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**FIELD AND LABORATORY METHODS USED BY  
THE GEOLOGICAL SURVEY OF CANADA IN  
GEOCHEMICAL SURVEYS: PREPARATION OF  
GEOLOGICAL MATERIALS FOR CHEMICAL AND  
SPECTROCHEMICAL ANALYSIS; SEPARATION AND  
CONCENTRATION OF MINERALS; AND A GUIDE TO  
THE IDENTIFICATION OF COMMON MINERALS**

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# **FIELD AND LABORATORY METHODS USED BY THE GEOLOGICAL SURVEY OF CANADA IN GEOCHEMICAL SURVEYS: PREPARATION OF GEOLOGICAL MATERIALS FOR CHEMICAL AND SPECTROCHEMICAL ANALYSIS; SEPARATION AND CONCENTRATION OF MINERALS; AND A GUIDE TO THE IDENTIFICATION OF COMMON MINERALS**

## **ABSTRACT**

This paper outlines methods for the preparation of rock, soil, drainage sediment, mineral concentrates, and biological samples for chemical analysis. The necessary equipment to carry out the preparatory work is listed and discussed, and a guide to the identification of common minerals is included.

## **PREPARATION OF GEOLOGICAL MATERIALS FOR CHEMICAL AND SPECTROCHEMICAL ANALYSIS**

### **INTRODUCTION**

The methods described in this paper are based on a number of years experience in preparing rock, mineral, soil, glacial materials, drainage sediment, and biological materials in a well established laboratory for chemical and spectrochemical analyses. With slight modifications the methods can be adapted for use in a field laboratory.

Most geological materials require some form of preparation before being chemically analysed or separated into their component minerals. Those involved in collecting the materials should supervise the preparations when possible. The reason for this recommendation is to ensure that proper procedures are followed at the preparation stage, thus guaranteeing that uncontaminated representative samples reach the analytical laboratories responsible for chemical and/or spectrochemical analysis.

Prior to chemical and/or spectrochemical analysis, it is generally necessary to crush and grind the solid

materials, to sieve out the fine fraction from unconsolidated materials, and finally to ball mill a portion of this fraction to a fine powder. Furthermore, it may be occasionally necessary to separate and concentrate certain of the minerals present in the samples.

During preparation of samples it is important to bear in mind certain basic rules that are necessary to conserve purity and integrity of the sample. These rules refer to such factors as attention to abrasion and wear of the machine parts, cross-sample contamination, and loss of sample material. In other words, the prepared sample on submission for analysis should contain the same type of elements as when found in nature.

Due to the wide variety of geological materials in nature, preparation methods may vary widely; the laboratory technician must, therefore, be flexible and imaginative in his choice of the technique(s) to be applied.

The procedure for preparing rock samples for analysis is described first and is followed by the procedures for drainage (stream and lake) sediment samples, biological materials and mineral separation. A guide to the identification of common minerals, a list



of the equipment required for crushing, grinding, and sieving rock samples and for the preparation of drainage sediments, biological materials, and mineral separates, and the use of ceramic disks on power grinders are given in Appendixes I, II, and III.

### **FIELD PREPARATION OF ROCK, SOIL, GLACIAL MATERIALS AND DRAINAGE SEDIMENT SAMPLES FOR ANALYSIS**

Field men selecting and shipping rock, soil, glacial materials and drainage sediment samples for analysis should bear in mind the problem of contamination from sample to sample, the loss of samples owing to incorrect labelling, and the ease of handling in the preparation laboratory. Some of the points to note include the following:

**Drying samples in the field:** Dry all rock samples before packing in plastic bottles or in canvas, plastic or paper bags.

**Packing of rock samples:** Pack each rock sample individually in a good grade canvas bag. If plastic bags are used some method of keeping the samples from cutting the bags and hence contaminating others must be devised. Avoid packing more than one sample in each bag.

**Packing of soil, till, drainage sediment, etc. samples:** Soil, till, drainage (stream and lake) sediment samples should be packed in heavy paper bags secured with a waterproof cement. This permits drying in the bags before shipment. If plastic bags are used for collecting soil, till, drainage (stream and lake) sediment samples, the materials should be dried before shipment in plastic bags. Fine-grained samples of soil, till, drainage (stream and lake) sediment, and limonitic and wad precipitates should be dried and shipped if possible in plastic bottles with tight-fitting caps. The bottles should not normally be filled more than three-quarters in order to facilitate mixing before preparation and analysis.

Carefully tie, or otherwise secure, all bags to prevent escape of the material from the bags. If plastic bags are used plastic-covered metallic wire ('twist ems') provides a good rapid method of securing the bags.

**Marking the containers:** Clearly mark the sample number on the bags or plastic bottles in two places if possible, using a good indelible marking pen or pencil. The field man is solely responsible for the sampling procedure in the field, the numbering system, and for making sure that the samples arrive at the preparation laboratory clearly marked. To avoid errors all unmarked or illegibly marked samples are discarded as a matter of routine.

**Kraft paper bags:** The use of kraft bags, size 5 x 10 x 2 inch (12.5 x 25 x 5 cm) with folding perforated closing flaps (Pl. 1, fig. a) is advantageous because a series of these bags can be strung together, thus maintaining the order of sample collection. Light gauge plastic bags can be used to cover the kraft bags as a security measure when samples are collected wet.

**Instructions to laboratories:** Include a copy of the sample descriptions, sample numbers and instructions in the box of samples, and send a duplicate copy to the laboratory supervisor by mail.

### **LABORATORY PREPARATION OF ROCK SAMPLES**

Since rock samples differ widely in their constitution, weathered characteristics, size, etc. the method employed in their preparation for chemical and spectrochemical analysis must be flexible and adaptable to give good results for any type of sample.

When the laboratory technician receives the samples he must decide the method of preparation to be followed either on written instructions or in consultation with the field geochemist or geologist. The method described below is a general one capable of modification to deal with most rock samples.

The routine preparation of rock samples for chemical and spectrochemical analysis is generally completed in three major stages. These can be listed as follows:

**Stage 1:** Crush the rock samples using steel plates to yield 3/4 inch (20 mm) chips.

**Stage 2:** Grind the chips with ceramic plates using all or a representative part of the chips to pass a 6 or 10 (3.35 or 2.00 mm) mesh

sieve; or alternatively, sieve the 3/4 inch (20 mm) crushed material using a 6 mesh (3.35 mm) sieve, sample the minus fraction, or recrush to pass a 6 mesh (3.35 mm) screen all or part of the 3/4 inch (20 mm) chips using a smaller sample crusher mounted with ceramic jaws and side plates.

**Stage 3:** Place a representative portion (15 grams or more) of the sample in a plastic vial and store in racks (vial holding boards; see below) followed by ceramic ball milling to pass 140 mesh (106 $\mu$ m).

Some of the points to note in the laboratory preparation of rock samples follow.

**Plastic vials and holding racks:** The representative portion of the prepared sample (Stage 3 above) should be kept in a clear plastic vial (size 14 dram; 35 x 60 mm). This particular size offers sufficient surface area on which to mark the sample number and other information. In addition, the sample can be mixed in the vial if necessary, and the lumpy material can be broken down in the vial by vigorously shaking by hand or by machine when a ceramic ball is included with the sample. Racks to hold the plastic vials (vial holding boards) should be designed with 4 rows of 10 holes each for convenience and ease of handling (Pl. 1, fig. b).

**Numbering of plastic vials:** One method frequently used to number a large quantity of capped, plastic vials, that will eventually contain the representative samples, is to load only the outside rows of vial holding boards and then to tape a stack of approximately 10 racks together. The stack is then laid on its side. The vials are then numbered from left to right and from bottom to cap (Pl. 1, fig. d). This simple precaution ensures that the last number on a vial is readily visible whenever a particular vial is sought among others in the rack.

**Sample handling:** Arrange all samples to be crushed and ground in the numerical order of their sample numbers in rock sample trays placed on a long table. Mark vials, bags, etc. to contain ground samples with proper identification numbers before proceeding with any of the preparation procedures. This step is essential when checking possible contamination from one sample to another. If

contamination does occur the order in which the samples were crushed and ground must be known to permit location of the specific source or sample responsible for the contamination. Rock storage trays (Pl. 1, fig. c) should be clearly numbered in the order of the original collection of samples.

Prior to preparation, the trays containing the samples should be placed on one end of a cart while the other end serves to handle samples in preparation. These details may appear unimportant, but daily output can be greatly reduced if they are ignored. Another useful practice is to operate using sample batches. Work if possible in groups of 24 or 48 samples and carry these through all the steps given below before commencing work on another group. Experience has shown that this procedure is time conserving and leads to fewer errors.

**Equipment readiness:** Clean the crusher, grinder, and ball mills according to the method given below under the heading "Precautions to avoid contamination".

**Washing of samples:** Certain types of samples, e.g., rocks and ores from mines, drill core, etc., should be scrubbed, washed and dried. This step ensures that any foreign material which may contaminate them, such as rock flour from blasting, will not affect the analytical results. For very precise analytical work metal-free water should be used for washing samples; for normal analytical work tap water is satisfactory.

**Weathered surfaces:** Remove weathered surfaces from rock samples if this is desired by the geochemist submitting the samples.

**Splitting and retention of a reference chip:** When all the samples are organized in numerical order, the first step in the sample preparation is to split large specimens into pieces that can be fed into the crusher. One representative piece should be retained as a hand specimen. Power-driven (Pl. 1, fig. e) or hand (Pl. 1, fig. f) rock splitters are generally used for this purpose and should be cleaned between samples by brushing and removing any dust with a blast of air. Samples are usually placed, before and after splitting, on sheets of heavy gauge kraft paper. When the splitting operation is complete, the pieces

that are typically 2 or 3 inches (5.0 or 7.5 cm) in width are returned to the original sample bag.

A mechanical splitter is not always necessary for splitting samples; with soft materials (e.g., shales, carbonates, or friable drill core) it is often satisfactory to place the sample in a heavy gauge (5 mil) plastic bag and reduce or split it by hammering on a steel plate.

### **Stage 1: Crushing the Solid Samples**

#### **Precautions**

Solid samples should be crushed to yield material having a size appropriate for the next phase of preparation, i.e., a grinding machine requiring 3/4 inch (2.0 cm) fragments as feed. Crushing the sample to give these large chips eliminates unnecessary machine wear which may ultimately lead to contamination from the crusher's main shaft bearings and jaws. Wear on these parts may introduce steel particles containing molybdenum into the sample. Care must also be taken to avoid an excess of lubricants in the crusher as this may also introduce certain trace elements into the sample.

#### **Crushing procedure**

Once the samples are arranged in order and in trays, as described in a previous section, crushing can commence. The container bearing the sample, is emptied into the crushing machine; a representative cut can be retained if required. When this method is not applicable a kraft paper sheet should be spread out and the sample emptied on it; sample chips can then be hand fed or funnelled into the crusher using the paper sheet. This latter method is recommended because sample material can be examined prior to crushing, and special precautions can be taken during crushing or when cleaning the machines to avoid cross contamination. When the material has been crushed it should be transferred to the original bag.

#### **Cleaning the crushing machine**

The crushing machine must be cleaned after each preparation by rasping the jaws with a steel wire brush, followed by a blast of air. When out of the crushing chamber, the

jaw is placed on a bracket in front of the crusher, level with the main operating axle (Pl. 2, fig. a). This procedure avoids the lengthy manipulation of placing the jaw on the bench for brushing, etc. The bracket should be of such a size to accommodate the jaw and should be attached to the crusher by bolts. If the receiving tray has an 'open end' design, air blasting the milling chamber should clean the tray sufficiently. For health reasons these steps should be carried out under a ventilation hood.

### **Stage 2: Grinding the Solid Samples**

#### **Introduction**

The second stage of sample preparation involves processing the coarsely crushed material to render it homogeneous and of suitable size (6 to 10 mesh-3.35 to 2.00 mm) for fine milling (3rd stage of preparation). The methods are as follows:

- 1) A grinding machine fitted with ceramic disks is most suitable and can prepare large amounts of material in a short time and without fear of breakdown.
- 2) A smaller sample crusher specially designed and fitted with ceramic jaws and cheeks can be used to reduce 3/4 inch (2.0 cm) size chips for the 3rd stage of preparation.
- 3) Another method can be employed when the above two mentioned machines are not available. This involves sieving the crushed 3/4 inch (2.0 cm) material using a number 6 mesh sieve (3.35 mm), 8 inch (20.0 cm) diameter, and sampling the undersize. This method may not be entirely representative since oxidized or weathered surface material is mostly carried into the fines.
- 4) If the movable jaw of the crusher is redesigned as described in Appendix II, or the standard rotation of the crusher is reversed and/or the rpm increased, the discharged material can be made sufficiently fine to by-pass the second stage of preparation (i.e. grinding) and go directly to the Coors ball mill (i.e. the third stage of preparation). A 6 mesh (3.35 mm) sieving may, however, be necessary in some cases to remove the

larger particles. This method has the disadvantage of possible sample contamination from the shaft bearings and from additional wear of the crusher parts.

### Grinding procedure

If a grinder is used, the sample material can be fed directly into the spout of the machine using the original bag, especially if the bag is a thick 5 mil, 5 x 15 inch (12.5 x 37.5 cm) polyethylene bag. If this method is not workable, material should be placed on a kraft paper sheet which can be used to funnel the chipped rock into the grinder. All large pieces of the crushed rock should be hand fed.

For most work ceramic plates should be used on the grinder. For certain types of work not requiring precise chemical analysis, or for mineral separation, steel plates may be used. These introduce small amounts of iron and other elements in the steel into the sample. A magnet can be used to remove the steel particles from the sample; it should be noted however that magnetic minerals such as magnetite and pyrrhotite are also removed by this procedure.

### Homogenizing and sampling

The ground product is transferred directly to the original container from the receiving tray of the grinder and mixed in the container. If the original container is not suitable, it can be replaced with a sturdy 6 x 15 inch (15.0 x 37.5 cm) plastic bag. When mixed, a representative portion is selected by digging directly into the sample using a plastic vial marked with the corresponding sample number. At times it is possible to mix the sample in the receiving tray of the grinder and to sample directly from it. Another method is to mix the sample thoroughly by rolling 40 times on a sheet of kraft paper (Pl. 2, fig. b) and to scoop approximately a 15 gram grab sample from the 6 to 10 mesh (3.35 to 2.00 mm) size material. Several methods can be employed to obtain a good grab sample. The best way is probably by sectioning the sample into four parts and taking equal amounts from each of the parts to make up the representative (approx. 15 gram) grab sample. Retain the remaining

6 to 10 mesh (3.35 to 2.00 mm) size material for other work.

### New ceramic plates

When new ceramic plates are used, processing is slower because of the smoothness of the plates. A few kilograms of quartz should be run through the plates to roughen them. Corundum can also be used initially. Another recommended method is to use slightly damaged plates; a few small cracks or chipped out parts seize and hold the fragments and thus expedite grinding. Large ridges and cracks may be formed by constant grinding; these can be removed by grinding coarse corundum or by resurfacing on lapidary wheels (Pl. 7, fig. e).

### Subdividing large samples

Large samples that may consume too much grinding time should be first subdivided. To do this place the sample on two pre-cut kraft paper sheets whose dimensions will depend on the size of the sample. Mix and roll the sample and divide it in half by hand or with a spatula. One half of the sample is split off by lifting the top paper in such a way as to let the other half of the sample slide onto the second paper.

One portion is kept with the representative chips in a plastic container (bag). When sample preparation is complete the two subdivided parts are returned to the original bag.

### Hammering samples in plastic bags

Hammering (crushing) rock samples in a heavy gauge (5 mil thick, 7 x 15 in) (18.0 x 37.5 cm) plastic bag can be an efficient and rapid method for disaggregating softer rock samples, such as dried clay, dried muck, friable shale, and weathered schist for the ball milling stage of preparation. Frequently the first and second stage of preparation can be eliminated using this method. Cleaning is simple as the plastic bag can be discarded. Ceramic-faced hammers (see the section on Preparation of Small Samples) and bases used in the operation can be cleaned with sandpaper or cleaning compound. If steel



hammers are used, cleaning of the faces can be done on a machine shop grinder.

### **Recrushing the sample**

A smaller jaw crusher fitted with ceramic jaws glued to both of the steel jaws and side plates with epoxy (Pl. 2, fig. c) can be used in place of the large power grinder for the second preparation stage, especially if the samples are small and similar in composition. It has been found that continuous preparation of tough 3/4 inch (20 mm) sized material by small jaw crushers with ceramic plates requires a larger motor than the type recommended by the manufacturer. This permits the crusher to run faster and thus reduce stalling, making it more dependable, although its mechanical endurance is not comparable to a power grinder using ceramic disk plates or to an all steel power crusher. Due to a design deficiency, which does not provide a jaw which can be removed for cleaning, a small crusher would normally be used only for samples of similar composition, even if fitted with ceramic jaws and side plates. Cleaning methods include use of sand paper, quartz, air blasting of jaws, etc. When used for samples of unusual composition it is necessary to scrub the jaws and sides with corundum. Such a cleaning procedure usually requires dismantling the moving jaw and the use of corundum cloth wrapped around a wooden block. The chipped ceramic plates facing the sides and the jaws of small sample crushers can be restored by grinding on a lapidary wheel. Occasionally it is necessary to unglue and reverse the plates.

### **Stage 3: Ball Milling the Representative Part of the Sample**

#### **Introduction**

The third preparation phase requires Coors AD-99 grinding vials and AD-99 grinding balls (ball mills) supplied by Coors Porcelain Company, Golden, Colorado, U.S.A. The ball mills are held on a holding board and mounted on a shaker to prepare the sample material to pass a 140 mesh (106  $\mu$ m) sieve (Pl. 2, fig. e). The approximate capacity of an individual mill is 15 grams, and the time required to process a sample varies from 1 to 3 hours depending on pulley ratios and shaking

frequencies which are not the same for each shaker model. In general, the faster the mills are shaken the shorter is the milling period. On the other hand, very fast shakers may cause damage to the mechanism (transmission). The material processed by the Coors mill is usually homogeneous, and all aliquots should be comparable to each other.

