ore.

Material balance data are basic to the design of equipment and the economic evaluation of the process.

12. REPORT WRITING

Properly prepared reports are essential for the successful development of a project. Ore testing reports should follow the same criteria and standards that apply to technical reports in general. The reports should cover all important aspects and should be well organized and accurately and clearly presented. They should be long enough to provide the level of detail required for further work but unnecessary material should be rigorously excluded. The literature of technical report writing is

very extensive: readers who may wish to review this topic in greater detail are referred to the Bibliography.

12.1. INITIAL RESEARCH

Before beginning the test work it is advisable to collect and assemble all available information on the type of ore body, ore reserve estimates, possible mining methods, and estimates of the amount of mineable ore, the waste to ore ratio and the production rate and on the nature of the ore and the host rock. This information will be immediately useful for guiding the test work and will be needed later for the preliminary economic evaluation. Ore body development and metallurgical process development are integral parts of the development of a project and cannot be done in isolation from one another. The metallurgist must have a clear idea of the nature of the ore body, of the ore and of the ore samples being tested, and should also be well informed of other aspects of the project as a whole.

12.2. LABORATORY NOTEBOOKS AND OTHER SUPPORTING DOCUMENTATION

All test work must be fully documented. There should be full descriptions of the equipment, methods and materials used and of the results obtained. This information is usually recorded in laboratory notebooks, but these should be supplemented by diagrams, graphs, photographs and other materials. This documentation will be the basis for the preparation of progress reports and of the final test report. It can also be useful for the verification of test results, should this be necessary.

The laboratory notebook should be bound and all the pages numbered. Entries should be made directly in the notebook to avoid the loss of important information.

12.3. CONTENTS OF TEST REPORT

The laboratory ore test report, together with the report on ore reserve estimation, the report on possible mining methods and other supplementary information, will serve as the basis for a preliminary evaluation of the project. An example of a suggested report layout is shown in Table XVII. The contents of a test report must, of course, be determined by the metallurgists in charge, according to the specific circumstances.

It is good practice to include an abstract, usually placed before the report itself. The abstract should not be more than one page long. It should describe the contents

TABLE XVII. EXAMPLE OF ORE TEST REPORT LAYOUT

Title page
(including title and date of report and name(s) and affiliation(s) of author(s))

Abstract

Contents list

Executive summary

1. Introduction

2. Summary description of ore body

3. Summary ore reserve estimates

4. Summary description of proposed mining methods

5. Description of samples studied

6. Chemical, mineralogical and petrographic characterization of ore

7. Grinding tests

8. Leaching tests

9. Solid–liquid separation

10. Uranium recovery and concentration (by solvent extraction or ion exchange)

11. Precipitation and filtration

12. Drying

13. Characteristics of concentrate

14. Discussion of laboratory test results

15. Proposed process flow sheets

16. Conclusions

17. Recommendations

18. Bibliography

Appendices

Index

of the report and should include the main results obtained and the conclusions reached but should not contain tables, diagrams, bibliographical references or acknowledgements. It is also desirable practice to prepare an executive summary for a long report.

The ore test report should contain all the information that will be needed for the preliminary project evaluation, such as:

(1)   A summary description of the ore body, specifying its location, type, geological setting, general shape (in three dimensions) and size (in three dimensions) and the disposition of the mineralization within the deposit. Maps and diagrams should be included, showing boreholes, trenches, shafts or other development work. The homogeneity of the deposit, in terms of both mineralogy and grade, should also be discussed.

(2)   A summary of the ore reserve estimates, including the tonnage, volume and grade of the various types of reserves. The method used for ore reserve estimation and the accuracy of the estimates should be discussed.

(3)   A summary description of proposed mining methods, including estimates of production rates, assumed or proposed cut-off grades, mining losses, waste to ore ratios and ore dilution. Diagrams should be included.

(4)   A description of the sample or samples used for analysis and for laboratory testing. The origin and type of the samples should be clearly stated. The procedures used for sample collection and preparation should also be described.

(5)   Results of the chemical and instrumental analyses of the ore and the host rock, including a brief description of the methods of analysis and the instruments used. Estimates of the precision and accuracy of the results should be given. The calibration and standardization procedures used should also be described.

