POWDER TESTING GUIDE
Methods of Measuring the Physical Properties of Bulk Powders

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Foreword

Standard methods of measurement of physical properties of powders in relation to bulk handling and processing have been adopted for specific materials and have sometimes been incorporated into a BS or other Standard. However, general standardised methods have yet to be established and the BMHB believes that much wider applications could be made of proven techniques which, while being well established in specific industries, have yet to be generally exploited. The aim of this Guide is to review, detail and recommend preferred test methods, to outline their significance and to identify test methods on powder characteristics which require further research, development or evaluation. The accent is on simple and inexpensive techniques.

The Guide draws on existing Standards, Trade and Research literature and on expert knowledge of selected designers and manufacturers of powder handling plant. It is anticipated that the Guide will prove valuable in disseminating knowledge on simple powder testing techniques and assist in establishing better communication between equipment users and suppliers.

The Guide was prepared by Dr L. Svarovsky, University of Bradford, with the help of a British Materials Handling Board Steering Committee of the following membership:

Dr P. C. Knight, Unilever Research
Dr D. Geldart, University of Bradford
Mr R. E. Pace, Simon Engineering

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An acknowledgement also has to be given to the authors of the Bulk Solids Physical Property Test Guide published by the BMHB as much of the background reference was drawn from this publication.

Scope

The Guide confines itself to dry solids (as opposed to wet cakes) but includes the effects of air humidity. It deals mostly with powders, typically finer than about 3mm, and it excludes detailed description of electrical and thermal properties and explosion/fire hazard testing. The reader is referred to Ref. 82 for these.

The emphasis is on bulk or "technological" properties of powders and the primary properties like particle size, shape and distribution are treated only as background. The problems and importance of sampling are included but merely for guidance rather than in technical detail.

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INTRODUCTION

In the process of compiling this Guide, it became clear that powder testing can be put into three categories as follows:-

Category A where standardised tests already exist and the interpretation of data is well established and accepted; the only work needed is in publicising their existence and use.
Category B where test methods exist but there is disagreement as to the significance of the measured values in equipment design and scale-up; validation testwork is clearly needed here.
Category C where no standardised test procedures are yet available for a particular powder property and these need to be developed experimentally.

The Guide is not written in the order of the above categories but they are assigned at the end of the discussion of each test method. The bulk powder tests are reviewed in the order of the following groups of powder properties to which they relate:

Properties dependent on single particle characteristics
Properties of non-aerated powders
Packing properties, bulk densities
Grinding and strength properties
Properties of aerated powders

Prior to embarking on the review, as a general observation, it is interesting to note that there is an imbalance in the area of powder testing in favour of very detailed particle size and shape

measurements using very sophisticated and expensive laboratory instrumentation, without yet having the fundamental correlations to translate the measured data into secondary behaviour. With the notable exceptions of research organizations or large companies with specialised goals and applications, such intense preoccupation with very detailed physical data is often made at the expense of testing for other important powder properties. The number and quality of the books available on particle size measurement, in contrast to those on other powder testing methods, is a good indication of this imbalance; it is hoped that this Guide will help to redress the balance.

Another point to be made here concerns the effects of gas properties. Generally, the effect of the interstitial and surrounding gas on the mechanics of dry solids handling is neglected. The effects of gas properties may be two-fold:

1. An aerodynamic interaction between the gas and the solids, mainly controlled by the dynamic viscosity of the gas and the elasticity of the packed solids.
2. A physical-chemical interaction through adsorption of the gas on the solid surface, which affects the resistance to breakage, attrition and abrasion. Thus the properties of the suspending gas have to be taken into account, or controlled, not just in the obvious applications where gas clearly plays a part like fluidization or pneumatic conveying but also in the not-so-obvious ones like grinding and discharge of powders from hoppers.

Moisture content in the gas is known to affect most solids handling properties to the extent that, if powders cannot be kept reasonably well sealed, air-conditioning is necessary if meaningful data are required from powder tests.

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Sampling

It is often said that any test on a sample of powder can be only as good as the sampling technique used for collecting the sample. As most laboratory tests use only a small sample, this has to be taken from a production stream or from an existing, stored material and it has to be representative of the whole.

Unlike fluids, the properties of powders are susceptible to change under applied load, they may consolidate with time, and attrition and segregation occur in transfer. In particular, the facts that powders have a size distribution which affects so many of the powder properties so much and that segregation of stratification by size is so common, make representative sampling absolutely critical for the success and relevance of any subsequent testing.