### **Assembling the Coors Ball Mills**

Provided efficient handling methods are used, large numbers of samples can be processed in a short period of time. Each ball mill assembly comprises three main parts, two end caps and a center sleeve together with two gaskets and usually three balls for sample weights up to 15 grams and 4 or 5 balls for larger amounts. A complete milling operation or "run" consists of loading, cleaning and unloading. As for the other two stages of preparation, a batch amount is prepared simultaneously; the recommended capacity for ball milling is 24 preparations (Pl. 2, fig. f) using two double arm shakers.

### **Holding racks for Coors ball mills**

The shaking mill holding racks (boards) are designed for four and six mills. The method described here is for four racks each holding six mills for a total of 24 preparations per run. If additional shaking machines are available, sets of 24 are used consecutively. The brochure from the manufacturer (Coors data sheet GV-22167) gives the method of fitting the mill holding racks to paint shaker machines, hence the following description covers only the sample handling.

### **Precautions during loading of Coors ball mills**

Firstly, it is necessary to mark clearly each wooden shaking rack and the corresponding area where each rack will normally be laid out for loading on a counter or table. This precaution is necessary to avoid intermixing the shaking racks since the position of the rack in its respective area will indicate corresponding samples in the vial holding board (rack). This method leads to quick recovery as marking of the Coors mills with sample numbers is unnecessary, and the mills can, therefore, be placed at random in the rack once washed, etc. Identification of

Coors mills shaking racks can be indicated by drilling different holes in one end of the shaking rack, this will also indicate the correct position of the rack on the counter or table.

The Coors mills must be assembled and loaded with samples carefully if loss of material is to be avoided during the long milling period. An important aspect of this operation requires the immobilization of the central part of the mill during loading of the samples. Next, it is necessary to check for the presence of grains under the sleeve by gently rotating the upper part. When grains are present between the component parts, the cylinder edge may become badly chipped when the assembly is tightened. If damage is extensive some of the sample will escape during milling and cause the balls to impact on the walls of the cylinder. The absence of a powder cushion may lead to permanent damage of the mill.

The best method of loading a Coors mill with a sample is as follows: Firstly, partly assemble the mill with the lower end cap, gasket and cylinder in position. To gauge the correct amount of sample to be added to each mill (15 grams), pour the crushed material into the cylinder approximately up to the gasket level. An inexperienced operator will need to weigh the correct amount and practice the above method. Once mills are loaded, the three balls, gasket, top end cap, and stabilizing sponge (the same size as the shaking board) complete the assembly. To guard against fine leaks of powder, 30 mil soft vinyl gaskets are used. The preparation of these gaskets is described in Appendix II (Pl. 8, figs. d and e). Persistent leaking of the Coors mills may also be due to damaged, uneven or warped, holding boards.

For particular samples, it is recommended that the two end caps be taped together with masking tape; in special cases, in addition to taping, the entire mill can be placed in a light gauge plastic bag. Such precautions ensure that all material will be recoverable and that no cross-contamination takes place.

### Coors ball mills in the shaking machine

For safety reasons, the two rack boards are secured together with a 3/8 inch (9.5 mm) diameter fully threaded steel bolt placed close to the centre of the shaking rack; this also serves to equalize pressure on the mill parts.

When placed in the paint shaker jaws, the assembly is solidly tightened using the shaker clamp and security pin. Maintenance of the parallel alignment of the mills is important, and the assembly must be tightened in such a way as to ensure this. For the fabrication of a wing-nut assembly for the security pin see Appendix II under Coors ball mill shaking machines.

If all of the assembling and mounting steps are carried out carefully the mills generally require no further attention; however, it should be routine practice to retighten the shaker jaws after a short period of milling (and occasionally) to check the operation of the mills.

### Material transfer

On completion of the milling operation, the powdered material is transferred to corresponding vials using a paper sheet placed on a small stand to eliminate contamination from counter dust. The stand also simplifies the transfer of the milling balls directly into a sieve (6 mesh (3.35 mm)) placed near-by. The sieve is then used to wash the balls when the transfer of samples is completed (Pl. 3, figs. a and b).

### Cementation of samples in the ball mills

When sample material solidifies or cements in a Coors ball mill, the sample should be transferred to a 100 mesh (150 mm) contamination-free sieve. The break-up of the caked sample is then done by hand in the sieve. (The hand should be covered with a light gauge plastic bag to prevent contamination.) Lumps are usually crushed against the sieve walls or on the screen. Another method is to mix a few ceramic balls with the sample and then shake the sieve vigorously.

Lumpy or caked material can often be disintegrated directly in a plastic sample vial, especially if a 14 dram (35 x 60 mm) size vial is used. This method consists of adding a ceramic ball to the sample and shaking the vial by hand or in a paint shaking machine for a short time as for a Coors ball mill. A special rack to better secure the plastic vial can be employed if desired.

To control cementation of a sample during ball milling it may be necessary to use a larger amount of material per run, of the order of 30 grams, to extend the milling period.

### Cleaning the Coors ball mills

For samples of similar character, it is sufficient to wash the mill parts under running water. The end caps should be scrubbed with a 1 inch (2.5 cm) circular paint brush from which the bristles have been cut short to stiffen the brush. The cylinders should be similarly scrubbed using a two inch tubular brush. The gaskets should be washed under running water while held between the fingers, and the mill balls can be scrubbed under running water in an 8 inch (20.0 cm) diameter 6 mesh (3.35 mm) non-contaminating sieve.

Another cleaning method for the cylinders is to scrub the interior with one's finger using a plastic bag as a glove. As the cleaning proceeds, the general surface condition will be evident to the finger's feel. Using plastic bags as gloves to handle equipment under running water should be routine in geological sample preparation. When contamination must be strictly controlled, excess water in the end caps is sponged out using wiping tissues (Pl. 3, figs. c and d).

For very precise analytical work metal-free water should be used for washing the ball mill parts and balls; for normal analytical work tap water is satisfactory.

### Quartz and acid cleaning

When cleaning mills between samples with a different lithochemistry, another method is frequently used and is strongly recommended. This procedure is to process pure quartz after each preparation. If this is

not considered sufficient, the end caps and cylinders of the mills can be water washed before bathing in an acid solution. The quartz rinse can be repeated followed by another wash. Excess of water on the gaskets and balls is removed by an air blast (Pl. 3, figs. e and f). Proper cleaning of the mill balls may be inadequate if only water is used, and simple cleaning and washing may be ineffective for some types of adhering materials. Under these circumstances, the balls are best cleaned by rolling or shaking with quartz chips in a porcelain barrel or in some other suitable container. Ultrasonic cleaning apparatus has given excellent results for cleaning gaskets.

### Ceramic balls

New Coors ceramic balls are slightly larger than 5/8 inch (16 mm) diameter and usually last for many milling operations. One way to select balls of proper size is to screen them with a 5/8 inch (16 mm) size sieve. Certain types of geological materials (e.g., quartz) have a marked effect on the wear of the balls. Twenty to fifty pounds of balls should be kept in stock.

Larger ceramic balls, 3/4 inch (20 mm) in size, can also be used, especially if the crushed sample chips are smaller than 5 mesh (4 mm) size. The use of larger balls requires that only one instead of three be used in the Coors ball mills because of size considerations. One disadvantage of using larger ceramic balls is the frequent breakage of mill sleeves. If large samples of more than 15 grams are to be prepared, or if the balls are smaller than 5/8 inch (16 mm), 4 or 5 balls should be used instead of the usual 3.

## **PREPARATION OF SMALL SAMPLES**

### Introduction

It is often convenient to use manual equipment rather than mechanical units when preparing small (1-15 g) samples. Abrasion and cross sample contamination are important factors in the choice of the appropriate equipment in addition to the cleaning methods for each preparation. Frequently, the conventional deep concave agate or ceramic mortar can be replaced with a flat steel or ceramic plate and a plastic tube to better

retain the rock chips (Pl. 4, fig. a). The choice of preparation tools should actually be dictated by considerations of the type of processing required. When a sample is prepared by a large power unit, complete recovery of materials is often difficult due to the retention of some material on the large milling surface area. Small ore samples can be prepared for mineral separations of sulphides, etc., without excess loss of the desired material, with power grinders by premixing the samples with a large amount of quartz or feldspar before processing.

Mineral separation procedures for small samples are discussed in a subsequent section.

### **Crushing with pestle on a steel plate**

In many cases it is best to bypass the difficult to clean power crusher for preparing samples high in specific elements and instead to hand crush them to 3/4 inch (2.0 cm) size on a heavy steel plate and pestle. Pulverization can then be done by the usual power grinder using easy to clean ceramic plates. To avoid contamination at the hand crushing stage a light steel plate, size 6 x 6 x 1/8 inch (15.0 x 15.0 x 0.3 cm), placed on the solid steel base can be utilized; this plate can be discarded after a sample preparation. The pestle is cleaned by grinding the face on a machine shop grinding wheel or on a sand paper sheet.

### **Coors ball mill used as mortar and pestle**

Another method, designed especially for manual fine mesh grinding with minimum contamination of very small samples, utilizes Coors ball mill end caps taped to either the 2" (50 mm) face of a hammer or steel pestle (Pl. 4, fig. b). Once primary crushing is done either by jaw crusher, hammering in a heavy gauge plastic bag, or by pestle and base as above, pulverization is continued using the hand made ceramic face pestle on a ceramic or porcelain flat base. The equipment can be cleaned quickly by pulverization of fine quartz grains, scrubbing the parts with cloth impregnated with corundum and bathing in strong acid.

To prepare very small (1 to 15 gms) samples one can use Coors ball mill parts as follows: a) cylinder as shield, b) end cap as mortar, c) pestle comprising ceramic ball (size 3/4 inch (2.0 cm)) taped to the end of a 3/4 inch (2 cm) diameter, 6 inch (15.0 cm) long steel rod. Once the sample has been reduced to 10 mesh (2.00 mm) size in the end cap, the cylinder and caps can be assembled to complete a Coors ball mill which can then be fitted to a shaker machine. Cleaning is as explained for similar ceramic materials. Samples from which too much material would be lost by ball milling can be powdered by hand in the end cap (Pl. 4, fig. c).

## **PREPARATION OF SOIL, GLACIAL MATERIALS AND DRAINAGE SEDIMENT SAMPLES FOR ANALYSIS**

### **Introduction**

The procedure to be followed in the preparation of these materials depends on the type of analysis to be carried out on the samples. In certain cases complete analysis of the whole sample may be desired whereas in other cases only the minus 80 mesh (180 mm) fraction of the samples is required for analysis. The latter is generally suitable for most types of geochemical surveys using soils, till, and drainage (stream and lake) sediments.

### **General preparation**

- 1) When the whole sample is required for analysis the same procedure as that given for rock samples is carried out beginning at Stage 2. Prior to this all samples should be dried and all lumps of clay, etc., broken up by means of some suitable non-contaminating instrument (Pl. 4, fig. d). Stones and other types of foreign material such as roots should be removed either by hand picking or by a large mesh stainless steel sieve.
- 2) When only the minus 80 mesh (180 mm) fraction is required, the sample, after the lumps in it are broken up, is sieved by an

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<sup>1</sup> Stainless steel sieves with the mesh soldered to the frame by lead-zinc solders are a serious source of contamination. Only sieves in which the mesh is secured to the frame by a friction flange or by epoxy should be used. If these are not available a coating of epoxy over the soldered areas will diminish the hazard of contamination from the solder.



80 mesh (180 mm) stainless steel sieve<sup>1</sup> (Pl. 4, fig. e).

- 3) If the sample is to be analysed by colorimetric methods, atomic absorption methods, etc. no further treatment is necessary. For spectrographic and other specialized analytical work, however, the samples should be ground to minus 140 mesh (106 mm) and ashed at 450°C for four hours to remove any organic matter. Loss on ignition (LOI) should be calculated by weighing the samples before and after ashing.

### **Drying the samples**

When wet samples are received for preparation they should receive immediate attention as certain moulds can rapidly destroy kraft paper envelopes leading to loss of identification number, etc. Samples contained in plastic bags and requiring drying should be transferred to numbered kraft paper bags. If the samples are already in kraft paper bags they need not be opened to dry, although the drying period can be greatly shortened if this is done. Opened bags may be subject to contamination by material which has accidentally escaped from other bags; this should be guarded against. All samples should be air dried in ovens or in suitable drying rooms.

The quantity of hot air circulating around the bags determines the length of the drying period. To obtain free circulation of air, sample handling trays and oven shelves should be perforated or lattice fabricated. Strong netting can also be used as shelves or tray bottoms. If hot ovens or space heaters are used consideration should be given to samples which may contain volatile compounds that may be lost at high temperatures. Whatever method is employed it is good practice to rotate samples frequently and to place fresh samples in the hottest part of the driers. Large numbers of samples drying in a room, may raise the humidity to high levels necessitating extensive ventilation or the installation of dehumidifying apparatus.

### **Sieving and sampling procedure**

The separation of drainage (stream and lake) sediment, sand, gravel, silt and clays, into fractions is achieved by sieving. The sized fraction (minus 80 mesh (180 mm)) is then homogenized, and a representative portion (15 grams) is powdered using a Coors ball mill as described in a previous section. However, some preliminary sample preparation may be required depending on the nature of the sedimentary material.

All sieving should be done in a well ventilated hood (Pl. 4, fig. e). Prior to processing the following steps should be carried out: a) the samples should be readied in storage trays in order of collection; b) opened; c) placed on a flat laboratory truck positioned conveniently such that samples can be handled without loss of time; and d) representative plastic vials marked. Most sieving should be done by hand, using an 8 inch (20.0 cm) diameter sieve and pan. Sieve covers should not be used because of additional weight.

### **Sieving large samples**

Fine mesh screens are frailer than coarser ones and can be easily torn under a heavy loading of sample whether shaken manually or mechanically. Fine mesh screens can be best protected by first sieving the bulk sample through a coarse screen placed above the finer one.

### **Sieving with ceramic balls to break lumpy materials**

Fine materials cementing coarser sediments can be dislodged by intermixing ceramic balls with the sample while sieving, especially if an automatic sieve shaking unit is available. Mixing of balls with the samples (diameter of balls selected depends on screen mesh) is also useful in speeding sieving, especially when using a fine mesh screen to size large samples. The balls vibrate the screen cloth and minimize clogging of the mesh (Pl. 4, fig. f).

### **Mud and clay sample preparation**

Lake sediments and some stream sediments may consist of fine-grained muds,

clays, and gyttja which dry as compact hard masses. These should be crushed or coarse ground mechanically or hammered in a heavy gauge plastic bag (5 mil) (Pl. 4, fig. d). Because of their general homogeneous nature, sieving or mixing is often not required. However, this type of material will often stick to the machine parts and processing of a quantity of quartz may be required between each sample preparation. When a power grinder is used the machine should be fitted with ceramic disks. In the case of small fast rpm crushers, fitted with ceramic jaws and side plates, cleaning is difficult if quartz is not used. Finally, the cleaning operation should be completed by air blasting the equipment as described below under **Precautions to Avoid Contamination**.

## **PREPARATION OF BIOLOGICAL MATERIALS**

### **Introduction**

Biological (biogeochemical) samples of living or dead plant or animal materials are generally collected in paper or plastic bags. In some cases the samples may have been air dried before shipment; if not, they should be unpacked as soon as received and air dried in a well-ventilated, dust-free room. It is particularly important to open samples in plastic bags as soon as possible, otherwise the samples will mould and putrify. Precautions should be taken to avoid contamination of biological materials by dust.

### **Treatment of materials**

Biological samples are generally dried at 80°C for eight hours or until a constant weight for the samples is obtained. This temperature is lower than that recommended for soils or stream-sediments because some organic compounds in animal and vegetable matter are volatile above 80°C. To avoid contamination it is advisable to dry biological materials in an oven used solely for this purpose. Samples in which mercury is to be determined require special treatment because of the extreme volatility of mercury compounds.

After drying, biological samples are generally weighed prior to analysis so that the results can be expressed on an oven-dry basis.

From this point onward a variety of methods for preconcentration of the elements in the samples may be carried out. These include dry ashing, wet ashing, solvent extraction, and specialized chemical preconcentration methods. These are not discussed in this paper because they belong more properly in the field of analysis of biological materials.

A general flow sheet for the preparation of biological materials is given in Figure 2. Briefly the steps are as follows:

**Unpacking:** Unpack the samples and open the bags in a dust-free room.

**Washing:** If the samples are dusty, or if the geochemist or geologist so desires, the biological materials may be washed in metal-free water containing a small amount of non-contaminating detergent.

**Splitting:** Split the samples into portions and number them a, b, c, etc. The number of portions will depend on the type of analyses required and will be determined by the geochemist or geologist for whom the work is done.

**Drying:** Dry the various portions (subsamples) in an oven at 80°C for eight hours or until the samples attain a constant weight. Avoid drying the biological samples with soils or other geochemical samples. It is best to use an oven specially set aside for biogeochemical samples. This step should be omitted for samples in which mercury and other highly volatile elements are to be determined.

**Weighing:** Weigh each of the subsamples and record the weights as oven-dry weights.

If desired by the geochemist or geologist the subsamples may be ground or milled or pressed into pellets prior to ashing.

Grinding with an agate mortar and pestle may be necessary in special instances, such as in analysis of molybdenum.

**Contamination precautions:** The procedure for milling plant materials is given by Ward and Johnson (1962). They recommend

a mechanical mill and stress that it should be thoroughly cleaned between the grinding of individual samples.

Biological materials should be prepared in a room entirely separate from those where rocks, soils, and ores are prepared.

**Pellet preparations:** The method for pressing samples into pellets is described by Ward et al. (1963). Briefly, it consists of drying the organic material at 60°C, grinding it in a Wiley mill, taking 5 or 10 grams of the material, and compressing it into a pellet by means of a stainless steel cylinder placed in a press. The advantage of using pellets is that more samples can be dry ashed simultaneously with greater ease, there is less chance of contamination, and more uniformity of dry ash. Pellets are also more convenient when carrying out wet ashing procedures and are imperative for neutron activation analysis.

**Coors ball mill preparation of dry biological samples:** A satisfactory method of preparing dry leaves, buds, twigs, and needles is to pack them in a Coors ball mill with three balls and shake as for the preparation of rock powders. Alternatively a swing (gyratory) grinding mill using non-metallic (ceramic or agate) grinding parts can be employed.

