SAMPLING

By G. S. Moberly

A meeting of the Natal Branch of the South African Chemical Institute was held at 8 p.m., at which members of the South African Sugar Technologists' Association were invited to be present.

The following paper was read by Mr. G. S. Moberly:—

It should be an axiom to all chemists that the prime object in sampling is to obtain a small fraction which shall be as closely as possible representative of a large bulk. The whole value of careful analytical work is negatived if the sample is not a true one. While much of the analytical work done in our laboratories is a mere routine repetition, the work of sampling is one which continually demands intelligent handling and is not one which may be safely left to unskilled or semi-skilled labour as is too often done. The importance of correct sampling cannot be over emphasised. Where materials are bought on test vast sums of money may be involved and faulty sampling may cause thousands of pounds to be spent in avoidable litigation.

Sampling falls naturally into two divisions, the bulk sampling and sub-sampling. The bulk sample consists usually of an aliqot portion of each package, load or other division of the main bulk. This sample is generally too large to be handled in the laboratory. Sub-sampling consists of the subdivision of this larger sample into a smaller portion or portions of a suitable size for use. Again a distinction may be made according to the physical state of the material, i.e., solid or liquid or gas. Of these liquids and gases present the least difficulties owing to the ease with which they may be sub-divided. Solids, on the other hand, are far more difficult to deal with, and this difficulty increases with the size of the pieces constituting the bulk. Powders, grain, etc., are comparatively easy, but coal, ore, etc., are much more difficult of all. Sugar cane is an example of difficult substance to sample; the bulk deliveries vary from 3 to 30 tons, consisting of sticks weighing about half-a-pound each, with considerable variations between stick and stick, while the sub-sample finally tested is only a few pounds.

Another difficulty is that with many materials, chemical and physical changes take place which necessitate very rapid sampling. This applies particularly to changes in moisture content, and in many cases a special sub-sample is made for moisture determination and another more finely divided one for chemical analysis.

In studying the various sampling methods employed it would be well to take the more difficult cases first and consider solids, such as coal and ore, which consist of large hard lumps of varying sizes mixed with finer portions and powder. The material is not homogeneous, as it contains portions of shale and rock, which must be present in correct proportion in the final sub-sample.

Bulk samples may be taken from the loading grabs, one shovelful being taken from each second or third grab according to the amount to be sampled. Where the material arrives in railway trucks, portions may be extracted with a shovel or trowel from regularly spaced points below the surface of the load. One method is to throw a wide-meshed rope net over the top of the truck and remove a portion from beneath each knot. The material on the surface has probably dried out, and this should be scraped away and the portion taken below the surface, care being taken that large and small portions and fines are removed exactly as the shovel takes them, and that the large pieces do not fall off or the fines blow away. Any large piece intercepting the shovel should be removed by hand and added to the sample. When sampling from the holds of ships portions should be removed in a similar way from below the surface of that part of the cargo which lies below the hatches and can be reached directly by the grabs. When this has been removed, portions are then taken from equally spaced points along the vertical face of the remaining bulk, the points being spaced out by using a shovel as a measuring rod. As this face is dug away fresh portions are removed from the uncovered face. If the bulk sample thus obtained is more than half-a-ton when it contains pieces more than 10 inches in diameter, or more than a quarter of a ton containing

pieces from 6 to 12 inches in diameter, it may be immediately sub-sampled to the required size in the following manner: The whole bulk sample is laid out on a hard flat surface and thoroughly mixed with shovels and then piled up into a conical heap, the top of which is flattened out. The pile is then marked off into quarters by two intersecting lines, and either one quarter or two opposite quarters shovelled away to one side and the remainder rejected. This can be repeated until the pile is of the desired size. The remainder should then be broken up with a hammer into pieces of two or three inches diameter and again divided as before. The remaining portion should then be broken up still smaller and again divided, the process being repeated until the sample consists of fragments no larger than will pass through a 3 mm. sieve. It should then be well mixed and a portion placed in an air-tight container and sent immediately to the laboratory for moisture determination. The re-remainder should be again divided and about 100 gm. crushed in a mortar to a fine powder, placed in an air-tight bottle and sent to the laboratory for complete analysis.

Various mechanical devices are in use for sub-dividing finer aggregate. The riffle is a rectangular hopper, the bottom of which consists of a number of parallel chutes leading out alternately to right and left so that material thrown into the hopper emerges in two separate portions on either side.

Finer materials are naturally easier to deal with, but the principle of sub-division and reduction applies to all aggregates consisting of hard lumps. Materials consisting of grains, crystals or powder may be sampled by taking small portions from each bag or from every second, third or other number of bags, pockets or other containers, and sub-dividing as before, but without reduction or grinding until the final sub-sample is reached.

Materials in bags, such as sugar, cement, flour, etc., may be sampled by means of a trier (a pointed auger of semi-circular section), which is stabbed into a bag and carries a portion of the contents with it when withdrawn. For loose dry material which does not cake, it is sometimes necessary to use a special design of trier with a trap at the point which closes when the trier is withdrawn. Another type consists of two concentric tubes, each with a longitudinal opening. When inserted, the openings of the two portions coincide and the inner tube is filled. The two sections are then given a half turn in relation with each other so that the opening is closed, and the trier is withdrawn. This last instrument may also be used for viscous liquids.