Sampling is therefore such an important element of powder handling that it demands careful scientific design and operation of the sampling systems. The purpose is to collect a manageable mass of material (= sample) which is representative of the total mass of powder from which it was taken. This is achieved by taking many small samples from all parts of the total which, when combined, will represent the total with an acceptable degree of accuracy. This means that all particles in the total must have the same probability of being included in the final sample. All parts of the total have to be equally accessible.

To satisfy the above requirements, the following basic "golden" rules of sampling should be followed whenever practicable.

1. Sampling should be made preferably from a moving stream (this applies to both powders and suspensions) but powder on a stopped belt can be sampled.
2. A sample of the whole of the stream should be taken for many (equally-spaced) periods of time rather than part of the stream for the whole of the time.

It is very likely that the re-combined, primary sample taken from the whole is going to be too large for most powder tests and it, therefore, needs to be sub-divided into secondary or even tertiary sub-samples. This sub-division may be built into the primary sampling system or it may be achieved with a separate sample divider. Allen[1] reviewed and tested most methods available for sample splitting and found the one based on the spinning riffler to be the best.

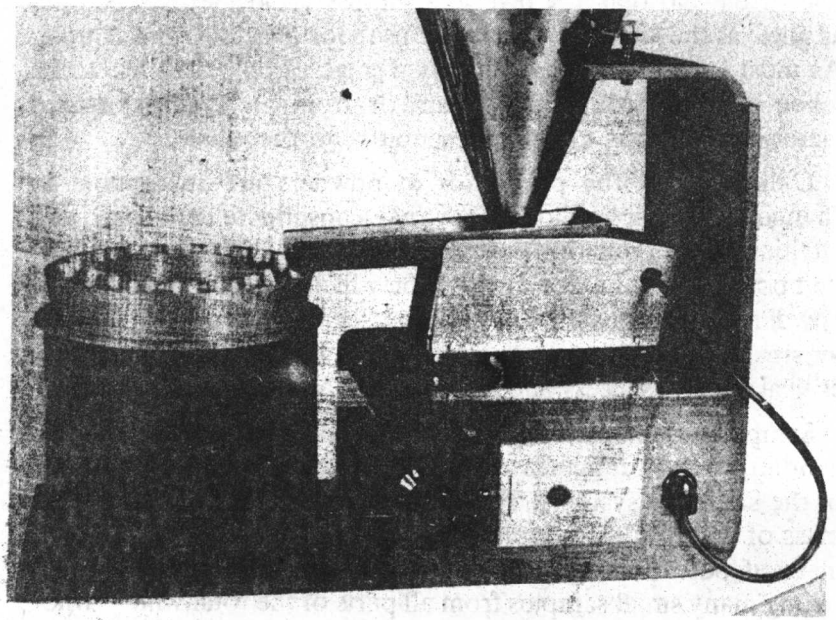

FIG. 1. A view of a spinning riffler.

The principle of the spinning riffler is shown in Fig. 1 and it embodies both golden rules of sampling. The sample is slowly conveyed by a vibratory feeder from the feed hopper onto a

rotating carousel where it is divided into many container ports via a machined rotary head. The sub-samples are collected in these; not all of the container ports may have the containers in (depending on how many sub-samples are required) and where they have not, the powder falls into a bucket underneath. Feed rate is controlled by varying the gap under the hopper and varying the electro-magnetic vibration of the feeder.

Many different commercial instruments based on this principle are available, and they may be built to divide as little as 25 ml or as much as 40 litres of powder or more.

Whilst it is possible to design a range of small sample dividers for general use, the sampling systems for the collection of primary samples from large-scale processes have to be designed specifically for a particular material and application; a short review of such systems is given below.

MECHANICAL SAMPLING

Mechanical, as opposed to manual, sampling is usually preferred because it collects samples with better overall precision, at accurate time intervals and can handle the samples and the whole operation automatically.

The primary samples are usually collected by a primary cutter which cuts the full cross-section of the stream in a way similar to that of each compartment in a spinning riffler (Fig. 1) cutting across the falling stream of powder. The design of such cutters is subject to guidelines laid down in several recent publications.[2,3,4,5,6] Thus, for example[2]:

* The minimum mass of the primary increments depends on the mode of operation (constant mass or constant time between the increments).
* The minimum aperture is related to the nominal top size D of the material being sampled ($=3 \times D$, refer[3] to BS 1017 for the precise definition of D).
* The cutter lips should be normal to the mean trajectory of the stream and of such shape that each part of the lip is in the stream

for the same period of time (i.e. linear cutters should have parallel lips whilst radial cutters should have radial lips).