## **MINERAL SEPARATION AND CONCENTRATION BY HAND PANNING, SUPERPANNER, HEAVY LIQUIDS AND MAGNETIC METHODS**

### **Introduction**

Mineral concentrates are prepared from geological materials for the following purposes: a) to locate the mineral or minerals in a rock, soil or sediment responsible for a particular element or elements, b) to use minerals as indicators of mineral deposits in geochemical (heavy mineral) surveys, and c) analysis of a mineral separate for its constituent elements. The following describes a method of mineral separation, utilizing specific gravity and magnetic susceptibility, that has been developed after many years of experience.

When specific gravity and magnetic susceptibility fail to give satisfactory separations, other methods utilizing flotation

techniques can be applied. If these fail hand picking of individual mineral grains should be employed. Both these methods are described in the next two sections.

A flow sheet illustrating the preparation of mineral separates and concentrates according to the methods described below is shown in Figure 3.

### **Representative sample**

Prior to concentration and separation of minerals, the material should be processed according to its physical nature. Unconsolidated materials such as sand, gravel, lake and stream sediments need only be dried and sieved. Heavy mineral pan concentrates made in the field will generally require further sizing. In the laboratory, the geological materials are dried and resieved to a much finer mesh; for most unconsolidated material sieving is usually to -80 mesh (180 mm) or if required to -100 mesh (150 mm) or finer. It is good practice to retain a representative sample of the -80 mesh (180 mm) fraction (15 g) in case a general chemical analysis is required.

### **Mesh size**

To free the mineral grains from one another in solid rocks it is necessary to crush and grind the material as described in a previous section for routine chemical analysis with the difference that reduction should be staged to eliminate production of excess fines (slimes). Reduction can be either by grinding with steel or ceramic plates. Steel plates have the disadvantage of introducing steel particles into the sample; if this is to be avoided ceramic plates must be used. (To protect ceramic plates refer to Appendix III.) Preparation of samples by ceramic plates reduces the amount of fines, a feature important in mineral separation.

If only small samples (15-30 grams) are available they are best prepared in Coors ball mills. One to three balls can be used depending on the size of the rock chips or grain size of unconsolidated materials (e.g. till, sand, etc.). To obtain the maximum amount of material for mineral concentration at 100 mesh (150 mm) the Coors ball mills

should be shaken about one quarter of the normal time required for other preparations.

Individual minerals are usually liberated from rocks, sand, etc. at 80 mesh (180 mm). For some types of materials a smaller mesh size (100, 140, 200) (150, 106, 75 mm) is more appropriate. The actual size can be determined by microscopic methods. The size of grains found most effective for water panning of concentrates is 140 mesh (106 mm). To obtain a sufficient amount of sample at this particular mesh size it is routine practice to use size 100 mesh (150 mm) for the larger grains and 200 mesh (75 mm) for the smaller grains and the intermediate size for mineral processing.

Preparation generally consists at first of grinding the sample two or three times to a size larger than required (usually 100 mesh (150 mm)) and sieving the product. The oversized material is again ground with a slight tightening of the disk-gap on the grinder. This grinding by stages and sieving is repeated until most of the sample has been reduced. When an automatic sieve shaker is used the 200 mesh size (75 mm) sieve can be nested to remove the finer products. If sieving is done by hand, individual sieving is usually preferred. When larger sieves are used (e.g. 24 inch (65 cm) diameter), and shaking is by a gyratory motion machine, internesting of the two sieves may be impractical as the sample in the top sieve may become motionless because of the total weight.

For most mineral separation it is unnecessary to remove the finer material by sieving as this can be done by washing with water as explained in a following paragraph. For unknown materials it is best to process small test batches sized at different fractions; e.g., -60 + 80 (-250 + 180), -80 + 100 (-180 + 150), -100 + 140 (-150 + 106), -140 + 200 (-106 + 75), -200 + 230 (-75 + 63), -230 (-63) mesh, all measurements in brackets being in mm. To protect fragile sieves refer to the section on: **Preparation of soil and drainage sediment samples for analysis.**

#### Mineral separation by specific gravity

The specific gravity properties of mineral grains should be utilized first to

concentrate or separate the minerals because of the large amount of basic information that is immediately available. When either the -80, -100, or -100 + 200 (-180, -150 or -150 + 75 mm) mesh fraction of the unconsolidated materials fraction of prepared solid materials is used the fraction should first be washed in a large beaker of water. For small samples (1 to 15 grams), or samples with certain minerals, e.g., molybdenite or pyrite, acetone should be used in the place of water. This particular step is designed to remove the adhering superfine and light grains which are not usually of much use. The procedure usually employed is to vigorously stir the slurry in a 4000 ml beaker and allow it to settle for 10 seconds. The floating materials (the lowest specific gravity and very fine sized grains) are then carefully decanted to leave the sinks (heaviest specific gravity and largest sized grains). This technique should be repeated three or four times or until the water becomes clear (Pl. 5, fig. a).

#### Superpanner

The superpanner offers many advantages for separating mineral grains. These are:

- 1) Mineral grains can be easily separated, provided differences in specific gravity of (1.0 or more) exist in the suite, and provided they can be separated by crushing. Some minerals, however, may require a larger separation interval in their specific gravity because of their shape, cleavage, composite character, and repellant action toward water.
- 2) The actual specific gravity of separated grains can be estimated and this provides primary information that may indicate the identity of the mineral grains.

The Haultain Superpanner takes full advantage of the specific gravity properties of mineral grains. This apparatus is capable of concentrating any type of mineral provided significant differences in specific gravity exist. The water in the superpanner is agitated mechanically, by various eccentric and off-centered pulleys and shafts located at



the pan front and side, together with an adjustable tilt (Pl. 5, fig. d).

The washed sample is transferred to the superpanner and shaken for a few minutes in an excess of water. The pan is then tilted while a stream of water runs down the pan; the lighter grains travel along the pan where they may be removed by suction, and the remaining grains form into a tip which is zoned into heavies, middles and lights according to their density. The heavies are concentrated at the head of the tail.

### Heavy grains

It is common practice to check at the final panning stage for an extremely heavy concentrate referred to as the "superpanner tip". This will appear at the head of the heavy concentrate and can only be detected by close inspection because the tip is usually composed of a few grains only. When the general panning procedure does not produce a tip, a second test should be made by first bringing all the material back to the front of the pan, shaking in a large amount of water, and then allowing it to proceed down the pan as in the first test. The tip, if any, should appear at approximately 1 inch (2.5 cm) from the head of the pan. Occasionally it is necessary to move (slide) materials a little further down the pan by making a small tilt adjustment to the pan and by manually separating with the fingers most of the lights from the heavies in order to concentrate and isolate the few heaviest grains. Minerals such as gold, electrum, uraninite, and galena behave in this manner. Usually the heaviest concentrates are removed first; reprocessing then permits the other heavies and lights to be removed later (Pl. 5, fig. b).

A simple sketch should be made of the order of separation of the minerals. A record of colour separates observed during panning is also useful for future reference, especially if mineral identification is to follow.

### Grain collector pipette

The apparatus commonly used to remove small concentrations of material in water is a specially designed mouth pipette (Pl. 5, fig. f). Larger concentrates are removed by a

suction hose discharged into a large beaker or by a gravity syphoning system.

### Separating the ferromagnetics

Ferromagnetic minerals can be removed from the pan by hand magnet usually while panning is in progress. The removal of the magnetic fraction is achieved by a specially made aluminum shelled magnet or by holding a hand magnet inside a light gauge plastic bag (Pl. 5, fig. c) and discharging the material into a container of water which should be immediately drained and the sample of concentrate acetone- washed to prevent oxidation.

### Water repellant minerals

Certain categories of mineral grains may be difficult to wet and sink, for example, the sulphide group. This problem can be overcome by mixing a few drops of liquid detergent with the water used during the panning. If this procedure is frequently required, a dispenser can be organized to automatically discharge drops of detergent solution at the superpanner front.

### Heavy liquids (methylene iodide and bromoform)

Flat grains of minerals such as gold, molybdenite and mica are poorly separated by means of panning in water mainly because air bubbles attach themselves to the grains even when totally submerged. Heavy liquids are usually used to separate such types of mineral grains. The liquids most commonly used are methylene iodide and bromoform with specific gravities of 3.32 and 2.84 respectively. The higher density liquids are not recommended because of their extremely toxic and volatile properties. When used in large quantities toxic heavy liquids require well-ventilated hoods for health reasons, principally the control of fumes. For faster filtration following separation, a vacuum pump should be used (Pl. 6, figs. 4a, 4b and 4c).

### Transfer of a few mineral grains

Small quantities of mineral grains can be transferred without loss to a final storage vial followed by drying. The general procedure is to remove water and excess

heavy liquid from materials with acetone, and then to wash the mineral grains into a glass vial by a stream of acetone followed by drying (Pl. 6, figs. 1a and 1b). The small glass vial should be stored in a plastic vial for easy labelling and identification.

### Heavy liquid loss

The use of heavy liquids can be expensive because approximately 15 percent of the heavy liquid is lost when separating 100 mesh (150  $\mu$ m) size material if filtration is vacuum assisted, and 25 percent when only gravity is employed.

Take for example a group of sandy stream and lake sediment samples sieved to minus 80 mesh (180  $\mu$ m) and yielding a fraction weighing 100 lbs. (45 kg) which is to be separated by methylene iodide. If filtering of the liquids is vacuum assisted, the liquid loss would be approximately 15 lbs. (7 kg). On the other hand, if the liquid is gravity filtered the loss would be about 25 lbs. (12 kg), almost double the first amount, even with good recovery methods.

### Separation by liquid densities

Lower specific gravity liquids can be made by mixing less dense liquids of low volatility such as dimethyl sulfoxide with bromoform, etc. Heavy liquid suppliers usually list the most appropriate liquids and the approximate ratio for use in preparing liquids of a given specific gravity.

Separations of minerals are only possible if the range of the heavy liquid densities permit a sink (s) and float (F) fraction of the mineral grains to be developed in a relatively short period of time. Centrifugation can frequently be employed to hasten the process.

When the heavy liquid density range is lower than that of the suite of mineral grains the superpanner, flotation, and Frantz Isodynamic Separator is used to complete the separation. Generally, heavy liquids are used to separate unsized material, establish precise heavy and light percentage ratios, separate flaky minerals, establish specific gravity of a particular mineral or group of minerals, or when a superpanner or other specific gravity apparatus is not available.

### Heavy liquid quality

Old heavy liquids, or liquids which have been contaminated with other liquids, are susceptible to formation of crystalline precipitates resulting in a slow filtration.

### Heavy liquid recovery

The recovery of heavy liquids (e.g. methylene iodide) contaminated with lighter liquids (e.g., acetone) can be done by simple evaporation. Thus, methylene iodide has a much lower vapour pressure compared to acetone, and hence the greater proportion of methylene iodide will remain behind and be recovered. Another method is to mix the liquids with water and to separate them in a separatory funnel after the combination has been vigorously shaken and allowed to settle. Discoloured heavy liquids can be clarified by shaking with activated charcoal, and in extreme cases by fractional distillation procedures.

### Separating large samples

When large samples are separated by high specific gravity liquids, e.g., methylene iodide, it is recommended that a first pass be made with liquids of lesser specific gravity, e.g., bromoform. In the long run this procedure leads to more economical and more efficient separations.

### Mineral separation by magnetic methods

When the specific gravity method is not effective in separating mineral grains from a matrix, the magnetic susceptibility properties of minerals can often be exploited. The Frantz Isodynamic Magnetic Separator (Pl. 5, fig. 5) is most effective in separating small amounts of material. Table 1 gives the magnetic susceptibility of a large number of minerals.

The following paragraphs describe only the general handling and necessary precautions for using the Frantz Isodynamic Separator.

Prior to all electromagnetic separations using either a Frantz separator, or drum or belt types, it is essential that the highly ferromagnetic materials be removed by hand

using a permanent magnet. This particular precaution is necessary to avoid material clogging the separating chamber of the separator, resulting in material loss. Use of a light gauge plastic bag to hold the hand magnet will prevent material sticking to the magnet.

When using magnetic separating machines, the material is first passed at the lowest magnetic field strength; the non-magnetic fraction is then repassed with a small magnetic field increase. For the Frantz separator the following steps are usually routine: 0.01, 0.1, 0.2, 0.3, 0.5, 0.8, 1.4, 1.8 amp. The setting at 0.01 amp will remove any ferromagnetic material not removed by the application of the hand magnet.

With time the Frantz separator loses some of its strength; this can be partly compensated by increasing the input voltage using a transformer. However, it is recommended that a time clock be included in the circuit to limit periods of use at higher voltages; this avoids possible overheating of the electromagnet coil. The Frantz Isodynamic Separator is normally cleaned by brushing, followed by an air blast.

#### Mineral separation by froth flotation methods

Certain mixtures of minerals can often only be separated by froth flotation methods. This is especially true when the physical properties such as specific gravity or magnetic susceptibility are similar for each constituent mineral. Certain types of flaky minerals (e.g., molybdenite) are ideally suited to froth flotation separation.

Basically the froth flotation procedure consists of stirring the water-mineral mixture at high speed in such a way as to aerate the hydrous mixture forming a stable froth when a pre-selected frothing agent and a collector agent are added. The froth overflow containing the specific mineral to be separated is gathered in a suitable container.

#### Mesh size

The most suitable mesh size is the same as for the previously described separation methods, i.e., -100 mesh (150 $\mu$ m), although

smaller sizes have also been found to be satisfactory.

#### Simple flotation cell

Extraction of the froth can be expedited if a cell is designed with a separation wall capable of sliding vertically. This adaptation allows the froth to pass under the wall into a tranquil zone where it surfaces and pours into a container. A separation wall is only used in the smaller cells where turbulent mixing affects the whole cell. The finer grains (140 mesh (106 $\mu$ m)) are readily collected by the froth formed by stirring; the larger grains may require flotation by introduction of a stream of air bubbles (Pl. 6, fig. b). Such a type of cell has only a limited separation capacity.

#### Flotation by Denver flotation machine

The following method is based on the use of a Denver Model No. D-2, "Sub-A" flotation machine (Pl. 6, fig. c).

Experience has shown that the 8000 ml, 2 kilogram capacity size tank is best for all processing even when the sample is small. With careful handling, the loss of grains is minimal.

#### Molybdenite concentration

The 8000 ml size tank is half-filled with cold water and a bulk sample (up to 2 kg ground to -100 mesh (150 $\mu$ m)) is poured into the water. The tank is then filled to within 1 inch (2.5 cm) of the discharge spout.

Machine speed is set at 2,000 rpm, and the tank is stirred for a few seconds and the machine is then turned off. Frothing agents Shell Chemicals MIBC (2 ml) and Hercules Yarmor F (2 ml) are added to the slurry. Immediately the machine is reactivated at the same speed. After a few seconds the aerator is slowly opened. Molybdenum concentrates should surface with the accompanying foam and be discharged. A long spatula is generally necessary to move the bubbles towards the discharge point. Experience indicates that the time required to complete a satisfactory concentration varies from 3 to 10 minutes.

### Examination of the concentrate

After a run, tailings in the processing tank can be discarded or transferred to large containers (beakers). The concentrate may then be returned to the processing tank to be reprocessed if necessary. Frothing agents MIBC and Yarmor F do not need to be readded. It is recommended that a small amount of the concentrate be examined under the microscope at this point. Normally, a few grains can be obtained during the last part of the transfer of the concentrate to the reprocessing tank. Once the few grains to be examined are in a beaker they can be acetone dried. A mineral purity record should be kept in order that progress can be compared between each reprocessing run.

The transfer to a container is achieved by a water jet.

### Purification of float concentrates

Once flotation is completed, the concentrate is transferred to a large beaker and mixed with a dewatering agent. Normally the sedimentation period is 1 hour. If the concentrate contains very fine undesired materials, purification can be accomplished by gyratory washing and decantation techniques usually in a 4000 ml beaker with acetone. The acetone washing and decantation is repeated until the acetone is clear at which point the suspensions are removed. The acetone can be filtered and recovered for further use.

Occasionally the dewatering agent will not promote effective wetting and settling of the grains. In such cases, vacuum filtering is recommended.

Next, the purified flotation concentrate is passed through the magnetic Franz separator to remove the iron sulphide (pyrite if containing particles of magnetite, pyrrhotite, etc.) and oxides (magnetite, ilmenite, etc.) grains. The magnetic separator will assist in disaggregation of composite or adhered silicate grains which may be present; this will reduce the remaining impurities to a minimum and result in a better separation by heavy liquids.

### Barite or zircon separation from pyrite

The bulk sample containing the minerals of interest, barite, zircon, or pyrite, is first superpanned using a relatively large quantity of liquid soap. The heavy mineral concentrate is removed, acetone washed, dried and introduced into the flotation cell. The procedure for the flotation of pyrite is similar to that for molybdenite. The treatment with soap and acetone leaves a thin film of the soap on each grain when dried. Pyrite is greatly affected by such a treatment and is immediately floated clear of barite or zircon by the froth.

### Cleaning of mineral grains

The concentrates should be washed with acetone either directly in beakers or by using filter papers assisted by vacuum apparatus. When ultrasonic cleaners are used the concentrates should be first washed with a water solution of liquid soap followed by acetone to remove all remaining traces of flotation agents and heavy liquids employed during the separation of the minerals.

### Micro sized samples

If the bulk sample is small (15 grams or less) and losses cannot be permitted, the material should be washed in a beaker using acetone after it has been ground to pass a 100 mesh (150 $\mu$ m) sieve. The acetone washing will remove most of the light and superfine (slime) grains. The remaining concentrate is then passed through a Frantz separator and further separated by heavy liquids. Flotation may be used, but further processing may be difficult because of the small amount of resultant concentrate. In some cases certain unwanted grains can be removed by chemical treatment.

### Purification of concentrates

A concentrate can be purified by alternatively repanning and refrantzing a particular fraction. Since the superpanner response of a mineral grain depends essentially on the size (140 mesh (106 $\mu$ m)) it is important that materials be carefully sized over a narrow range. Superpanned concentrates are dried, and in the case of



separation by Franz Isodynamic Separator, a particular fraction is selected for processing. When flotation or the heavy liquid methods of separation are used, the recovered heavy concentrate is washed carefully in acetone before drying to prevent grains from sticking together once dried.

#### **Concentration of mineral grains according to their hardness**

Reducing material gradually both by power grinder and manual methods may be necessary in some cases. Thus, when a solid which contains both soft and hard grains is crushed the softer mineral grains are subject to immediate comminution after a few passes leaving the harder mineral(s) in the coarser fractions. A partial separation can be made by sieving the product. The hard fraction is then returned to the grinder for further processing.

