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Technical Guide No. 4

# Dustiness Estimation Methods for Dry Materials Part 1, Their Uses and Standardization and Part 2, Towards a Standard Method

by The B.O.H.S. Technology Committee



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by
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**Editors' Note**: This Technical Guide combines two papers prepared by the Working Group on Dustiness Estimation of the Technology Committee of the British Occupational Hygiene Society and represents the first two modules of their objectives. The final module of their work is not yet complete, but will be published as a future Technical Guide.

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CONTENTS

# PART 1. DUSTINESS ESTIMATION METHODS FOR DRY MATERIALS, THEIR USES AND STANDARDIZATION

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Abstract.

Methods of dustiness estimation are reviewed.

The uses, benefits and possible standardization of the methods are discussed. The value of a standard method universally applied by manufacturers and users alike is outlined, especially with regard to the development of more effectively dust reduced materials. Gas dispersion methods are considered as unsuitable for development as standard methods for general use.

## INTRODUCTION

'Dustiness' in this paper refers to the tendency of dry materials to liberate dust into the air when handled under specified conditions. It is restricted to materials transfer and processing operations and does not include for example the generation of dust during machining or deliberate comminution. It would however include the dust which could result from previous machining or comminution.

Here it must be stressed that the methods referred to in this paper relate to the estimation of the dust liberation potential of products and not to the usual occupational hygiene concept of concentrations of airborne dust in work areas. A variety of methods exist. They may be qualitative or quantitative, relative or absolute. The products referred to in general are fine powders, dust suppressed forms of powders and alternative forms, e.g. pellets and granules, but all otherwise solid or lumpy materials capable of producing potentially airborne dust are included.

Consequently, the range of application is very wide and covers or could cover for example, rubber chemicals, coal, cement, pharmaceutical products, dry foods, and pigments. In general, most of the methods described provide a means of comparing the relative dustiness of products, thus allowing products to be categorized and assigned a dustiness index based on an arbitrary scale of values. This may for example be a scale of 1 to 10 corresponding to a range of concentrations of dust actually collected and expressed subjectively to cover the range from 'practically non-dusty' to 'extremely dusty'.

Here it becomes rapidly apparent that for the dustiness index values to be meaningful, standardization of both the method and the index is essential.

None of the methods takes toxicity into account. However, for more toxic products, more demanding objectives could be set in terms of the dustiness index indicated by the chosen method.

Commonly the method involves the pouring of a sample of the product into a test chamber. In other methods the sample is placed in the test apparatus and then acted upon to liberate the airborne dust. The dust liberated by whatever means, may then be allowed simply to disperse and settle onto a surface or it may be sampled gravimetrically, photoelectrically or by particle aerodynamical size selection techniques.

Many different industries and research organizations have developed methods for estimating dustiness for particular purposes. Few of these methods have been described in the scientific literature and only one device is known to be marketed commercially (the Heubach Dust Meter, Heubach, Langelsheim, Germany). Recognizing both the increasing importance of the control of dustiness in relation to occupational hygiene and the variety of methods for dustiness estimation in existence, in 1980 the BOHS Technology Committee accepted a proposal that a Working Group should be formed to study such methods with a view to ultimately developing a standard method which could be used by a wide section of industry. This paper represents the first module of the initial objectives of that Working Group, i.e., 'Review the existing techniques for dustiness estimation, together with a review of the benefits accruing from the control of the dustiness of chemicals and details of possible application for industry and industrial research'.

As will be seen later in this paper there exists a most diverse range of dustiness estimation methods. The usefulness of having a single standard method applicable to a wide range of products and giving repeatable and well correlated results between laboratories cannot be overestimated. For example, one of the primary uses of certain of these methods to date has been in the rubber chemicals industry to aid in the development of improved dust suppressed forms such as pellets or granules instead of powder, or pastilles instead of a rather friable flake form.

However, the various manufacturers concerned have different dustiness estimation methods which cannot at present be correlated: indeed, some manufacturers and probably the majority of customers have no method. The ordinary customer would not know, except where extreme differences exist, whether one product was less dusty than another. Whereas, if a standard method existed and was used by both manufacturers and customers, the former would have to compete with each other to satisfy a much more knowledgable customer. Less dusty conditions in workshops and less potential worker exposure are the important end results.