(6)   Mineralogical and petrographic characterization of the ore. Macro- and microphotographs should be included if possible.

(7)   A description of the tests performed for each unit operation, including a complete description of the equipment, materials and procedures used. Diagrams or photographs of the equipment should be included whenever possible, particularly when this is not commercially available and was custom built. The descriptions should be sufficiently complete to allow other metallurgists to reproduce the tests and verify the results.

(8)   A summary of the results obtained. A formal report usually includes only the reduced data and not the raw data. A consistent system of units (preferably SI) should be used throughout and estimates of the precision and accuracy of the results should be given. Some results, such as those for leaching rates, pulp settling rates, ion exchange column loading and elution rates, and solvent extraction loading and stripping rates are best shown in graphic form.

(9)   A discussion of the results. The meaning and significance of the results obtained should be explained.

(10) A description of the proposed flow sheet or flow sheets based on the laboratory results. Flow diagrams or, as a minimum, block diagrams must be included. Material balances should also be included.

(11) Conclusions. These should be based solely on the data in the report.

(12) Recommendations. These should include a statement of future actions that are needed.

(13) Bibliography. The sources of information used should be fully identified according to standard practice so that they can be consulted if needed.

(14) Appendices. Detailed information that is related to the test work but not essential for following the main discussion should be presented in the form of one or more appendices. For instance, the detailed description of a particular piece of equipment or the detailed description of a specific analytical procedure might be best included in an appendix.

It may be necessary to prepare one or more preliminary (or progress) reports during the development of the test work and before its completion. These preliminary reports, by their very nature, cannot be as comprehensive and detailed as the final report but should essentially cover the same topics, omitting only those items that have not been sufficiently developed at the time of writing.

Reading some test reports prepared by experienced metallurgists can be very instructive and is recommended. Some of these reports are cited in the Bibliography.

Appendix I

LABORATORY SAFETY

Laboratory testing of uranium ores involves risks that must be understood and guarded against. Two broad types of risk may be considered: general risks, common to most chemical laboratories, and radiological risks.

I.1. GENERAL SAFETY PRECAUTIONS

All the safety precautions that apply to a chemical laboratory apply also to a metallurgical laboratory for ore testing. These precautions have been well documented (see Bibliography). Standard precautions against the following should be observed:

(a)   Corrosive substances, such as sulphuric and other acids and caustic solutions.
(b)   Explosive and flammable chemicals, including diluents and extractants used for solvent extraction.
(c)   Dusts, especially ore dusts and uranium concentrate dusts.
(d)   Chemical spills, including acids, leach solutions and other process pulps and liquids.
(e)   Mechanical hazards, especially during the crushing, grinding and other handling of ores.
(f)   Electric shock.
(g)   Toxic or irritating chemicals. Virtually all chemicals used in the laboratory are toxic or irritating in some degree. Special care must be taken with liquids used in heavy media separations, extraction solvents and ammonia used for precipitation of concentrates.
(h)   Pressurized vessels, such as autoclaves. It is extremely important to study carefully and follow the manufacturer's instructions when using this type of equipment.
(i)   Burns from hot plates and furnaces.

Laboratory coats or overalls should be worn at all times. Appropriate safety equipment such as hard hats, face shields, safety goggles, gloves, respirators and hard tipped boots should be used as required.

I.2. RADIOLOGICAL SAFETY

Radiological risks in a uranium metallurgical laboratory are small and can be easily controlled (see Bibliography). The main risks concern the inhalation of

uranium concentrate dust and uranium ore dust. External irradiation and inhalation of radon and radon daughters are also possible although minor risks.

Substantial amounts of dust can be generated during sample preparation: crushing, grinding, splitting, screening, sieving and blending. Sample preparation rooms should be isolated from other areas and should be equipped with dust control systems such as hoods and filters. Access to these rooms should be controlled. The personnel preparing the samples should be properly attired with overalls, boots, gloves, dust masks and caps. Workers should also shower and change into their normal clothing before leaving the controlled area.

Inhalation of uranium concentrate dust is less likely because the amounts handled in the laboratory are usually small, seldom more than a few hundred grams. Nonetheless, dry concentrates should be handled with care, especially during screening, blending or any other operation that can generate dust. These operations can be carried out under a hood or, better still, in a glove box. Appropriate attire should also be worn.