Micaceous minerals can be concentrated in the same manner; flat grains are recovered by frequent sieving as the sample is being reduced.

When ceramic balls are shaken with coarsely ground material, containing both soft and hard minerals, in a stainless steel sieve the softer grains will be reduced more readily than the harder ones which remain in the sieve.

#### **Manual concentration of mineral grains**

Occasionally minerals can be separated only by hand picking. Since reduction is usually with a mortar and pestle, some time can be saved by discarding the undesired material as the sample is reduced. The limit is reached when the mineral grains can only be separated on a glass plate using a low power microscope.

An engraving machine is useful for the removal of a particular mineral present along veinlets or in other similar sites. The vibrating pin will dislodge the desired mineral, and further purification can be continued by other means. Occasionally, when mechanical methods of mineral separation would be considered impossible, such separation methods can be tried.

Whenever possible the same person should perform the work at all mineral separation stages. This ensures uniformity of results through individual familiarity with the minerals in the sample, their behaviour and other aspects of the desired mineral separates.

#### **PRECAUTIONS TO AVOID CONTAMINATION**

##### **Specific sources of contamination**

Contamination from sample to sample, from samples with high concentrations of specific elements, from crushers and grinders, from other preparatory equipment, and from sample bags, bottles, etc. must be avoided. Checks for contamination at all stages of sample preparation should be carried out frequently to avoid costly repetitive work. Water used for washing the various types of crushing, grinding, milling, separation, etc. equipment and apparatus is normally tap water. Metal-free water should be used if very precise analytical work is required on the samples. Listed below are a few points that will assist in avoiding contamination, cleaning equipment, etc.

##### **Selection of machines**

The choice of sample preparation equipment depends on the requirements for rapid turn-around and quality of processing. At present, commercial companies offer various models of laboratory crushers, grinders, separating tables, etc. with interchangeable milling parts which can be selected according to the physical and chemical nature of the samples. As required these machines can be opened or dismantled completely for cleaning the jaws and interchange of milling parts.

##### **Cross sample contamination**

Samples of ores or materials with a high concentration of any specific element must not be milled in the same equipment as those for normal rocks, soils, till, drainage sediments, etc. In fact, two separate sets of equipment in different rooms, one for ores

and the other for rocks, soils, till, drainage sediments, etc. must be provided.

### Equipment contamination

The crusher and grinder are the principal sources of contamination in the preparation of rock, soil, till, and ore samples. When a mortar and pestle are used these may also be sources of contamination. In general, a very low (commonly negligible) amount of contamination in the form of steel flakes is introduced into rock and other samples by the crusher jaws. Relatively little can be done about this minor amount of contamination except to make sure that the plates of the crusher are not set too close, and to avoid if possible repeated abrasion by the jaw plates on the larger fragments of rock.

Considerable amounts of steel particles, containing various elements for the hardening of steel (e.g. Mo), are introduced into rock and soil samples by grinders with steel plates. For certain types of work this is not serious. For other types of work, especially trace-element work, ceramic grinding plates are essential. These introduce a little alumina and insignificant amounts of other elements into the samples.

### Grinders

The grinders are cleaned according to the sample composition and physical size. For samples of similar chemistry or of large size the milling chamber is air blasted using a high pressure stream of air from an air line (1 inch (2.5 cm) diameter, 30-60 psi) or vacuum cleaner rear air flow directed upon the grinding plates, chamber, etc. as the parts are being brushed. For samples of dissimilar chemistry, a quantity of quartz is processed between each preparation; this usually thoroughly cleans the plate surfaces when followed by air blasting as described above. Often it is essential to remove the plates and to air blast the plate-holding flanges to remove any lodged grains.

In some laboratories it is conventional to use a sets of plates according to the chemistry of the samples.

Whenever a blast of air is used to clean the machines an appropriate dust hood with good suction is a prerequisite on each of the

machines; otherwise the machines should be cleaned with a vacuum cleaner having good suction.

If the power supply available permits, a reversible switch should be installed on the grinding machine making the rotation of the motor reversible; this allows cleaning both sides of the disk grooves and also ensures that grooves wear evenly on both sides.

### Crushers

Crusher jaws should be cleaned with a steel wire brush as an air stream (air pressure and hose arrangement as for grinder) is directed on the jaws. After this treatment the jaws should be wiped with a damp cloth and immediately dried with a dry cloth.

Crushing machines comprise numerous parts which can be a source of contamination, especially when samples with an unusual elemental chemistry are prepared immediately before those which are normal. Small chips have a tendency to lodge between the parts and cannot be removed by the routine cleaning described above. The best way to clean a crushing machine under these circumstances is to dismantle it and clean each part individually. When this technique is not practical, cleaning can be accomplished by passing a large quantity of quartz through the machine, followed by brushing and air blasting the chamber. An alternative method is to replace the jaws with a pair used routinely for normal rock samples.

Tough fine-grained rock samples such as hornfels, certain varieties of metavolcanic rocks, and cherts are generally difficult to prepare without breakage of and contamination from the crusher jaws and grinding wheels. To minimize contamination and yield better results mild steel movable and stationary jaws together with surface plates should be fitted to the crushing machine in place of any cast-iron parts. In addition to these modifications for the crusher it has been found desirable to use ceramic plates rather than steel plates on the grinder for the preparation of tough fine-grained rocks. To prevent excessive breakage and cracking of the ceramic plates they should be reinforced with a steel ring as

described by the writer in a previous publication (Lavergne, 1965).

### Coors ball mills

Ceramic ball-mill cartridges and balls should be washed under running water or in water containing a little non-contaminating detergent, drained and dried after each sample (Pl. 7, fig. a). If this is not satisfactory pure quartz sand should be milled in the cartridges for a short time followed by washing in water. During the milling of the pure quartz sand the frame holding the cartridges should be reversed at least once for thorough cleaning of the cartridges. An occasional bath in boiling concentrated hydrochloric acid or in aqua regia followed by rinsing in metal-free water will clean both ceramic balls and cartridges thoroughly and give them a fresh appearance. This latter type of treatment is recommended when all traces of contamination from one sample to another are to be eliminated.

Gaskets are generally cleaned under running water but can also be cleaned in ultrasonic apparatus. Here again having gasket sets suitable to sample composition is recommended.

### Sieves

For most types of trace-element work stainless steel sieves with the stainless steel screens attached to the frame by friction or by epoxy should be used. Screens and covers soldered with soft metal (e.g. lead, zinc) may introduce serious contamination and should be avoided.

The screens of sieves can be cleaned with a stiff paint brush whose hair has been cut to a length of approximately 1 inch (2.5 cm). A forced air blast is also useful in cleaning sieves, although in many cases the grains tend to stick in the screen and have to be dislodged with the paint brush or in certain persistent cases with a needle. For certain types of work where contamination is to be reduced to a minimum the stainless steel screens should be washed carefully in metal-free water and dried immediately after each sample.

Screens should be cleaned after each preparation depending on the chemical nature and physical size of the sample processed. Tapping the sieve and pan together is sufficient for certain samples, whereas others require air blasting, tapping the sieve on a solid base, washing under hot running water, ultrasonic cleaning and brushing. Heating the screen of the sieves will dilate the pores; therefore before dry cleaning it is best to heat the screen for a short period of time over a bank of heat lamps whose heat is electronically controlled. One method, only occasionally used because of possible screen damage, is to process a quantity of coarse ground quartz between samples. Another method is to have a selection of sieves for each type of sample composition or chemistry e.g. routine chemical work, trace element analyses, or macro-element chemical analyses, etc.

### Superpanners

The stainless steel pan of superpanners is best cleaned with very fine grained silica or scouring powder. This treatment should be carried out frequently to maintain the pan in a sparkling condition.

### Magnetic separators

Magnetic separators and other apparatus used in mineral separations are generally cleaned with a paint brush and an air blast. After cleaning, the electro-magnetic separators should be allowed to vibrate for a short time to remove any particles still adhering to the operating parts.

Couplings, chute supports, or any other parts which may contaminate the sample as a result of wearing should be made of non-metallic materials such as aluminum or nylon.

### Sample preparation room

Preparation of more than one type of sample at the same time in the same room should be avoided, because chips or dust from one type of sample may contaminate others.

### Special precautions

To strictly control contamination during the preparation of special samples certain

parts or materials (e.g., nylon screens) must be discarded after each use. When the equipment is difficult to clean properly (e.g., crushers and grinders) a second set of crushers and grinders should, if possible, be set-up in a separate room. For instance, a room should be retained for samples requiring trace element analysis, i.e., those averaging less than 0.5% of any specific element and another for ore grade materials averaging more than 0.5% of the specific element. In some laboratories it is advantageous to have a third facility with all necessary equipment to prepare materials for special projects such as age determinations.

The risk of contamination of biological materials is much greater than in the case of rocks, soils, till or drainage sediments, owing to the high factor of preconcentration involved in the analysis of the biological materials. For example, suppose a small flake of molybdenum lubricant containing 10 micrograms molybdenum inadvertently falls into a 10 gram soil sample containing 10 ppm molybdenum. If the sample is homogenized and subjected to chemical analysis the result will be about 10 per cent higher, owing to the contamination, which is not significant in many cases. If the same molybdenum-bearing flake falls into a 10 gram sample of oven-dried plant material, and the plant material is then dry ashed and subjected to a chemical preconcentration step a forty-fold increase in the molybdenum content of the sample can be expected. This is admittedly an extreme case, but it does focus attention on the need for care in the preparation of samples of biological materials.

### Dust hoods

All crushers, grinders, and tables for mixing, rolling, splitting, and sampling the ground materials must be fitted with dust hoods, both for health reasons and to avoid contamination of samples by the dust, which otherwise would spread around the sampling room. If the machines are blasted with air after each preparation the resulting quantity of dust is much greater than during actual grinding; hence great care must be taken in the choice and installation of suction fans, dust collectors, etc.

In general it is preferable to have separate ducts and suction fans for each type of machine and for each preparation table, or to have one large suction fan and central duct with cut-off dampers in each of the subsidiary ducts serving the various machines and tables. The latter arrangement facilitates better cleaning since the suction can be increased at one site by cutting off all of the other ducts. A sketch of duct hoods for crushers, grinders and mixing tables using minimum space is shown in Plate 9, fig. a.

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## APPENDIX I

### GUIDE FOR THE IDENTIFICATION OF COMMON MINERALS

#### Introduction

Equipment used to prepare geological materials for general analysis can often be effectively employed to give a large amount of information useful in the identification of an unknown mineral. For example the superpanner can give an estimate of the specific gravity of each mineral in a sample, and the magnetic separator (Frantz) will give indications of iron or chromium content. If all these data are combined and compared with descriptions in a mineralogy textbook, the possibility of identifying an unknown mineral is enhanced.

To retain the full benefit of the information available from the preparation machines, known minerals are processed first and their behaviour is closely observed. This method is applicable to all geological materials including rocks, soils, till, drainage sediments, etc. (Pl. 8, figs. ai, aii and b).

#### Grain size

Experience has shown that minerals can be more easily identified when very small. The optimum size for identification purposes is -80 + 100 mesh (-180 + 150  $\mu\text{m}$ ). This particular size is best because when the mineral fragments are glued in epoxy a sufficiently large fresh surface can be left exposed for scratching with a needle point. A binocular microscope having a magnification factor of about 50X is usually adequate to observe the behaviour of the scratching procedure. If minerals are present in sizes larger than -80 + 100 mesh (-180 + 150  $\mu\text{m}$ ) they should be crushed to this size as explained in the section on mineral separation.

#### Containers

To keep the test materials free from dust, contamination, and to avoid loss or

identification, they should be placed in containers such as 14 dram, clear plastic vials held in specially designed racks or trays.

#### Mineral preparation

Once a set of known minerals has been obtained for identification purposes they should be prepared as follows:

- 1) The mineral sample is split, and half is kept in a vial, the name of the mineral is marked on the side of the vial and an assigned number is marked on the cap.
- 2) The other half of the mineral sample is crushed to pass a -80 mesh (180  $\mu\text{m}$ ) screen.
- 3) The fines (dust) are removed using a 100-mesh (150  $\mu\text{m}$ ) sieve and the -80 + 100 mesh (180 + 150  $\mu\text{m}$ ) material is placed in numbered vial. About 2 or 3 grams of the -80 + 100 mesh (-180 + 150  $\mu\text{m}$ ) size fraction is sufficient for identification purposes; this amount of material should be kept in a numbered vial on which the name of the mineral is marked.

An 80 mesh (180  $\mu\text{m}$ ) screen (6 or 8 inches (15.0 or 20.0 cm) diameter 3 inches (7.5 cm) high) is mandatory in all preparations and should be obtained from a specialist supplier. One method of in-house fabrication is to seal a metal or nylon screen to plastic or metal tubing.

A 100-mesh (150  $\mu\text{m}$ ) screen is not particularly critical, although if fabricated to permit nesting of the two screens (80 and 100 mesh) (180 and 150  $\mu\text{m}$ ) allow easy collection of grains of uniform size. If a 100 mesh (150  $\mu\text{m}$ ) screen is not readily available, most fines can be removed by washing and decanting with water. The process is repeated as necessary. The

unconsolidated materials can simply be sieved to pass a -80 mesh (180  $\mu$ m) screen.

### **Study of grains glued to a plexiglass plate**

A rapid method of becoming familiar with minerals is to place a few of the prepared identified (known) grains on a plexiglass plate and to examine them under a microscope. A good permanent mount can be made by placing a drop of epoxy glue on the plexiglass plate and gently anchoring the grains in the epoxy with one's finger leaving a fresh part of the mineral exposed. Sample numbers are inscribed or affixed on the side of the mounts. Once firmly mounted the minerals are ready for study (Pl. 8, fig. ci).

### **Hardness number using window glass as a gauge**

The approximate hardness of a mineral grain can be determined by placing a few -80 + 100 mesh (-180 + 150  $\mu$ m) grains on a plate of window glass and crushing them with a flat-ended sewing needle while viewing the operation under a microscope. The condition of the crushed grains once crushed is characteristic and informative.

A quartz mineral grain is slightly harder than window glass and thus marks the surface when crushed on it. Because of its common occurrence and association with many other minerals it can be utilized as a "hardness standard". As each mineral grain is broken on a plate of window glass it can be assigned a hardness number, more or less relative to quartz. The glass plate is therefore a "hardness gauge" (Pl. 8, fig. cii).

### **Study of mineral grains by microscope**

When the surface of a mineral grain glued in epoxy is scratched or when the mineral is crushed on a window glass plate while the operation is viewed under a microscope much information can be obtained. For those not familiar with minerals the exercise should be repeatedly performed to familiarize one with all the details.

To obtain full benefit of the method for the identification of a unknown mineral, the known and the unknown mineral should be

crushed side by side with a flat-ended needle on plate glass. Comparison of the hardness of the two minerals can be evaluated as well as the color, streak, crystal form, etc. of the unknown. For good results 3 to 5 very pure mineral grains are isolated from the others and crushed very carefully on a clean part of the plate glass.

For such a study a good microscope is important. Models with interchangeable lens, zoom lenses, or combinations of both having magnification factors up to 100X are recommended.

### **Magnetic susceptibility identification method**

To test the magnetic character of a mineral, a few grains of the -80 mesh (180  $\mu$ m) size are placed on a plate of window glass and a magnetized sewing needle is brought close to the sample. The behavior of a grain as observed under the microscope gives an indication of its magnetic properties and some clues as to its identity.

A large powerful magnet (Frantz) will separate grains having a minimal iron content. This is accomplished by controlling either the amperage or the voltage.

Figure 4 shows an ideal mineral fractionation scheme according to the magnetic field strength of the Frantz isodynamic separator. Plate 8, fig. ciii shows mineral grains on a plexiglass sheet after subdivision by the Frantz separator.

### **Finding the relative specific gravity by panning**

The concentration and separation of minerals by panning gives information about the relative specific gravity of minerals. If one mineral in a sample can be identified, some knowledge of the specific gravity of its associates can be inferred.

### **Haultain superpanner and prospector's pan (gold pan)**

The Haultain Superpanner is ideally suited for determining the relative approximate specific gravity of mineral grains. The shaking period for optimum separation is approximately 3 to 10 minutes depending on the nature of the minerals

present and on the purity of separates desired.

The efficiency of a superpanner or prospector's pan (gold pan) depends essentially on the uniformity of grain size since specific gravity determines the mass of the particle. Careful sieving to a specific size is essential to achieve the best results when determining the range of specific gravities of minerals present in a sample.

The minerals with highest specific gravity will move to the front of the pan of the superpanner, others will follow in their order of decreasing specific gravities. The heavies are usually referred to as the "tip", intermediates as "middlings", and the lights as "tailings". It is important to carefully watch for the presence of "super tip" throughout the separation to detect the occurrence of minerals such as gold, platinum, etc. with very high specific gravities (Pl. 5, fig. b).

To determine more precisely the relative specific gravity of a mineral concentrate, standard minerals are selected for comparison. These should be zircon, pyrite, etc, for the heavies or tips; pyroxene, hornblende, etc, for middlings; and quartz, feldspar, etc, for lights (slimes). These particular minerals separate easily and are frequently present in rocks, soils, till and drainage sediments. Depending on whether a mineral positions itself, for example, in front of or behind zircon or between it and quartz in the train on the pan, the relative specific gravity can be estimated and compared with textbook data. If a sample under study contains neither of the above mentioned

minerals, small amounts of these can be added to permit specific gravities to be estimated for unknown minerals. Other minerals can also be used for specific gravity standards as experience dictates.

### Heavy liquids and mineral separation

High specific gravity liquids, or "heavy liquids", fractionate minerals by density. Liquids in common use include bromoform and methylene iodide with SG 2.80 and 3.20 respectively. A disadvantage in using these materials is their high cost; also if minerals to be separated (e.g., zircon and galena) have specific gravities higher than the liquids in use fractionation is unlikely.

The specific gravity of lighter minerals can be precisely determined especially when a mineral grain becomes suspended in a heavy liquid of known gravity. As a further check, mineral grains of known specific gravity are selected and compared with the unknown. Minerals of known specific gravity should be kept solely for this purpose.