It is important to recognize that the use of dust reduced products has benefits to industry beyond that of health. For example, a very dusty, sticky product will quickly cause seizure of moving parts of materials handling machinery such as conveyors and weighing machines. The following list shows those aspects of industry or processes most affected by high dust problems, and hence those aspects most likely to benefit from dust reduced materials. The major implications of each effect are given in brackets.

a) worker exposure (health risk/worker compensation);
b) dust explosivity (major risk control);
c) loss of product to workshop air or local exhaust systems (economic loss);
d) the need for local exhaust ventilation and the sizing of such systems, hence loss of heat from buildings (capital, running and heating costs); and,
e) the effect on moving parts and the likelihood of seizing or clogging (productivity, maintenance costs).

It may well be that different methods of dustiness estimation will be needed to cover the broad range of aspects indicated above and indeed this would certainly be the case in methods involving direct sampling of the liberated dust cloud. For example, occupational hygienists would be interested in respirable or inhalable particle size fractions, while a plant maintenance engineer will be concerned with dust sizes likely to settle into and interfere with the operation of machinery and electrical control gear.

Returning to the question of dust reduced products, a standard dustiness estimation method would allow the study of the efficacy of the dust suppression treatments to various chemicals to go beyond the manufacturer's laboratory because users could assess how dustiness potential was affected by transportation, standing time in storage, and general attrition of the product as it passes through the process prior to incorporation into a mixing process. Apart from the development and the testing of the efficacy in use of dust suppressed chemicals other applications of a standard dustiness estimation technique can be envisaged, e.g.:

a) product process and quality control, e.g. the design of conveyor transfer points according to the tendency to attrition of the conveyed product.
b) product research (a knowledge of the dustiness of a new product in its various possible forms would be useful).
c) new process design, i.e. assisted by the selection of the most suitable raw materials with regard to their dust suppression qualities, or, given unavoidable dustiness levels, the planning of the necessary environmental control facilities.

## HISTORICAL BACKGROUND

The American Society for Testing and Materials, ASTM (1975) described an 'Index of Dustiness for Coal and Coke', originally adopted in 1939, in which a sample was dropped within a chamber. Subsequently, slides were introduced to collect coarse and fine dust respectively. An apparatus called the 'shatter test apparatus' was described by the NATIONAL COAL BOARD (1957). In this apparatus samples of coal treated with various de-dusting additives were tipped down a tall vertical duct and the resultant dust was sampled by a filter at the end of an adjoining horizontal duct.

HAMMOND (1980, 1981) reviewed some of the methods developed from 1969 onwards. A method developed by HILL & ROBINSON (1969) was purely qualitative and visual in effect and was used for the 'assessment of dust in solid rubber chemicals'.

Following this in 1972 and again in 1974 a detergents' manufacturer developed and patented a simple gravimetric technique (PROCTER & GAMBLE Ltd., 1972, 1974). This was further refined by sampling via an elutriator by a dry food and detergents' research organisation (WELLS & ALEXANDER, 1978). At about the same time rubber chemical manufacturers had developed a test chamber in which a collimated light beam was scattered by the dust to vary the light falling onto a photo-electric cell according to the dustiness of the product (SPIVEY, 1981, RIJNDERS & KATZANEVAS, 1979).

In the US cereal industry a rotating drum technique (COCKE, PERKINS & GETCHELL, 1978) was used to assess the effectiveness of treatments for reducing the dustiness of grain samples. In 1980 Warren Spring Laboratory, Stevenage, UK developed this method as part of a multi-client co-operative project of dust and materials handling. This has involved research into dustiness estimation methods, such as a comparison of dust liberation by allowing the product to fall into a chamber, by tumbling in a rotating drum (WSL, 1981); and by a fluidized bed technique (SCHOFIELD, SUTTON & WATERS, 1978).

Other methods have been developed. RHODEN (1976) described a method in which the loss in weight from a bulk sample of powder after air had been passed through it determined the weight of the fines lost below a certain particle size. DU PONT (1981) patented a method designed for metal chromate pigments in which the sample was tipped from a container within the sampling chamber. A vacuum applied to the chamber after a suitable time delay drew airborne dust onto a filter at the top of the chamber.