Radon and radon daughters are normally not a problem, provided that the laboratory is well ventilated. The risk of external irradiation is usually negligible, except perhaps when working with very high grade ores. It is advisable to check the level of radioactivity in the laboratory, especially in the sample storage room and in the area where uranium concentrates are stored. If the levels of radioactivity are significant the risk can be controlled by limiting the residence time in these areas, although this time is likely to be small in any case.

Care should be taken to keep the laboratory clean at all times. Special care must be taken to clean up all spills of uranium bearing pulps or liquids. Surfaces such as bench tops and floors should be regularly monitored for possible contamination.

Eating, drinking and smoking must not be allowed in any area of the laboratory.

I.3. SAFETY RULES

Safety rules, covering both general and radiological safety, should be drafted and put into effect. It is strongly recommended to consult specialized books on these subjects (see Bibliography).

Appendix II

GUIDELINES FOR LABORATORY DESIGN

A well designed laboratory provides important advantages mainly by minimizing radiological and other risks and the risk of contaminating the samples being investigated.

A metallurgical laboratory for testing uranium ores will usually have the following separate areas (Fig. 40):

(1) A sample reception room, which may also house the scales, the crushers and the coning and quartering platform.

(2) A sample preparation room, housing the pulverizers, mills, screens, sample splitters and blenders, stoves and other related items.

These first two rooms should be provided with hoods and cyclones or filters to control dust emissions and should be segregated from the rest of the laboratory to minimize the risk of contamination.

(3) A sample storage room, to keep ore samples for future reference.

(4) Showers and lockers, with direct access from the sample reception and preparation rooms so that the workers who prepare the samples can shower and change into clean clothes before leaving the area so as not to spread ore dust.

(5) The main laboratory, housing the equipment used for pretreatment, leaching (except autoclaves), solid–liquid separation and purification and concentration. The equipment used for these unit operations may also be housed in separate rooms.

(6) A high pressure room, housing autoclaves and any other high pressure equipment. When working with this type of equipment there is the risk of explosion. The probability of an accident is small if the equipment is properly used, but should an accident occur the consequences could be very serious. This room should be specially designed and should preferably include a labyrinth, mirrors and a knock-down exterior wall or knock-out panels.

(7) A concentrate room, used for precipitation, filtration, drying and calcining of concentrates. This should be a separate room in order to minimize the risk of contaminating ore samples and process samples. This is particularly important when working with low grade ores.

(8) An analytical laboratory, provided with chemical hoods, to decompose ore samples and to prepare them for chemical or instrumental analysis.

(9) An instrument room, housing the analytical balances, colorimeters, fluorimeters, microscopes and other instruments. This room should be well isolated from the rest of the laboratory to protect the instruments from dust,

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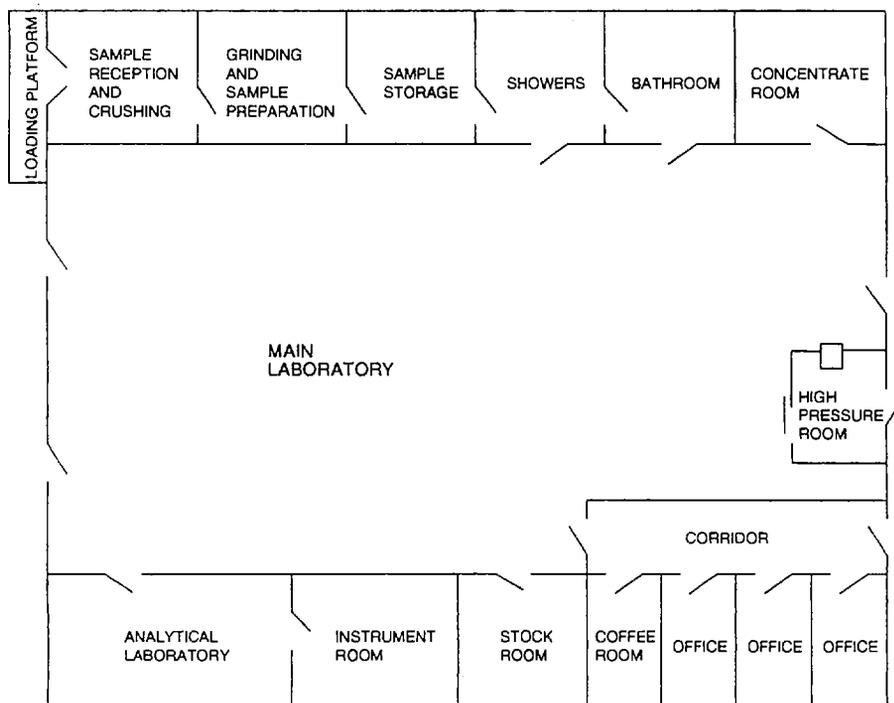
FIG. 40. A suggested laboratory layout.