### List or chart of minerals

A list or chart of minerals with corresponding numbers for identification stating their physical properties, and characteristic features is important for quick references. The minerals in this list should be arranged according to their similarities in groups, e.g. quartz, feldspar, pyrite, gypsum, hornblende, mica, etc. Specific gravities and hardness numbers should accompany the minerals on the lists or charts. Such information is of great assistance for quickly comparing unknown and known minerals.

## APPENDIX II

### LABORATORY EQUIPMENT FOR PREPARATION OF GEOLOGICAL MATERIALS

The following lists the various equipment required for the preparation of rock, mineral, soil, till, drainage sediment, and biological materials for chemical and spectrochemical analysis, mineral separation and identification.

#### Mortar, pestle, base and shield

Metallic (mild or hardened steel) (Pl. 4, fig. a).

- Base (a) square, 12 x 12 x 4 in (30 x 30 x 10 cm)
- (b) round, diameter 6 in, height 3 in (15 x 7.5 cm)
- Pestle (c) length 8 in, base 2 in, handle 1 1/4 in (20 x 5 x 3 cm)
- Shield (d) clear plastic tubing, diameter 4 in, height 4 in (10 x 10 cm)

Non-metallic (ceramic, porcelain, agate, etc.) (Pl. 4, figs. b and c).

- Base (a) 12 x 12 x 2 in (30 x 30 x 5 cm)
- Mortar (a) Coors ceramic ball mill end cap
- (b) other designs (eg. agate) as available commercially
- Pestle (a) Coors ceramic ball mill end cap taped to the face of a hammer or to the end of a 2 in (5 cm) diameter steel pestle

- (b) ceramic ball taped to the end of a steel rod, bolt, etc.
- (c) other designs available commercially

- Shield (a) Coors ceramic ball mill center part "sleeve"
- (b) clear plastic tubing, diameter and height according to one's own design.

#### Rock splitter

**Type:** Friction type, hydraulic, either manually or power operated. Pl. 1, figs. 5 and 6 shows a hydraulic, power-operated rock splitter having 1 1/2 in (3.7 cm) knife, pressure rated at 20 tons and compressor (pump) (2 hp motor, 550 volt) rated at 3000 psi, suitable for most types of work.

**Bench supporting rock splitter:** The bench should be of heavy wooden construction, 30 inches (75 cm) high, and have a wooden or steel top. Caging the splitting knife with a clear flexiglass panel or building a tray into the top of the bench below the rock splitter facilitates the collection of stray rock fragments.

**Compressor unit:** Due to the high rotating speed of motor and pressure pump, these should be installed behind a wall to screen the noise from the operator.

**Hood:** For long periods of operation ventilation must be provided. For rock splitters the ventilation system should be located outside of the preparation room.

**Type:** Friction type, manual, to split core 6 to 8 inches (15.0 to 20.0 cm) long.

**Bench:** The bench should be of heavy wooden construction at least 36 inches long, 36 inches wide, and 30 inches high (90 x 90 x 75 cm). An ancillary bench or laboratory truck at least 6 feet long, 3 feet wide, and 4 feet high (180 x 90 x 120 cm) should be provided for laying out drill-core boxes. The excess height permits the examination of the core without undue fatigue from bending.

**Hood:** As for rock splitter.

**Motor and pressure pump:** As for rock splitter.

### All steel crusher

**Type:** All steel crusher models are available with or without motors. Two capacities are generally quoted by the manufacturers: (1) a standard model with 400 lb (190 kg) per hour capacity; 2 1/4" x 3" (5.6 x 7.5 cm) jaw opening and a reduction size of 3/4" to 1/2" (20 to 12.5 mm); fitted with a v-belt and 1 hp electric motor; (2) a heavy duty model with 800 lb (380 kg) per hour capacity; 2 3/8" x 4" (6.0 x 10.0 cm) jaw opening and a reduction size of 3/4" to 1/2" (2.0 to 1.25 cm); fitted with a v-belt and a 2 hp electric motor. The standard model is preferable (Pl. 2, fig. a).

**Bench:** The bench should be of heavy wooden construction 2 feet wide 3 feet long, and 2 feet high (60 x 90 x 60 cm). The crusher should be securely bolted to the wooden top of the bench, and the bench as a whole either weighted at the bottom or the legs should be embedded in a cement block; alternatively the legs can be bolted to the floor.

**Hood:** A good ventilation hood with a 7" (18 cm) stack should be provided. This hood should be so constructed as to permit ready access to the crusher openings.

**Modifications:** For field use the crusher can be fitted with a 3 or 5 hp gasoline engine if electric power is not available. For trace element work the tray receiving the crushed rock should be made of stainless steel and designed with an open end so that material can be transferred directly to a bag. For

exceptionally hard rocks the movable and stationary jaw together with the plates as supplied by the manufacturer may require modification to prevent breakage and undue contamination. The movable jaw plate should be constructed of mild steel with no change in dimensions of the original design. The stationary jaw plate should also be made of mild steel but should be redesigned so that it is 1 1/2 inches (3.75 cm) thick at the top end and 1/2 inch (12.5 mm) thick at the lower end and curved along its length in a manner such that the pieces of rock are repeatedly squeezed at right angles as they pass downward through the jaws to the point of discharge. This results in the production of smaller fragments.

Occasionally a rock sample will jam between the jaws of the crusher and must be pushed downward using a tool such as a steel rod in order to resume crushing operation. Dislodging can also be done by hammering the sample using a modified prospector's hammer (pick) to which a 3 inch (7.5 cm) long rod has been welded to the base.

Replacement of the jaw surface plate can be accomplished with a mild steel plate modified as explained above by drilling and tapping a hole 1/2 inch (1.25 cm) diameter in the plate. The holding bolt should be a full-length threaded rod.

Dust proof motors are desirable on all machines, suction fans, etc. If a dust proof motor is not available for the crusher it is best to install the motor under the bench in such a way that the v-belt can be conveniently adjusted. The same applies to grinding machines using the v-belt drive with the exception that the motor should be installed on the bench outside the hood. Installing the motor outside the dust hood or under the bench greatly simplifies general cleaning of the interior of the hood.

Whenever possible, the power supply (usually 550 volts) for the preparation machines should have plug-in devices. This arrangement is necessary for efficient servicing as machines can then be entirely cut-off from their power source, thus facilitating their repairs or replacement.



It has been found that motors larger than those supplied or recommended by the manufacturer give better service; this reduces stalling of the preparation machines (e.g. all steel crushes, small laboratory sample crushes with ceramic plates, etc.). When a machine stalls, much time can be lost dislodging caught material with the result that some of the sample is lost or becomes contaminated.

### Pulverizer

**Type:** Several models are available. The model selected should be capable of pulverizing at least one pound of 1/4 inch (6.3 mm) quartz fragments in one grinding to 100 mesh (150  $\mu$ m) in one minute. To ensure that the machine will not be subject to stalling the motor size recommended is 3 or 5 horse power. Where electric power is unavailable in the field a belt-driven rotating disk pulverizer fitted to a 3 or 5 hp gas engine is satisfactory. The direct-driven rotating disk- type pulverizer with a built-in sealed electric motor is recommended and has been used successfully by the writer for many years both in the field and headquarters laboratory.

**Bench:** The bench should be of heavy wooden construction and of suitable size to accommodate both the pulverizer and engine.

**Hood:** A good ventilation hood must be provided. This hood should be so constructed as to permit ready access to the entry spout and ground sample tray and also to permit rapid and efficient cleaning (Pl. 9, fig. a).

**Modifications:** A reverse polarity switch to change the direction of rotation of the moving disk should be provided on electric models. This permits the cleaning of both sides of the grooves in the disks when using quartz sand and also distributes the wear on the disks during sample preparation.

For trace-element work the sample tray should be made of stainless steel.

Either steel or ceramic disks may be used on most pulverizers. For ordinary work the steel disks are satisfactory; but for trace-element work ceramic plates are nearly obligatory.

Disks which are most enduring for fine grinding are those which are almost worn to the steel supporting ring. The immediate presence of the steel support offers excellent protection to the fragile edges of the ceramic disks. It should also be noted that the disks should be free of any fractures when preparing fine mesh material since some of the sample will escape along the cracks before the final size is reached even if the disks rub one onto the other. Most of the damaged disk edges or chipped-out parts can be restored by grinding a quantity of coarse corundum. Corundum has a marked wear effect on ceramic disks, hence the distance between the disks must be adjusted continuously to maintain an appropriate spacing. Once a rough restoration has been achieved the disks are removed from the grinder machine for further grinding using finer corundum on a lapidary wheel.

### Compressed air

The source of pressurized air for cleaning the preparation equipment may be a commercial compressor. While 70 psi is suitable for cleaning power machines, 30 psi is more suitable for cleaning sieves, the Frantz isodynamic separator, and other equipment. If a compressor is not available a source of forced air can be obtained from the discharge of a strong vacuum cleaner.

### Automatic cleaning mechanism

If the air supply permits, grinders can be cleaned automatically by the installation of air blasting jets. To do this, two jets are placed under the grinding chamber level with the bottom of the receiving tray which is removed during air blasting. Another jet, coupled to the same solenoid is installed by drilling a hole in the top service door over the grinding disks. A third jet with an independent solenoid is arranged such as to allow the operator to hand blast the feeding spout, and at the same time, the space between disks. The last jet should be spring suspended at the proper distance above the feeding spout. The jets under the machine should be constructed of 1/2 inch (12.5 mm) copper pipe, the top of the door should be of 1/2 inch (12.5 mm) flexible plastic, and the hand operated jet should be made of

1 1/4 inch (3.15 cm) rigid plastic pipe. Type of solenoids used to operate the jets are 1 inch (2.5 cm) diameter, normally closed, 24 volt (Pl. 7, figs. c,d).

A third line having an independent solenoid and using universal vacuum cleaner hose is conveniently hooked to the hood and is used to hand clean the hood interior and base.

The total automatic cleaning operation should take only a few seconds since it is not necessary to stop or open the grinder. For efficient cleaning the solenoids are opened alternately 5 or 6 times.

In practice, it has been found that the automatic cleaning operation is approximately 95% effective. For a series of larger samples of similar type, the method is considered adequate.

When quartz is used to clean the plates between each preparation air blasting is done only after the quartz cleaning operation. When the last part of the sample of cleaning quartz is slow to pulverize completely, the two service doors can be set slightly ajar to let the few chips discharge while the machine is running.

For convenience in cleaning the crusher parts, the air hose should be hung over the crusher machine by a light, long extension spring or clipped to the side of the hood. As for the grinder, electric solenoid valves (1 inch) (2.5 cm) are used to provide the instant air blast and are operated by a simple 24 volt switch mounted on the end of the cleaning hose. Common vacuum cleaner hoses are used as air lines from the solenoid valves. For ease in air blasting the crusher jaws the air should be directed via a curved coupling (1 inch (2.5 cm) diameter) fitted to the hose end. For safe operation of the switch, the line voltage should be reduced by a step down transformer. If a vacuum cleaner is used as the air blast, the hose and switch can have a similar arrangement with the exception that a relay should by-pass the high amperage switch of the vacuum cleaner.

### Sample Mixing Table

Mixing tables should be of solid wooden construction, with a treated wood, stainless steel or arborite top; the first is preferable.

The dimensions may vary to suit the individual, but should not be less than 30 inches wide by 30 inches long and 36 inches (75 x 75 x 90 cm) high.

It is usual practice in geochemical sample preparation laboratories to cover tops of tables, carts, counters, etc. with kraft wrapping paper. Whenever fresh surfaces are required, kraft papers are simply replaced.

### Coors ball mill shaking machines

The machine used to shake the Coors ball mills is the common type of paint shaker. Specially designed wooden racks for holding 4 or 6 mills are fitted in the jaws of the shakers (Pl. 2, fig. e).

Using racks to hold 4 Coors mills rather than the usual 6 will reduce much leaking possibilities during the milling period because the parts will rest more evenly one onto the other: but, will reduce on the other hand daily output.

The security pin which prevents the rack holding the Coors mills from coming apart if would accidentally slip-off the shaker jaws is also a tool to parallel the Coors mills. As the security pin is placed toward the other end of the rack it is also used the counterbalance the shaker holding jaws pressures, ensuring mills pats are resting one onto the other properly and evenly, sealing sample inside the mills.

For quick and easy adjustment of the security pin it is best to design a large wing nut as shown in Pl. 9, fig. e.

### Construction of Coors ball mill holding racks

Steps to construct racks holding 6 mills

- 1) Cut 2 pieces of plywood 7/8" thick, size 8 1/2" x 11" (2.24 x 22.0 x 28.0 cm) These constitute the top (1a) and bottom (1b) boards.
- 2) Cut 1 piece of plywood 1/4" thick, size 8 1/2" x 11" (0.63 x 22.0 x 28.0 cm).
- 3) Cut 6 holes evenly spaced and large enough to take the ceramic cartridges, through the 1/4" (0.63 cm) thick piece of plywood.

- 4) Glue and secure with screws the piece of plywood prepared in (3) to piece prepared in (1).
- 5) Cut a sponge pad to fit the plywood board in (1). The pad stabilizes the mills when placed between mills and top board.
- 6) Install a 3/8" (0.95 cm) bolt with wing nut to hold the frame together.
- 7) When in use seal the ceramic cartridges with vinyl gaskets and adhesive tape to prevent escape of finely ground material. Note: Slightly warped, damaged, etc., boards will cause Coors mill parts to be unevenly positioned thus leading to leakage of sample material.

#### **Ball milling laboratory requirements**

The counter area used to service mills should be close to a wash basin and high rated current outlets. Generally one side of the basin is used to drain excess water off the mills once wash, while the other side is used to reset mills in the shaking racks. A ventilated heater is commonly used to dry the cylinder parts remaining on the draining side of the basin while another is used to dry the end caps fitted in the shaking boards. If a flat top laboratory truck is used to service the Coors mills, width and length should be sufficient to permit opening of all racks simultaneously.

#### **Spare parts**

The Coors ball mill end caps are of thick material and are not subject to breakage during milling. The same cannot be said, however, about the central cylinder which is much more delicate. It is recommended that about half the number of the sleeves in actual use be kept in reserve to cover breakages, etc. In addition a small supply of support springs and transmission parts should be kept in reserve since these tend to wear out with excessive usage.

#### **Coors ball mill gasket**

The Coors mill gaskets are made from 30 mil thick vinyl material. The knife used to cut the gaskets is machined in two parts which are then fitted together so that an entire gasket can be cut in one operation. An

aluminum block should be used to counter the cutting pressure of the knife. A vertical press is an ideal machine for rapid fabrication, but a mechanical vise can also be used. The approximate required pressure for a complete cut is 5 tons. Spongy material is used in the cutter groove to spring return the gasket when cutting is finished. Air can also be used (Pl. 8, figs. d and e).

#### **Drying cabinet (field)**

A drying cabinet is essential for drainage sediment, soil and till samples. The cabinet should be of sufficient size to dry at least 200 samples simultaneously. The following type of cabinet is suggested, especially for field use.

**Size:** 4 feet (1.2 m) square by 7 feet (2.1 m) high.

**Construction:** Of 1/4 inch (6.3 mm) plywood on a 2" x 4" (5.0 x 10.0 cm) wooden frame. A lift-up type door with counter balance should be fitted on the front, and if possible this door should have a glass port hole.

**Insulation:** The walls, roof, and floor should be lined with aluminum foil stapled to the plywood. This serves as a heat reflector and reduces the fire hazard.

**Heat source:** A propane gas stove with at least two burners is advisable. The stove should have a precise adjustable control. A metal sheet should be installed 12 inches (30 cm) above the flame to prevent samples from accidentally falling into the flame.

**Trays:** Four or more trays to hold the wet samples are required. These trays should have a latticed bottom to permit circulation of the air around the samples and should fit into the cabinet one above the other. The lower tray should be at least 2 feet (60.0 cm) above the gas burners, and the other trays should be spaced at intervals of a foot (30 cm) above one another.

**Ventilation:** A 6-inch (15 cm) hole in the top of the cabinet is sufficient for ventilation purposes. The door on the front of the cabinet should be left open an inch (2.5 cm) or so during drying.

**Temperature control:** An inexpensive, insert-type thermometer should be fitted in the side of the cabinet in a convenient place. The temperature inside the cabinet should be maintained at about 60°C during the drying operation. At this temperature it requires approximately 24 to 48 hours to thoroughly dry wet- stream-sediment samples in kraft paper sample bags.

### Haultain Superpanner

The following lists modifications that will increase Superpanner performance.

- 1) A direct current motor (1/12 horse power, 1724 rpm) to provide smoother operation.
- 2) The v-pulleys that transfer power from front to side can be replaced with those of larger diameter. However, care must be taken to maintain speed and stroke as set by the manufacturer from 0 to 400 per minute.
- 3) The wires holding the pan can be replaced with stiffer ones for longer life. However, this modification requires that the ends of the wires be inserted loosely in the holding flange, and that springs be used to hold the pan in place.

## BASIC LABORATORY EQUIPMENT

### General

A preparation laboratory must also be well equipped with electric power (550, 3 phase, 230, 115 volts), gas, water and air outlets, sufficient counters and tables together with sample storage trays, plastic vial holding boards, drying rooms and ventilation hoods to expedite handling and processing of a large number of samples. If any of these basic facilities are deficient or lacking, considerable time can be wasted and preparation becomes inefficient and boring. In practice, the absence of well organized preparation methods, or a well-designed laboratory, can lead to prepared samples of inferior quality.

Concerning health and other hazards it should be mentioned that the operation of several shaker machines and air blasting of

crushers creates a high noise level which can be annoying and distracting for other personnel working in the same room. Separate rooms should be made available for crushers and grinders and shakers when possible and ear protectors should be issued to all personnel. A similar situation exists with dust and odours. Dust masks must be worn by all persons involved in the sample preparation laboratory. A steady and adequate fresh air source is a prerequisite.