More recently in the USA, some large experiments in which quantities of radioactive material were tipped from different heights to simulate accidental spillages, have potential applications in dustiness estimation (SUTTER, JOHNSTON & MISHIMA, 1982).

## REVIEW OF DUSTINESS ESTIMATION METHODS

All dustiness estimation methods consist essentially of a means of liberating airborne dust from the product under test and some means of measuring the resultant airborne dust. It is convenient to classify the methods under discussion here in terms of the processes involved in liberating the dust; namely gravity, mechanical and gas dispersion respectively.

### 1. Gravity dispersion

This class of method allows a defined mass of product to fall into an enclosed space, usually a box-shaped chamber, or to be tipped from a container within the chamber.

Further classification is obtained according to the method used to evaluate the resultant dust cloud i.e. mass determination, light obscuration or visual inspection.

### 1.1 Mass determination

A variety of methods exist which either sample airborne dust by drawing air through a filter for mass concentration determination or collect 'fine' dust which has settled onto a surface for subsequent weighing.

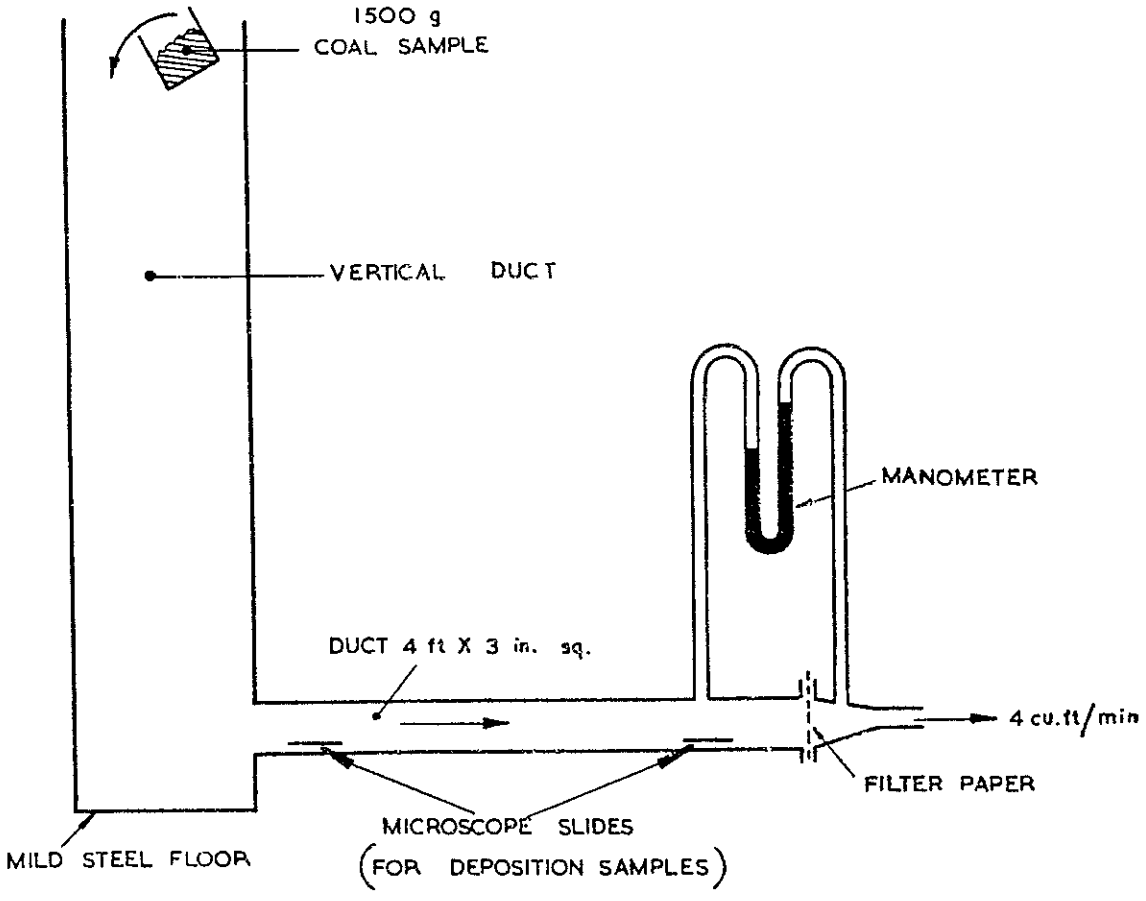



Fig. 1 Shatter-test apparatus. (Courtesy of the Mining Research and Development Establishment, Burton-on-Trent, Staffordshire)