heat, water and corrosive vapours or chemicals and to keep the samples and instruments from being contaminated.

(10) Stock room.

(11) Offices.

(12) Coffee room. Eating, drinking and smoking must not be allowed in any other area of the building. This rule can be more easily enforced if an accessible alternative area is provided for these purposes.

The total area for the laboratory should be 400 to 500 m^2, although it is possible to work with a smaller area. Figure 40 shows an example of a laboratory layout that includes all recommended areas and follows the criteria indicated above.

The laboratory should be well lit and well ventilated. Provision should be made for the following services:

— Electricity (110 and 220 V)
— Mains water (hot and cold)
— Deionized water
— Compressed air (40 lb/in^2)
— Vacuum

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— Gas (natural or propane)
— Emergency showers and eye washing fountains
— Conventional drainage
— Special drainage for the laboratory itself, discharging into a sump.

The electrical supply for the instrument room should preferably be independent from the electrical supply for the main laboratory.

Appendix III

SUGGESTED LABORATORY EQUIPMENT AND SUPPLIES

The extractive metallurgy of uranium is not very different from that of other non-ferrous metals, and therefore the equipment needed is largely the same as in any metallurgical testing laboratory.

III.1. EQUIPMENT FOR SPECIFIC OPERATIONS

The following list of suggested equipment has been arranged according to the main unit operations usually involved in uranium ore processing. A given piece of equipment may, of course, be useful in more than one unit operation.

(a) *Crushing*

— Jaw crusher: size of opening: 2.5 in × 3.5 in
                discharge size: 0.5–0.25 in
                capacity: 50 kg/h
                power: 1 hp

    or

— Jaw crusher: size of opening: 3 in × 4 in

(b) *Grinding*

— Disc/plate pulverizer
— GY-ROLL laboratory crusher ('coffee mill'), 10 in model (1 hp)
— Laboratory rod mill and rod charge; mill size: dia. 8 in, length 9.75 in
— Paul Abbé ceramic mill, 17 cm long and 20 cm in diameter
— Bond grinding test mill with standard tachometer, or Allis–Chalmers standard test mill
— 19 mm steel balls (20 kg)
— Timer and wattmeter.

(c) *Sample preparation*

— Coning and quartering platform
— Quartering cross
— Shovels

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— Sampling table
— Screens (0.25, 0.5, 1 and 2 in)
— Jones riffle splitters, 6 in × 8 in and 10 in × 18 in
— Sample blenders
— Scale, 500 kg
— Scale, 10 kg
— Beam scale, 1 kg
— Jar mills
— Jar rolls
— Rolling cloth
— Set of standard testing sieves (mesh sizes: 400, 325, 270, 200, 150, 100, 65, 48, 35, 28, 20, 14, 10, 8, 6; two 200 mesh sieves should be included)
— Cyclosizer, or
— Haultain Infrasizer
— Sieve shaker
— Sieve brushes
— Sample handling pans
— Air compressor

(d) *Pretreatment*

— Flotation machine
— Wet concentrating table (Wilfley table)
— Magnetic separator

(e) *Leaching*

— Autoclave, 2 L
— Standard hot plates
— Stirrer hot plates
— Stainless steel beakers, 2 L, 1 L
— Timers
— Interval test timers
— Chronometer
— Laboratory pumps
— Volumetric pumps
— Balances
— Electric stirrers (with variable speed motors, 0.1 hp) and impellers
— Centrifuge
— Drying oven
— Muffle furnace
— Batch vacuum filter