### Ventilation

When samples are prepared either by hand or by machine, or when equipment is cleaned, a considerable amount of dust and odours are generated. Unless adequate ventilation is available those immediately involved can be adversely affected healthwise. It is, therefore, imperative to carefully study the operations of all preparation stages and to design suitable ventilation hoods, dust collectors, and waste disposal systems. For example, cleaning the crushers and grinders by air blasting raises considerable dust which can only be effectively controlled with suitable hoods. For the grinding phase of sample preparation two hoods are necessary, one for the grinder and another for sample handling. The two hoods should be coupled in such a way that the grinder receiving tray can be reached by an appropriate opening inside the sample handling hood. Since the front window is used frequently it should slide freely. The best design is one with a full-sized plexiglass plate which runs smoothly in hardwood frame grooves. Use of a back counter weight and large pulleys will provide finger-tip operation. An opening at the rear of the hood, level with the base, permits efficient air blast cleaning and removal of debris. Openings can be kept closed by simple springs or controlled by a lever at the front door. Plate 9, fig. a is a sketch showing hoods and bases for crusher, grinder and sample handling. It should be kept in mind that air discharged by hoods, fans, etc. must be replaced continuously. To obtain good positive air circulation in the sample preparation room, the source must be well designed. When pressurized units are designed, the personnel operating the sample

preparation room should be consulted about the temperature and quantity of air required.

### Dust collector

Dust collectors come in different sizes and models. Models which have a motor rating of 5 to 10 hp, 550 volts, and that automatically clean the filter cloth are recommended. Preferred dust collectors are similar to those just described but equipped with a 10 hp direct current motor whose speed can be electronically controlled or adjusted according to need.

### Vial holding boards (vial racks)

The vial holding boards (racks) are made with 3/4 inch (20 mm) plywood and are 22 inches long and 7 1/2 inches wide (56 x 19 cm), with 4 rows of 10 holes (depressions) per row. The actual width and length can be varied according to the particular storage requirements, laboratory facilities, etc.

When large numbers of these boards are constructed it is best to drill a standard one and use it as part of a jig as follows: Another board is made, but instead of holes impressions are fixed to it such that they match perfectly the holes of the standard board. In other words a female and a male board are made, one having impressions and the other depressions which enter one into the other perfectly. The board to be drilled is attached on the flat side of the impression board. During drilling of a new board the impression board is moved depression to depression in the standard board thus ensuring the correct drilling distance for each new hole (Pl. 9, fig. d).

### Push cart

Push carts should be of all steel construction with a lower shelf. The dimensions are; width: 20", length: 36", height: 30" (50 x 90 x 75 cm). A piece of plywood, 24" x 48" x 3/4" (60 x 120 x 2.0 cm) can be fitted to the top to better handle samples and sample trays.

### Sieve repairs

The stainless steel sieves should be repaired with epoxy. A gun dispensing warm epoxy is ideal for repairing torn cloth sieves.

### Rock sample trays

Rock sample trays are usually made entirely of plywood, 1/4 inch thick for the bottom and 1/2 inch thick for the sides (6.3 x 12.5 mm). For stronger trays sides of solid wood (pine) are recommended. The overall dimensions of rock sample trays should be: width 23 inches, length 24 inches, and height 2 1/2 inches (58 x 60 x 6.3 cm).

### Ancillary equipment

In addition to the above basic equipment a great variety of ancillary machines and articles are required for the preparation of geological materials for analysis. Some of the more important of these are listed below:

#### Air compressor

- |                  |   |  |
|------------------|---|--|
| Balance          | - | 800 gram capacity                          |
| Beakers          | - | assorted sizes                             |
| Brushes          | - | assorted sizes – bristle and steel         |
| Handbook         | - | on flotation                               |
| Coors ball mills |   |  |
| Crusher          | - | all steel                                  |
|                  | - | jaws and cheek covered with ceramic plates |
| Dust collector   | - | permanent or portable on casters           |
| Dust masks       |   |  |
| Ear protectors   |   |  |



Engraver		Hand mortars and pestles - agate, ceramic and steel types
Grinder	- steel and ceramic disk-plates	Paint shaker machines
Flotation machine	- 8000 ml, 2 kg cell capacity	Plastic vials
Funnels	- assorted sizes	Plastic vial holding boards
Heat lamps		Prospector's gold pans - assorted sizes
Laboratory catalogues		Sieves - 5, 10, 20, 40, 60, 80, 100, 150, 200 mesh (4.0, 2.0 mm, 850, 425, 250, 180, 106, 150, 75 mm). For trace element work stainless steel sieves should be used. The mesh of these sieves should be secured by epoxy and not by soft lead solder.
Heavy liquids	- methylene bromoform	Sieve shaker - mechanical (gyratory)
Magnets	- permanent hand type	Superpanner
Magnetic separator	- isodynamic type (Frantz)	Vacuum cleaner
Magnetized needle and handle		Ultrasonic cleaner
Magnifying glass		
Microscope	- binocular, up to 100X magnification	

### **APPENDIX III**

#### **USE OF CERAMIC DISKS ON POWER GRINDERS**

##### **Introduction**

Occasionally large amounts of geological materials must be ground very fine for analytical controls, standards, and mineral concentrates, without contamination by steel particles from preparatory machines. When steel disks are used to pulverize the samples, the product is commonly contaminated with Fe, Mo and V contained; in steel particles, only rarely can these contaminating particles be removed without losing some of the sample (e.g., magnetite) when using hand magnets. To avoid this type of contamination ceramic disks must be used for powdering. Use of ceramic disks also diminishes the amount of superfine material that is commonly lost during pulverization of samples.

##### **Surface of ceramic disks**

When the grinding machine is fitted with ceramic disks for fine powdering, precise adjustment of the disk gap is difficult because of the risk of breakage. This type of damage is usually caused by insufficient sample coating on the disk surfaces, causing the disks to run "dry". Other effects of non-parallel or poorly adjusted disk gaps may be due to fusion of the sample material on the disks, a feature that generates uneven surfaces. If both disks are solidly bolted to the frame, fracture is likely to occur.

It has been found that mounting one of the disks on rubber stoppers to make it flexible minimizes damage during grinding operations, thereby prolonging disk life.

##### **Installation of disks**

The following describes how plate flexibility can be accomplished by simply installing three rubber test tube stoppers properly spaced, and in certain cases one stopper in the centre, between the holding flange and disk. Using this modification compensation can be made for fusion deposits, and the disk faces can be maintained in a parallel position (Pl. 7, fig. b).

Rotary ceramic disks as delivered contain small counter-sunk grooves for fitting tightly to the shaft flange, thus allowing only a part of it to be used for the required disk play. Rubber stoppers (1 inch diameter (2.5 cm)) are cut such that sufficient pressure can be maintained for all types of grinding by the normal adjustment controls. For a more permanent installation, the flange and stoppers should be perforated and the rubber installed with safety lock bolts and nuts. To maintain the disk in its groove with a little flexibility, longer bolts than usual should be used, and these should be slowly tightened until they touch the bottom of the threaded hole.

The general condition of the disks should be checked by squeezing the disk and flange together at different places with the finger and palm of the hand. For maximum effect the disk should constantly be movable in the flange groove evenly when adjusted for extreme fine delivery.

##### **Pulverization using a rubber mounted rotary disk on a Braun grinder**

To operate with the above modification in place, it is necessary to apply sufficient

pressure on the disks using the motor shaft while the front service door is kept slightly open. This is necessary for final adjustments. Due to the flexibility of the rotary disk, disk faces will be maintained parallel. The same operating precautions must be observed, i.e., disks must never become dry when operating. To ensure sufficient sample feed, the spout should be filled with material before the power is turned on. If the motor refuses to run, the service door is adjusted and readjusted immediately afterward. With experience, it will be found that the necessary adjustment will depend on motor performance and quality of discharged material. The door may be completely closed if adjustment pressure permits, but it is advisable that a small gap be retained for possible future adjustments. Rubber mounting the rotary disk will not prevent all breakage, especially if proper care and attention are not paid to the grinder during operation.

An experienced operator gains a feel for the proper operation of pulverizers and becomes sound sensitive to disk abuse and its effects on the quality of discharged material. He should make the proper adjustment whenever necessary or turn the power off before damage can occur to the disks. Much damage can be prevented by ensuring a constant feed of sample material in the entrance, for example with the aid of a long screw driver. If material has been previously ground fine, it is best to sieve the product and remove the finer fraction as larger sized materials feed faster.

The modification noted above can only be used for fine grinding. All other routine preparation of the solids should be made as explained previously, i.e., the disk must be solidly fitted to the flange. The original bolts or washers of proper thickness should be used. Usually the bolts inflate the rubber stopper and the disk rests solidly on the designed flange.

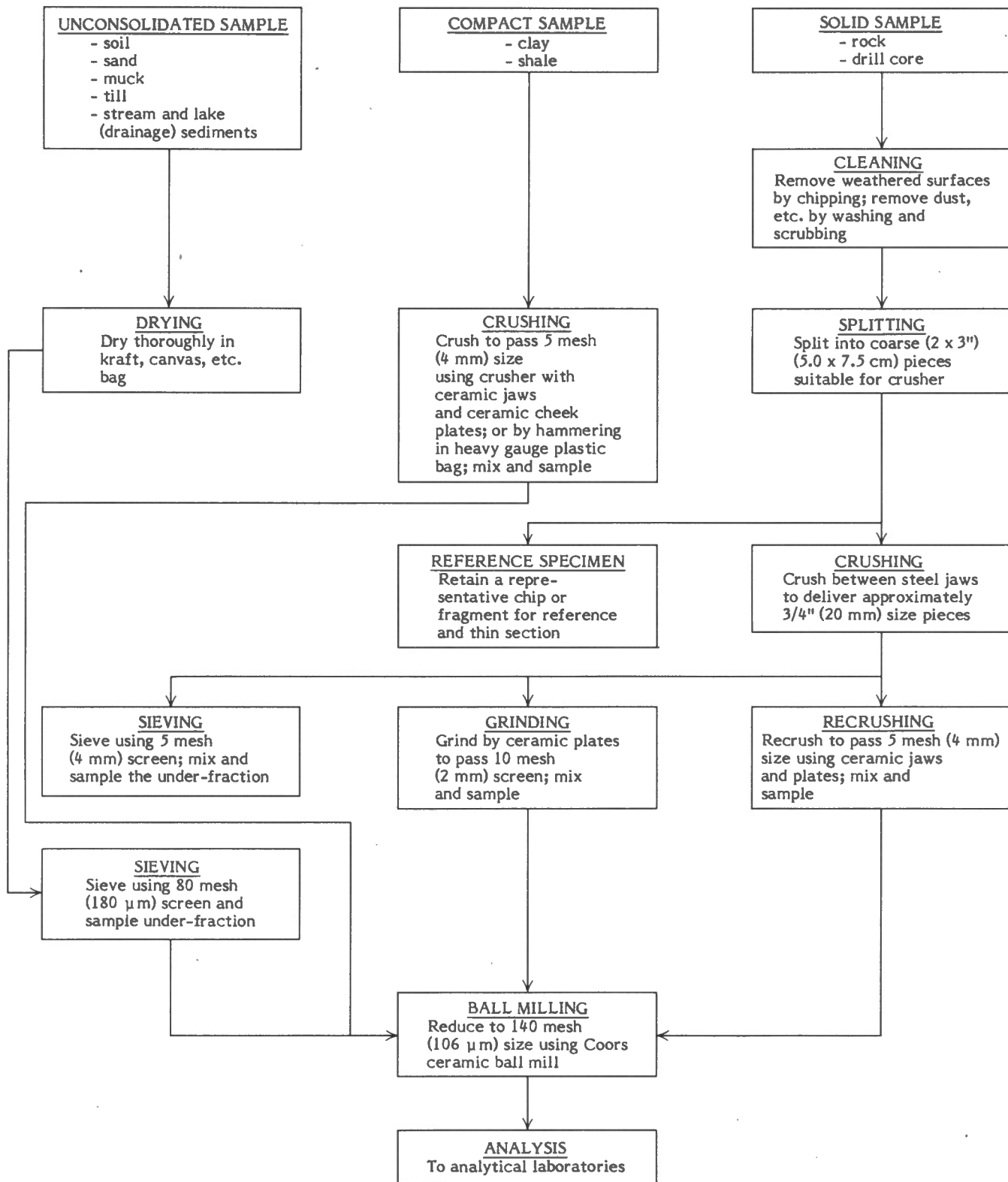


Figure 1. Flow sheet showing steps for the preparation of unconsolidated, compact and solid samples for analysis with minimum contamination.

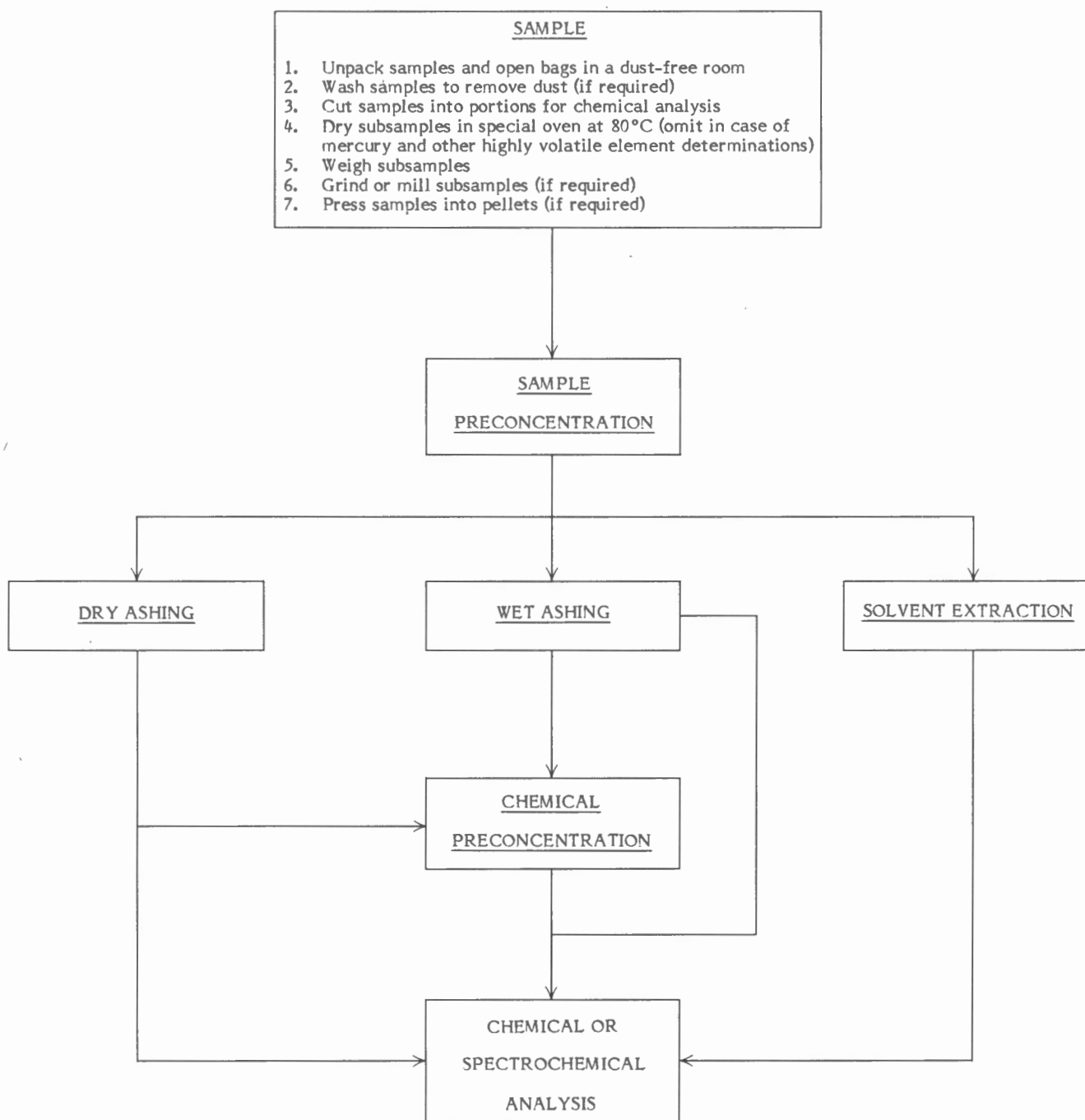


Figure 2. Flow sheet showing the steps in sample preparation of biological samples and the main stages in the chemical analysis of biological materials.



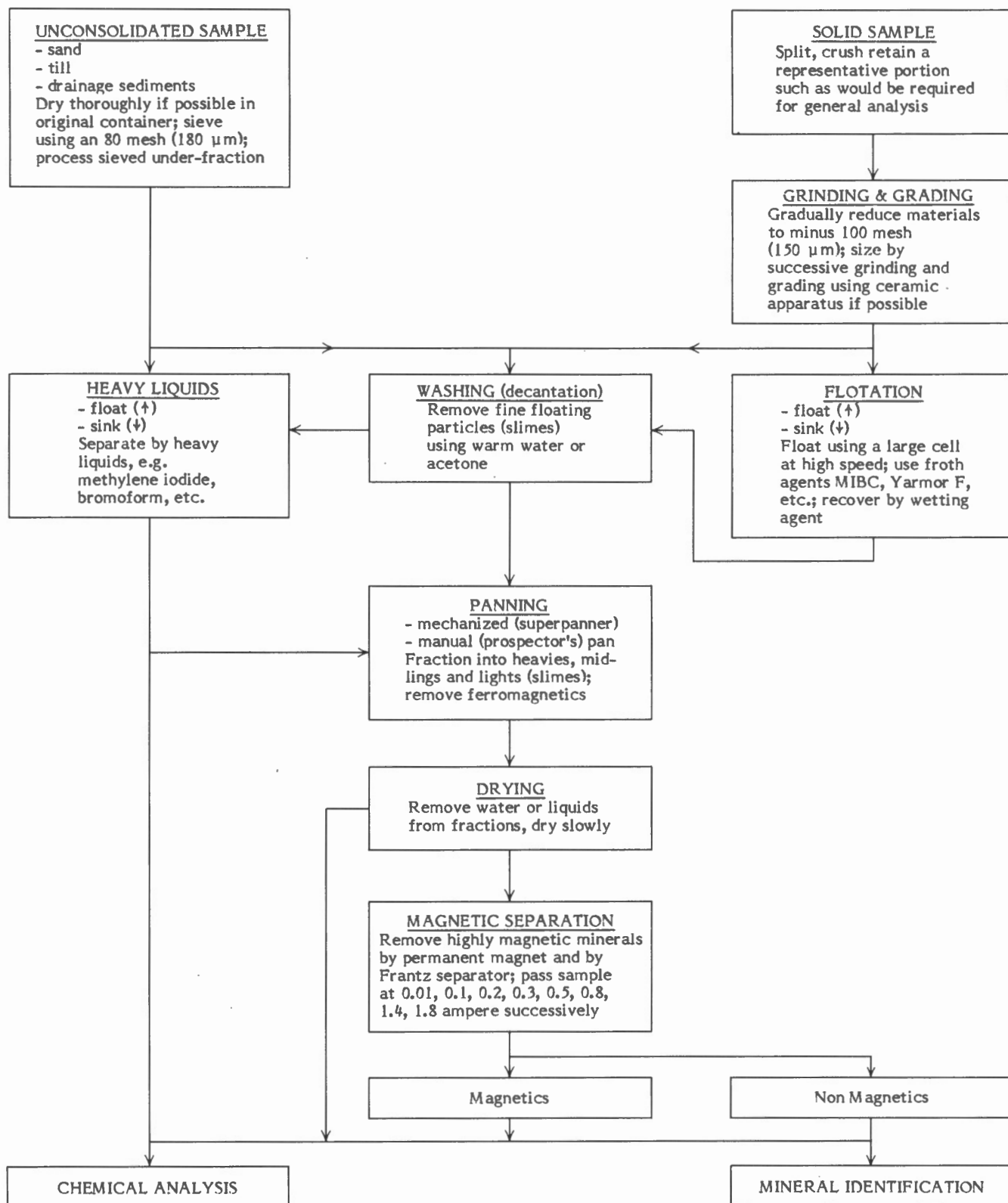


Figure 3. Flow sheet for the preparation of mineral separates and concentrates for chemical analysis and mineral identification.