* The cutter velocity should be less than 0.6 m/s if the minimum cutter aperture of $3D$ is used but can be increased for large apertures according to an empirical formula[2]:

$$v_c = 0.3\ (1 + w/w_1)$$

where v_c is maximum velocity of the cutter (m/s)
w is the actual width of the cutter aperture
w_1 is three times the nominal top size D of the material being sampled, i.e. $w_1 = 3D$.

It should be pointed out here that the above quoted limits of minimum cutter width of $3D$ and the maximum cutter velocity of 0.6 m/s have not yet been accepted universally but Gy[4] has published some experimental work to support this recommendation. The US standard ASTM D 2234 recommends the limit of 18 in/s (0.457 m/s) whilst the Australian Standard AS2646 allows cutter velocities up to 1.5 m/s except for secondary (and subsequent) sampling stages when the limit is 0.6 m/s; both standards referring to sampling of coal.

It is absolutely essential, of course, that the cutter velocity is constant during sampling in order to avoid bias; the maximum permissible deviation in the velocity is usually quoted as 5%.

Types of Cutters

The cutters in use in mechanical sampling are divided into diverter types and bucket types. Both types are used to cut a stream falling due to gravity off the end of a conveyor belt or from the discharge end of a pneumatic conveying pipe or a chute.

The diverter cutters divert the stream increment clear of the main stream and, providing they are properly designed, they do not allow accumulation or sticking up of the powder anywhere inside; they require considerable head-room, however, and can only deposit the increment below the point of sampling and not very far from it laterally.

The bucket-type cutters have the advantage of being able to collect and transport the sample laterally, without the loss of headroom; they collect and hold the sample, however, and thus allow material build-up within the bucket if the powder is a little sticky. Another disadvantage is that, whilst the mass of the diverter-type cutter remains the same during its traverse across the falling stream, the mass of the bucket cutter increases rapidly and the drive systems must be powerful enough to maintain its speed.

As to the different designs available in each of the two groups, the reader is referred to two excellent recent publications by Plowman[2] and Merks[5]. One design worthy of a special mention is the cross-belt type cutter (or rotating hammer sampler) which swings in plane perpendicular to the movement of a conveyor belt and scoops a well-cut sample off the belt. Unlike the other, linear cutters, this one is not limited in its speed but it should traverse the bed of powder on the belt in the shortest possible time.

As was mentioned earlier, the primary sampling system can be operated either in intervals of constant time or constant mass. The constant mass option makes the design and operation of the secondary, subdivision system simpler. It requires a continuous weighing system, like a belt scale, installed near the primary cutter, preferably before it. This monitors the mass flux of the solids conveyed and adjusts the speed of the primary cutter before each cut; this generates a primary increment of constant mass, thus preventing collection of excessive amounts which would overload the secondary system and yet always more than the minimum amount specified for the top size if the handling rate is low.

The secondary sample dividers are used to reduce the size of the primary sample; they can be classified[5] as intermittent and reciprocating cutters, and continuous and rotational dividers. The rotational dividers are usually considered more suitable than linear cutters.

Most standards define the minimum number of cuts that the secondary divider has to take from each primary increment: ASTM D2234, for example, requires six secondary increments to

be collected from each primary increment (for coal). This indirectly controls the division ratios from 1/16 to 1/37. This may not be enough to reduce the sample to an acceptable size (the final system sample should not exceed 20 kg) and tertiary or quartenary dividers may be necessary in large scale systems. Merks[5] recommends that ratios in excess of 1/50 in those stages are not acceptable because they would cause the variance of division to be excessively high.

One aspect of mechanical sampling that is particularly relevant to this guide is bias testing[6]. These are procedures agreed between buyers and sellers in bulk solids handling designed to evaluate the performance of mechanical sampling systems. Such evaluation is based on a statistical comparison of paired measurements in system samples (i.e. samples taken by the mechanical sampling system) and reference samples taken manually (usually from a stopped belt, see below). The reference increments are usually collected in pairs at a spacing of 30 m or less (if from a belt) in such a manner that one system increment is interposed between a set of two reference increments.

The series of paired measurements is then evaluated statistically (usually by Student's T-test) to determine the correlation between the analyses (particle size distribution or some other parameter) of the reference increments and the system increments, to determine any bias (positive or negative) and compare it with a previously agreed maximum permissible bias.

MANUAL SAMPLING

Manual sampling is usually performed in low capacity handling and when the top size of the material is low. It can be done from a falling stream, from a stopped belt or from a stationary pile or hopper.

The first option, from a falling stream, is usually used at a transfer point between conveyor belts, from under a discharging hopper or from the end of a pneumatic conveying system. Open ended scoops or shovels are unsuitable for manual sampling