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— Pressure filter
— Storage containers
— Laboratory glass thermometers
— Steel or aluminium scoop

(f) *Solid–liquid separation*

— Vacuum pump
— Buchner funnels
— Leaf filter kit

(g) *Purification and concentration*

— Laboratory mixer–settler system for solvent extraction complete with electric mixers, pumps, rotameters, tubing and valves
— Ion exchange columns
— Peristaltic pumps

(h) *Precipitation*

— Stirrer hot plates
— Stainless steel beakers
— Buchner funnels, 10 cm diameter

III.2. GLASSWARE

(1) *Beakers*

  20 mL (6)
  50 mL (6)
  100 mL (6)
  150 mL (6)
  250 mL (6)
  400 mL (6)
  500 mL (4)
  600 mL (4)
  1000 mL (4)
  2000 mL (2)
  3000 mL (2)
  4000 mL (2)

(2) *Plastic containers with lids (for mixing stock solutions)*

  1 gal (2)
  2 gal (2)
  5 gal (2)

(3) *Graduated cylinders*

  5 mL (2)
  10 mL (2)
  25 mL (2)
  50 mL (2)
  100 mL (2)
  250 mL (2)

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500 mL (2)
1000 mL (2)
2000 mL (**1**)

(4) *Stoppered graduated cylinders*

  100 mL (2)
  250 mL (2)
  500 mL (2)
1000 mL (1)
2000 mL (1)

(5) *Erlenmeyer flasks*

   25 mL (2)
   50 mL (2)
 125 mL (4)
 250 mL (4)
 500 mL (2)
1000 mL (2)
2000 mL (2)

(6) *Volumetric flasks*

  100 mL (2)
200 or 250 mL (2)
  500 mL (2)
1000 mL (2)

(7) *Separating funnels with stoppers and Teflon stopcocks*

   60 mL (2)
 125 mL (6)
 250 mL (6)
 500 mL (2)
1000 mL (2)
2000 mL (**1**)

(8) *Buchner funnels*

Plate diameters:

  75 mm (2)
126 mm (2)

186 mm (1)
240 mm (1)

(9) *Filter flasks*

  250 mL (2)
  500 mL (2)
1000 mL (2)
2000 mL (1)
4000 mL (1)

(10) *Watch glasses*

4.5 in dia. (6)
 6 in dia. (6)
 8 in dia. (6)

(11) *Pipettes*

   5 mL (2)
  10 mL (2)
  15 mL (2)
  25 mL (1)
  50 mL (1)
 100 mL (1)

(12) *Funnels*

100 mm dia. (1)
215 mm dia. (1)

(13) *Long stem funnels*

65 mm dia. (12)

(14) *Powder funnels, plastic*

 80 mm dia. (2)
100 mm dia. (1)
150 mm dia. (1)

(15) *Plastic jugs*
(for storing stock solutions)

1 gal (4)
2 gal (2)
5 gal (2)

III.3. INSTRUMENTATION

— pH meters (at least 2 or 3)
— Glass thermometers (5 to 10)
— Fluorimeter
— Conductimeter
— Colorimeter
— Analytical balance
— Binocular microscope
— Relative humidity and temperature recorder

III.4. SAFETY EQUIPMENT

— Hard hats
— Hard tipped boots
— Rubber gloves
— Dust masks
— Safety goggles
— Face shields
— Respirator
— Fire extinguishers
— First aid kit
— Safety storage cans (for organic liquids)

III.5. ADDITIONAL EQUIPMENT

— Geiger counter
— Electronic desk calculator
— 35 mm camera
— Typewriter

III.6. REAGENTS

— Acetone
— Methanol
— Kerosene
— Sulphuric acid
— Hydrochloric acid
— Ammonium hydroxide

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— Sodium hydroxide
— Isodecanol
— Distilled or deionized water
— Sodium chlorate
— Manganese dioxide
— Sodium chloride

III.7. ASSORTED SUPPLIES

— Soap, powdered and liquid
— Test tube brushes
— Filter paper, assorted sizes and filtration rates
— Paper towels
— Glass tubing
— Rubber tubing
— Plastic squirt bottles
— Cotton
— Rubber and cork stoppers, assorted sizes

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