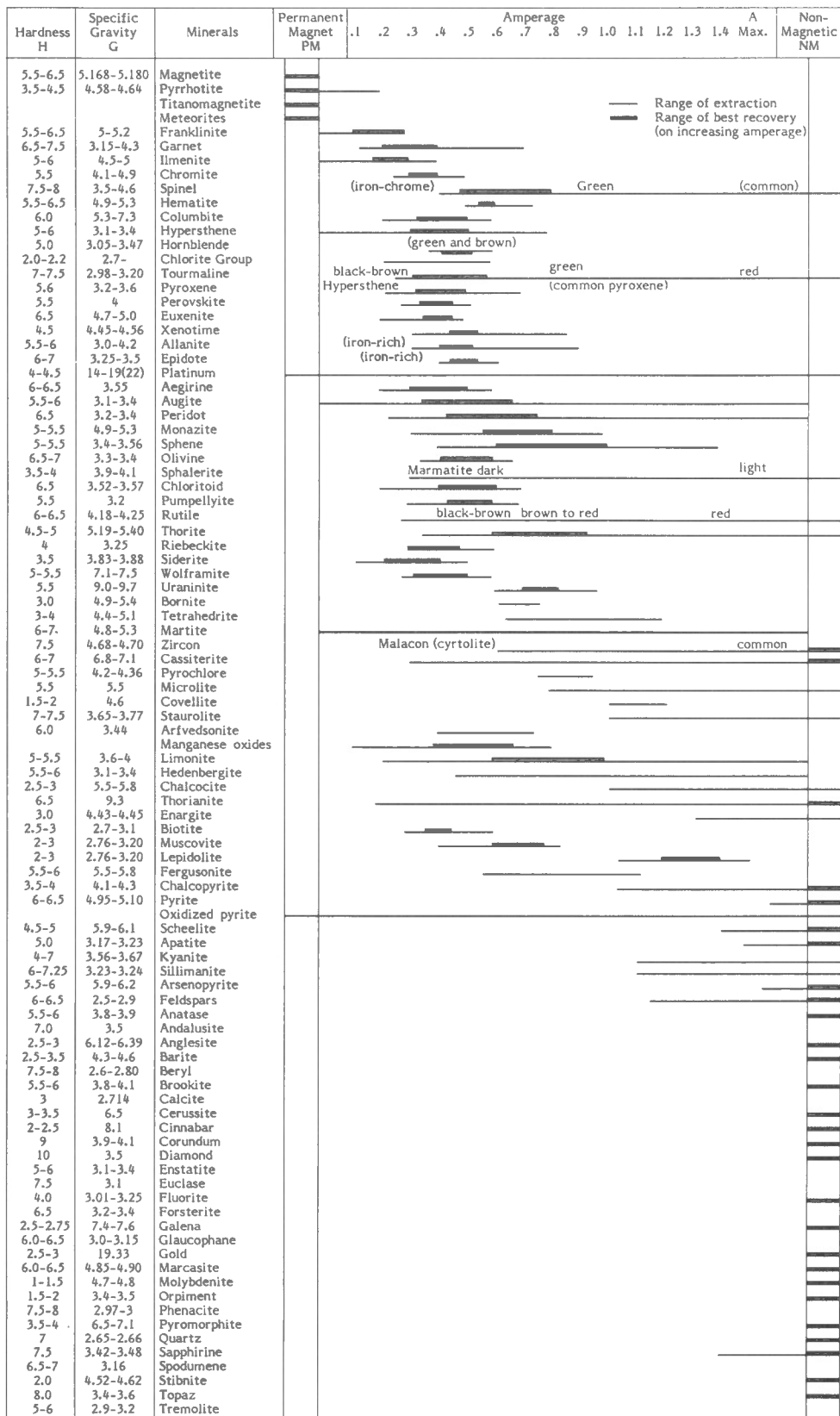


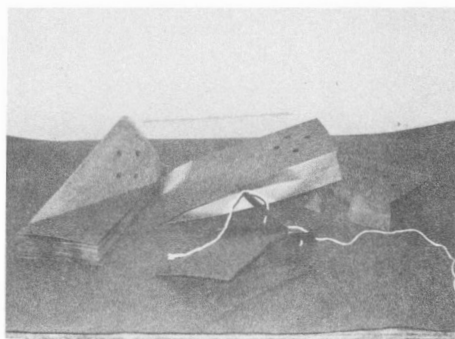
Figure 4. Hardness, specific gravity and magnetic susceptibilities of some heavy minerals in Frantz isodynamic magnetic separator. Mineral hardness and specific gravity data after Dana (1964) and Buttgenbach (1929). Magnetic behaviour after Rosenblum (1958) and Parfenoff (1967) modified by author.

## **PLATE 1**

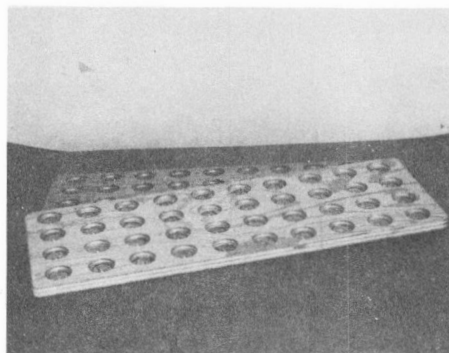
### **Figures**

- 1. Water proof kraft paper bags, size 2 x 5 x 10 inches (5 x 12.5 x 25 cm) used for collecting wet materials such as drainage sediments, soils, etc.**
- 2. Vial holding board.**
- 3. Laboratory truck loaded with sample trays.**
- 4. Large quantity of sample vials taped together in racks for identification marking by indelible pen.**
- 5. Power-driven rock splitting machine using 3000 psi hydraulic compressor and 1 1/2 inch (37.5 mm) diameter, 20 ton pressure points.**
- 6. Manually operated rock splitting machine using 5 ton hydraulic jack.**

# PLATE 1



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b. Vial holding board.



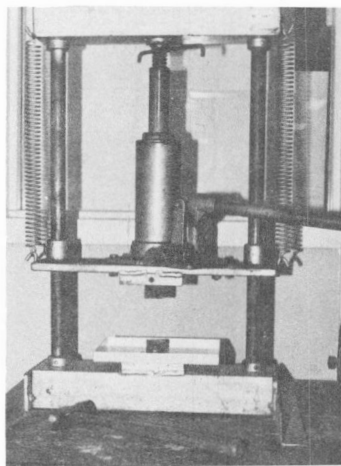
c. Laboratory truck loaded with sample trays.



d. Large quantity of sample vials taped together in racks for identification marking by indelible pen.



e. Power-driven rock splitting machine using 3000 psi hydraulic compressor and 1 1/2 inch (37.5 mm) diameter, 20 ton pressure points.

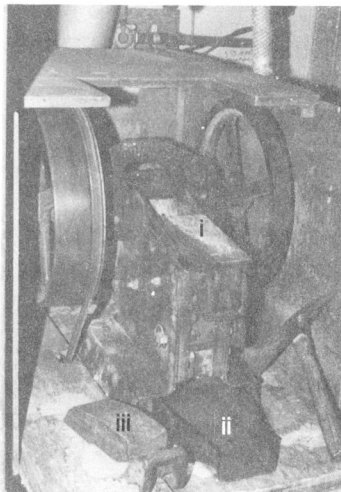


f. Manually operated rock splitting machine using 5 ton hydraulic jack.

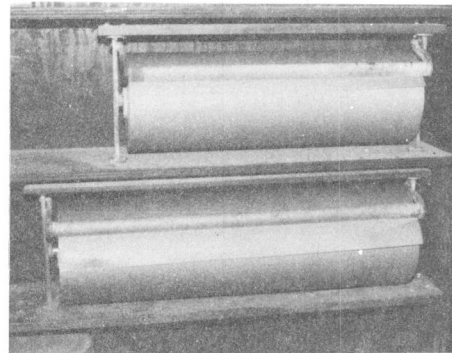
## **PLATE 2**

### **Figures**

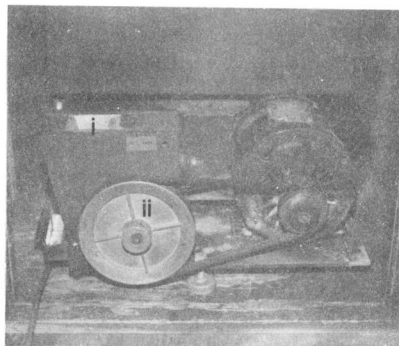
1. All steel laboratory rock crusher in a ventilation hood showing: a) crusher stationary jaw placed on a bracket for cleaning, b) open end sample receiving tray, and c) modified stationary jaw.
2. Kraft wrapping paper on a roll to cover counters, table, etc, and to handle wet or dry samples of rocks, drainage sediments, soils, etc.
3. Small sample crusher: a) ceramic plates glued to cheeks and jaws, b) large motor and pulleys.
4. Opened rack holding 6 Coors ball mills.
5. Ceramic ball mill: a) double arm ball mill shaker, b) Coors ball mills in rack, c) wooden box containing ceramic balls mixed with quartz chips placed in shaker jaws and, d) time clock.
6. Samples in plastic vials placed near the Coors ball mills prior to loading.



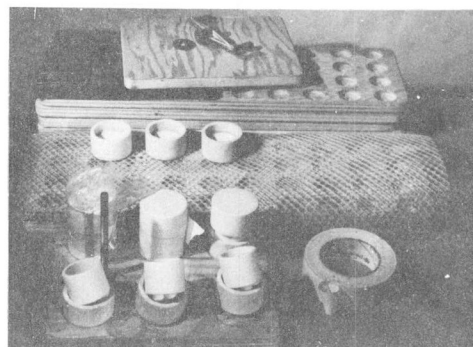
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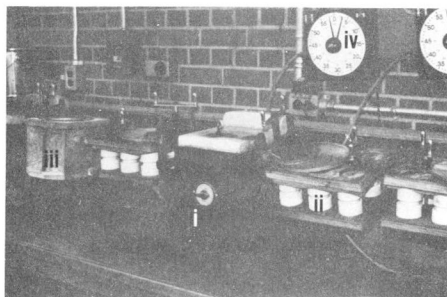
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f. Samples in plastic vials placed near the Coors ball mills prior to loading.



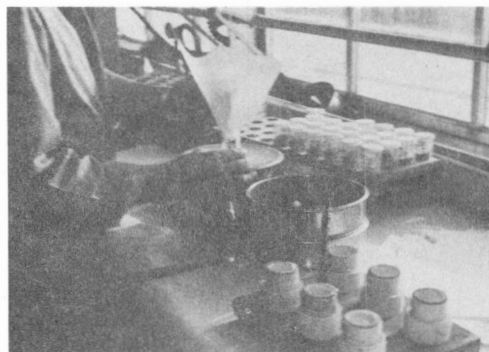
### **PLATE 3**

#### **Figures**

1. Removing the balls from a sample powdered by Coors ball mill.
2. Funnelling the powder into a plastic vial using paper sheet.
3. Washing Coors ball mill end caps with a stiff bristle circular brush.
4. Washing Coors ball mill center parts. Note the use of a light gauge plastic bag as a glove.
5. Drying ceramic balls in a 5 mesh (4 mm) screen by forced air from a commercial vacuum cleaner.
6. Drying ceramic balls in a 5 mesh (4 mm) screen by forced air from a forced air electric heater.



a. Removing the balls from a sample powdered by Coors ball mill.



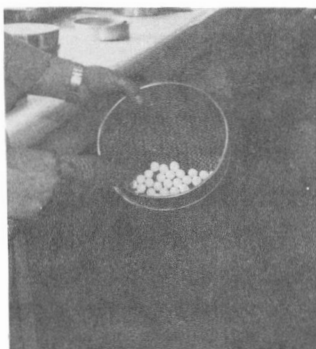
b. Funnelling the powder into a plastic vial using paper sheet.



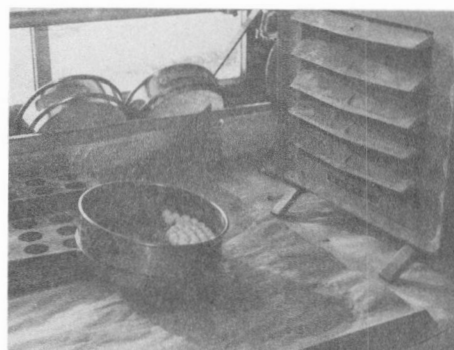
c. Washing Coors ball mill end caps with a stiff bristle circular brush.



d. Washing Coors ball mill center parts. Note the use of a light gauge plastic bag as a glove.



e. Drying ceramic balls in a 5 mesh (4 mm) screen by forced air from a commercial vacuum cleaner.

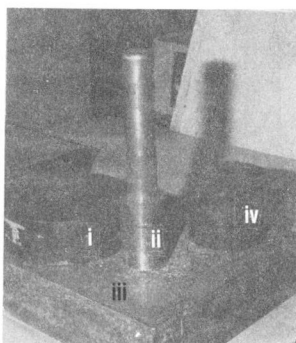


f. Drying ceramic balls in a 5 mesh (4 mm) screen by forced air from a forced air electric heater.

## **PLATE 4**

### **Figures**

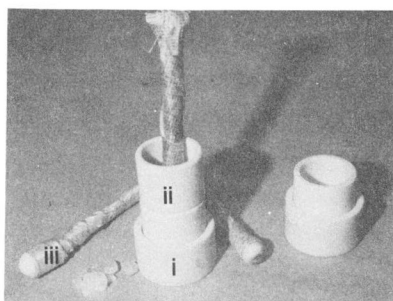
1. Steel sample preparation tools: a) mortar, b) pestle, c) base, d) plastic shield.
2. Ceramic sample preparation tools. Coors ball mill end caps taped to: a) mallet, b) pestle, used for minimum contamination.
3. Coors ball mill parts used as: a) mortar, b) shield, and c) pestle comprising ceramic ball taped to the end of a steel rod.
4. Hammering cemented stream sediment, lake sediment, etc., samples by a modified mallet in kraft paper bag placed inside a plastic bag.
5. Set-up for sieving unconsolidated materials in a ventilation hood.
6. Sieving with ceramic balls mixed with sample to disintegrate lumps and to keep screen free from clogging.



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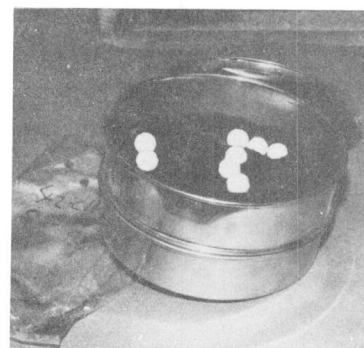
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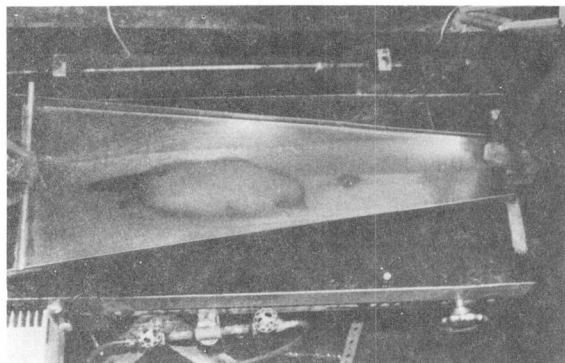
## PLATE 5

### Figures

1. Sample stirred in water to suspend the finest and lightest grains, and to leave behind on decantation the heaviest and largest grains. The washing is usually carried-out prior to mineral concentration by superpanner, heavy liquids or examination of minerals by microscope, etc.
2. Separation of mineral grains in a superpanner according to specific gravity. The grains at the extreme left are the heaviest while those at the extreme right are the lightest.
3. Removal of ferromagnetic grains from the Superpanner.
4. Superpanner modified a) by electronic speed control, b) by a soap column, c) by a dripping pan, and d) by larger pulleys.
5. Frantz isodynamic magnetic separator fitted with: a) a variable step-up transformer, b) an aluminum chute, c) nylon supports, and d) a time clock.
6. Mouth pipette used to remove minerals from the Superpanner concentrate.



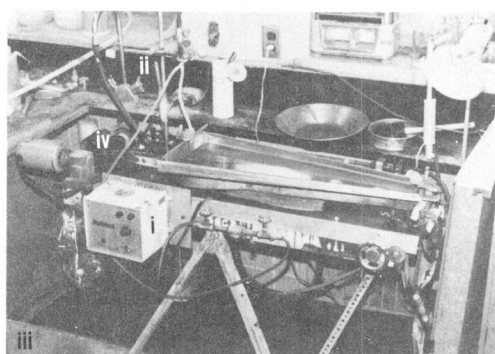
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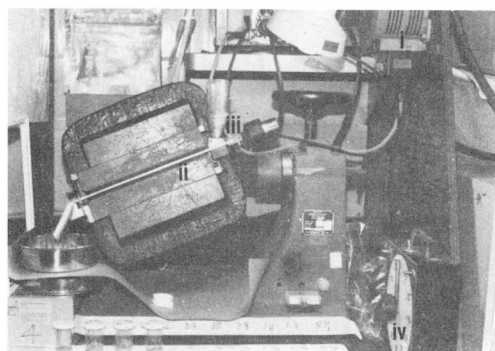
- b. Separation of mineral grains in a superpanner according to specific gravity. The grains at the extreme left are the heaviest while those at the extreme right are the lightest.