When the material is weighed the sample may often be taken at the scales, a portion being taken from every scaleful or every second or third scaleful.

When sampling rock in situ, portions should be broken off the working face, care being taken to include the correct proportions of the different substances that will have to be quarried. This is a form of sampling for which it is impossible to lay down definite rules, and reliance must be placed in the intelligence and experience of the sampler. A number of methods of sampling soil have been evolved. The following is the official method of the South African Department of Agriculture:

> " Having selected a representative spot, any vegetation upon it is removed, and a hole dug with a sharp spade to the necessary depth so as to be smooth and vertical, and the hole is cleaned out. A slice of uniform thickness (back to front) and width (side to side) is then removed by means of a spade, chisel, or other suitable instrument, down to the required depth. This slice, which should be about three inches thick is placed on a clean board or sack and mixed with similar slices, obtained in the same way from holes dug in the same type of soil at other parts of the field. Finally all the samples are thoroughly mixed together and a portion (about 9 lbs.) placed, with a label giving details, in a clean box, or suitable bag of canvas cloth, or other material through which the fine soil will not sift during conveyance to the laboratory. The sample of the sub soil is then taken in the same way and must be kept separate from that of the soil proper."

The " necessary depth " referred to above is the depth of the top soil which depends on the depth of cultivation, which is usually eight or nine inches. As a rule a clean line of division is noticeable between the top soil and the sub soil. Soil samples may be taken with a soil auger, a large auger being used for the top soil and a small one for the sub soil, thus avoiding contaminating the sub soil with the top soil. Another useful method is as follows: Make a wooden box 12in. square and as deep as the depth of the top soil. Place this box with the open top downwards on the surface of the ground from which all vegetation and loose stones have been removed. Now dig away the soil around the sides of the box and push the latter downwards as the soil is removed. When the bottom of the box is flush with the ground, insert the shovel under the box and remove the latter with the section of soil it contains. Whatever method is used, samples should be taken from as many evenly spaced points in the field as is practicable, then united and sub-sampled.

The following method of sampling sugar cane has been recommended by the South African Sugar Technologists' Association Sub-Committee on Fibre Testing:—

> " Sixteen sticks should be taken at random from different points along the carrier (from at least 75 per cent. of the consignment). These sticks are taken to the laboratory and each one is cut up

into four lengths. These lengths are placed in four piles—all the tops in one pile, all the butts in another, and so on. These sixteen lengths are then chopped into sections one inch long. (This can be done with a suitable machine.) These small sections are then well mixed in a basket or bucket and a double handful extracted and passed through the shredder. A Hyatt reducer or a Gallois cutter is recommended for this purpose. One hundred grams of the shredded cane is taken for testing."

The following is the official method of the South African Sugar Technologists' Association for sampling cane in the fields:

"All the canes from a representative stool are taken as a sample, each stool to be taken not less than fifteen yards from any boundary or break. The number of stools to be taken for a sample from a number of fields of the same age should be as follows:

Area in Acres.	No. of Stools.
10	1
25	2
50	3
100	4
200	5

Each cane should be trashed and topped one node below where the lowest green leaf joins the stem."

The sampling of liquids is much simpler than that of solids. Where the sample represents the contents of one tank (or other large container), the contents of the tank should be agitated, and after thorough mixing a small portion removed as the sample. When several tanks are to be sampled together, a proportionate amount should be taken from each tank, these amounts mixed together and a smaller sample taken from them. When the liquid is contained in carboys, barrels, etc., a sampling pipette or tube is inserted through the whole depth, the end sealed by the finger and the contents removed. All liquids should be shaken before sampling unless it is specially intended to exclude sediment. When liquids are pumped, a small hole may be made in the delivery pipe, to which a small nipple is attached, so that a small quantity drips into a receiver. If the hole is made in the pump head a small quantity will be ejected at each stroke of the pump. When the liquid flows in gutters, a small wheel, such as a bicycle wheel, may be suspended in the flow with small vanes attached so that the wheel rotates. The juice is caught in cups attached to the wheel, and these cups empty into a trough when they reach the top of the wheel. This method can be used for cane juice leaving the milling plant. When liquids are weighed during the course of manufacture, a small portion may be removed from each scale. Viscous liquids may be sampled by plunging a smooth stick through the whole depth of the container and then wiping off the adhering liquid. This method is used for sampling cream, and would be suitable for sampling molasses. The method of inserting a cup into a tank of molasses and taking its contents as representative is not satisfactory.

Gases do not present any difficulty. Gases under pressure may be tapped and collected over water, or if they are absorbed by water, they may be collected over mercury or other liquid. Gases at atmospheric pressure can be withdrawn by means of an aspirator. The following method can be used and is useful for sampling flue gases. The vessel in which the gas is to be collected is connected at the upper end via a stop-cock to the gas supply. The lower end is connected by means of a flexible tube to another vessel containing a liquid. When the stop-cock is opened and the vessel raised, the gas collector fills with the liquid. When the second vessel is lowered the liquid in the collecting vessel falls and the gas is drawn in. By timing the gradual lowering of the second vessel, a sample representative of the gas over a given period can be drawn.

The above is but a broad outline of the general methods used in sampling. Every material and every set of conditions presents its own problem which the chemist may solve for himself, always remembering that unless the sample is representative of the whole bulk, he might save himself the trouble of making any analysis at all.