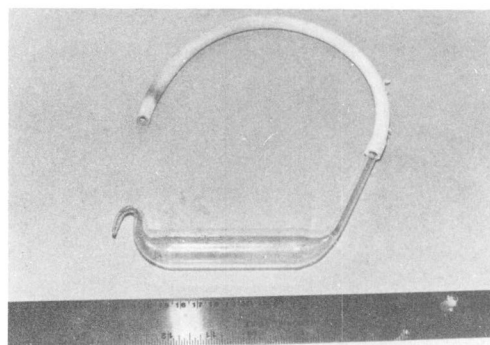
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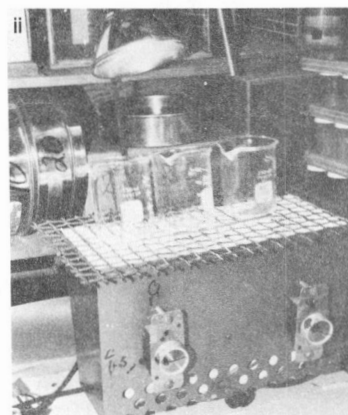
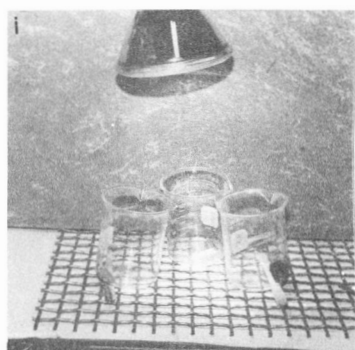
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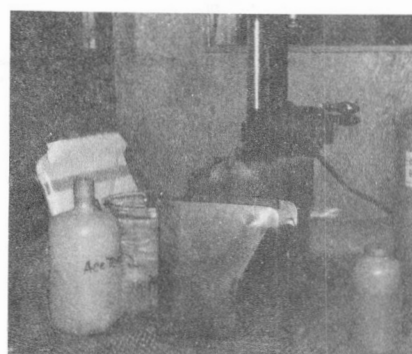
## PLATE 6

### Figures

1. Drying concentrates in beakers by heat lamps: a) over, and b) over and under the sample. The amount of heat is controlled electronically by a lamp voltage regulator.
2. Simple flotation machine in which the source of air is external and introduced below the water by a tube.
3. Laboratory flotation machine, 8000 ml size tank sample (2kg) capacity (Denver Model D-2).
4. Heavy liquid separation apparatus: a) Filtering of the liquids by gravity, b) sketch of hose attached to funnel stem for separation of samples composed mainly of large grains, and c) by vacuum assisted apparatus.

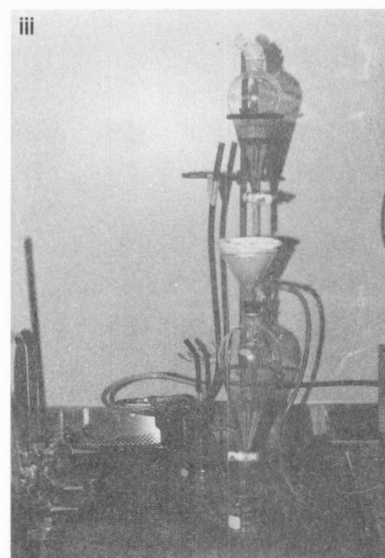
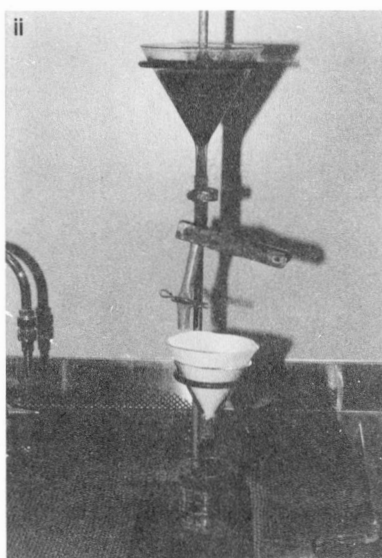
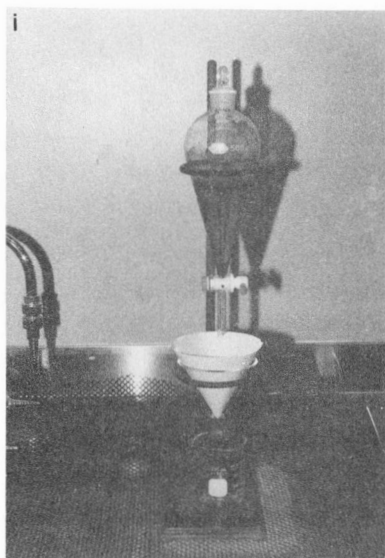


a. Drying concentrates in beakers by heat lamps: i) over, and ii) over and under the sample. The amount of heat is controlled electronically by a lamp voltage regulator.



c. Laboratory flotation machine, 8000 ml size tank sample (2kg) capacity (Denver Model D-2).

b. Simple flotation machine in which the source of air is external and introduced below the water by a tube.

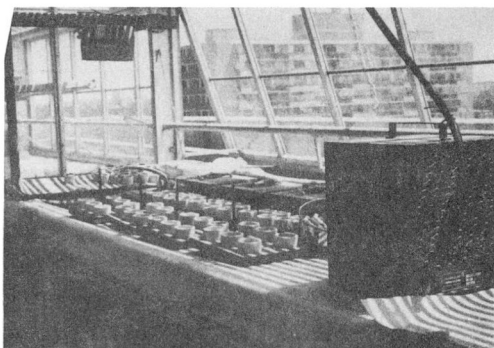


d. Heavy liquid separation apparatus: i) Filtering of the liquids by gravity, ii) hose attached to funnel stem for separation of samples composed mainly of large grains, and iii) by vacuum assisted apparatus.

## PLATE 7

### Figures

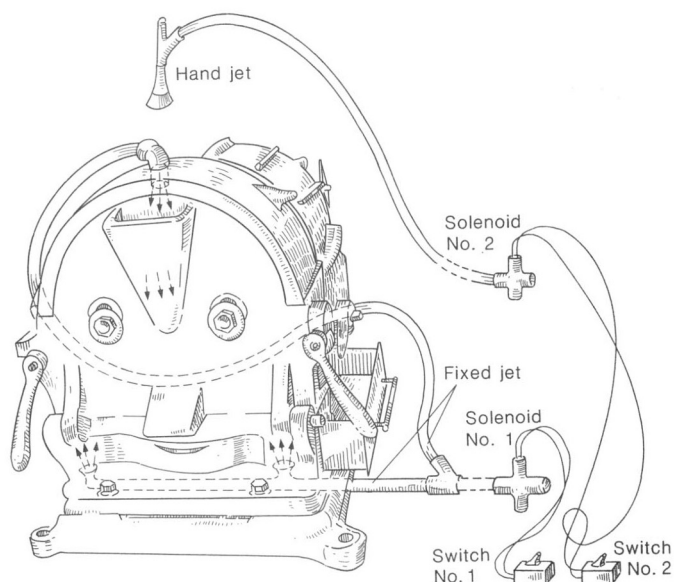
1. Opened Coors ball mill assembly being dried by forced air electric heater.
2. Braun pulverizer rotary disk-plate placed on rubber mounts to obtain a certain degree of flexibility for very fine grinding of samples.
3. Sketch of air jets controlled by solenoid valves to automatically clean the Braun grinder after each sample preparation.
4. Electrically operated 1" (2.5 cm) diameter solenoids, used to clean the sample preparation machines automatically.
5. Dressing a ceramic disk-plate on a lapidary wheel.
6. A 24 inch (60 cm) gyratory vibrating machine used to classify grains according to size. The screen frame can be dismantled for the employment of nylon, plastic, stainless steel, etc., screens. Ceramic balls together with a brush can be used to break up lumps in the sample and thus keep the screen free from clogging.



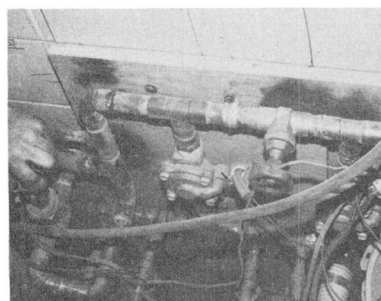
a. Opened Coors ball mill assembly being dried by forced air electric heater.



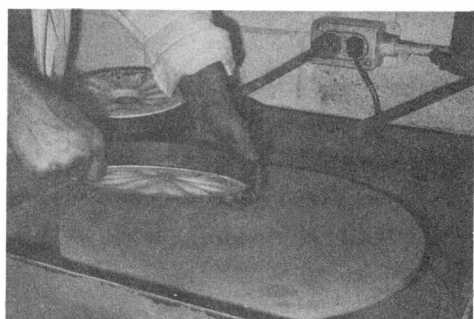
b. Braun pulverizer rotary disk-plate placed on rubber mounts to obtain a certain degree of flexibility for very fine grinding of samples.



c. Sketch of air jets controlled by solenoid valves to automatically clean the Braun grinder after each sample preparation.



d. Electrically operated 1" (2.5 cm) diameter solenoids, used to clean the sample preparation machines automatically.



e. Dressing a ceramic disk-plate on a lapidary wheel.

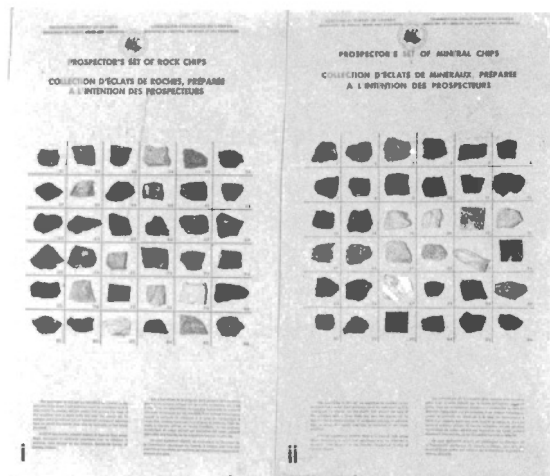


f. A 24 inch (60 cm) gyratory vibrating machine used to classify grains according to size. The screen frame can be dismantled for the employment of nylon, plastic, stainless steel, etc., screens. Ceramic balls together with a brush can be used to break up lumps in the sample and thus keep the screen free from clogging.

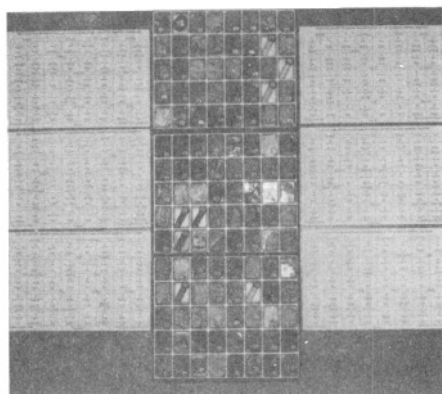
## PLATE 8

### Figures

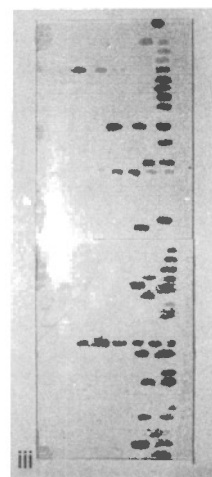
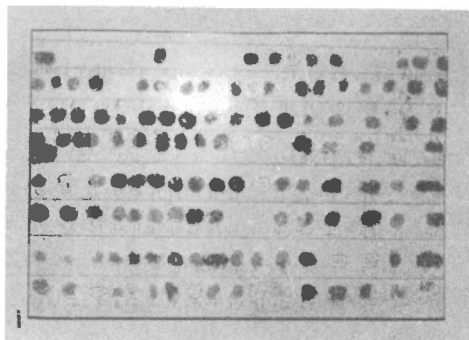
1. Study set of (a) rock and (b) mineral samples prepared by the Geological Survey of Canada.
2. Study set of larger mineral and rock samples prepared by the Geological Survey of Canada.
3. Mineral chips crushed to  $-80 + 100$  mesh ( $-180 + 150$   $\mu$ m): a) glued to plexiglass, b) studied under microscope, and c) glued to plexiglass in order of magnetic susceptibility separation by Frantz isodynamic separator.
4. Coors ball mill gasket cutting tool: a) aluminum base, b) gasket return sponge, c) knife, and d) 30 mil vinyl material.
5. Cutting Coors ball mill gaskets with a 5 ton hand press.



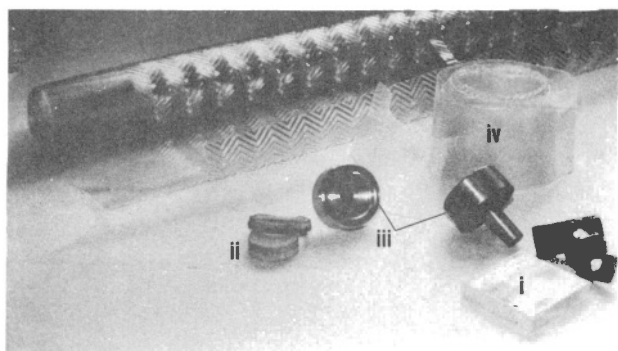
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b. Study set of larger mineral and rock samples prepared by the Geological Survey of Canada.



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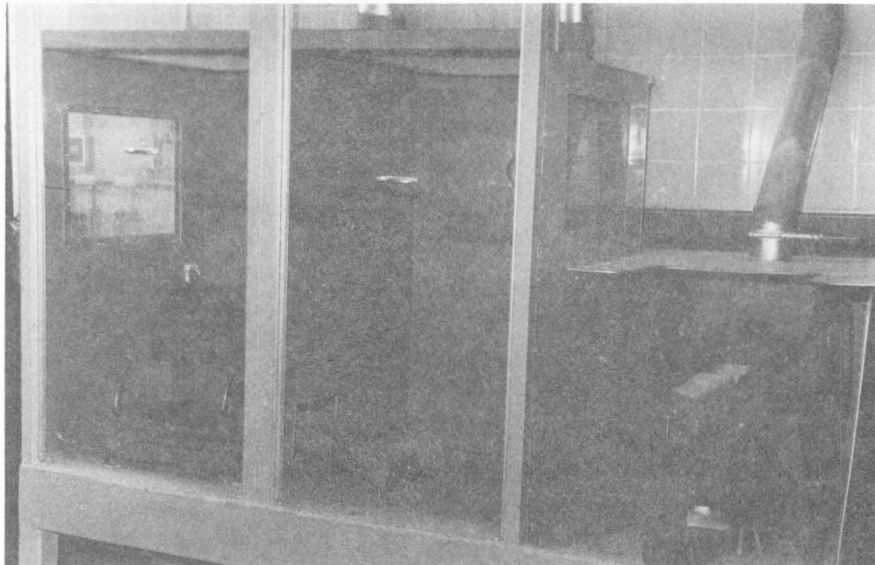


## PLATE 9

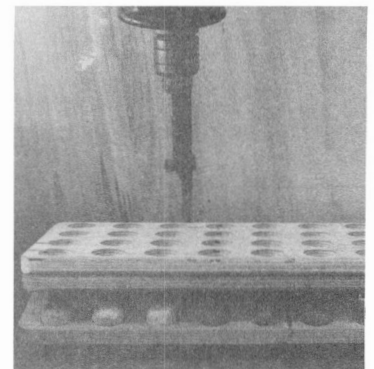
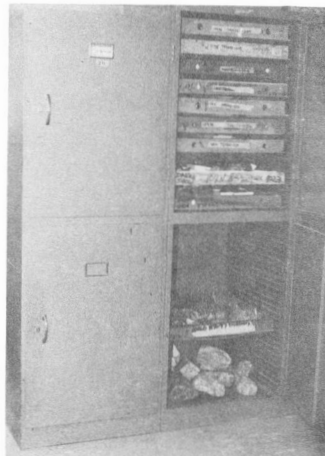
### Figures

1. Sketch showing hood and base for crusher, grinder and sample handling table.
2. Open sample tray storage cabinet.
3. Closed sample tray storage cabinet.
4. Sketch of a jig template used to drill holes to hold plastic vials in a plywood board.
5. Handle with slug used as nut to tighten Coors ball mill holding rack security pin.
6. Mechanical mortar and pestle manufactured by Thos. Dryden & Sons. Preston, USA.

# PLATE 9

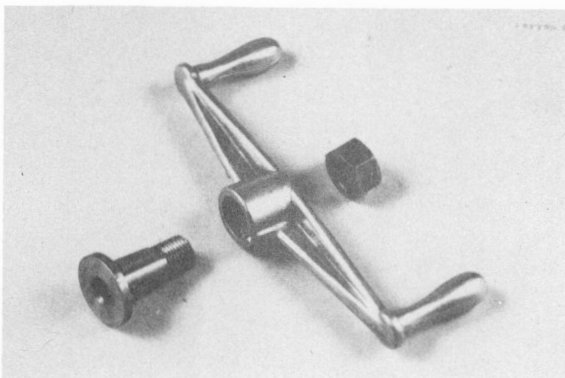


a. Hood and base for crusher, grinder and sample handling table.

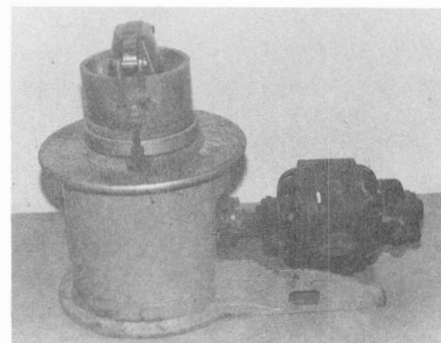


d. A jig template used to drill holes to hold plastic vials in a plywood board.

b. Open sample tray storage cabinet. c. Closed sample tray storage cabinet.



e. Handle with slug used as nut to tighten Coors ball mill holding rack security pin.



f. Mechanical mortar and pestle manufactured by Thos Dryden & Sons, Preston, USA.



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