One of the earliest methods reviewed here is that of the NATIONAL COAL BOARD (1957) in which prepared coal samples are tipped down a vertical duct of 18 inches (0.46m) square section and 8 feet (2.4m) height (Fig. 1). Air is drawn along an adjoining 4 foot (1.2m) long horizontal duct at the rate of $4ft^3$/min (113 l/min) which results in turbulent flow conditions. A filter paper at the end of this duct collects dust for gravimetric purposes and glass slides on the floor of the duct collect dust for microscopic study and weighing. Results are expressed in terms of the mass of dust collected on the filter per unit of air resistance measured across the filter, or the same mass per unit optical density as measured by a selenium cell optical densitometer. The glass slides are weighed if the deposits are sufficient, otherwise the number of particles are determined by microscopy. Hence the assessment of dust deposition both along the duct and that reaching the filter is possible. The effects of adding water, wetting agents, salt solution, soluble oil solution, oil alone and a solid binding agent together with the effects of mechanical mixing were all evaluated in the same NCB report.

Subsequently, PROCTER & GAMBLE (1972) described a method in which coarse granular enzyme material is dropped through a funnel directly into one end of a chamber as shown in Figure 2. A flow of filtered air induced by a fan (8) enters the chamber via ports (22), entrains fine dust and carries it to a filter (11) contained within a sampling head (6). Heavy particles and granules are trapped by a baffle plate (23) and deposited into a glass dish (5) or onto the floor of the chamber. The patent claims that different particle size fractions can be selected by adjustment of the air flow, which is controlled by the use of the inlet valve (21) and measured by means of a manometer connected across an orifice plate (19). For collection of enzyme dust, flows of 450 to 550 l/min are used. The enzyme material collected on the filter is subsequently weighed, and analysed if required.

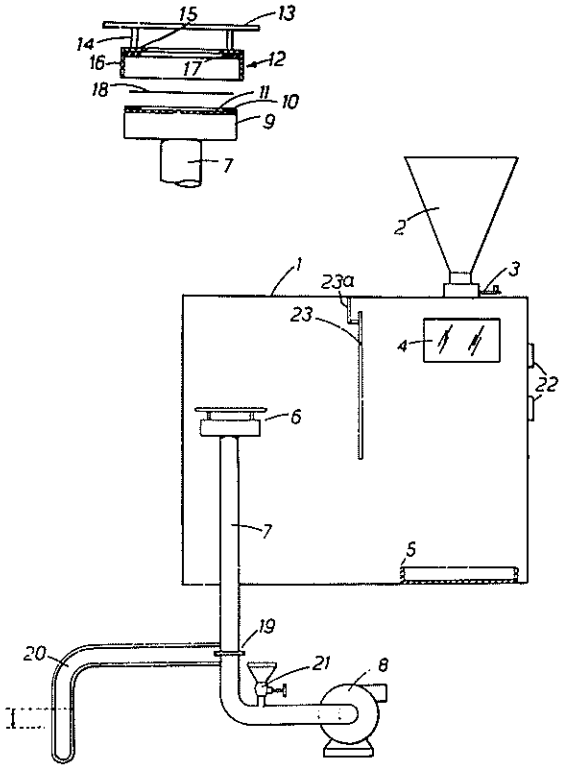



Fig. 2 Apparatus for measuring dust properties of granular material. (Courtesy of Procter & Gamble Ltd., Newcastle-upon-Tyne)

As a logical development from the foregoing, WELLS and ALEXANDER (1978) described an apparatus (Fig. 3) specifically designed for estimating the dust yield of powders. Similarities to the preceding method can be seen, but the apparatus is much smaller and uses the much lower flow rate of 50 l/min required by the Hexhlet horizontal parallel plate elutriator which is used to determine 'respirable' dust. This obviates the need for a baffle plate. The elutriator is replaced by a filter in a filter holder under similar air flow conditions to obtain 'total' dust.

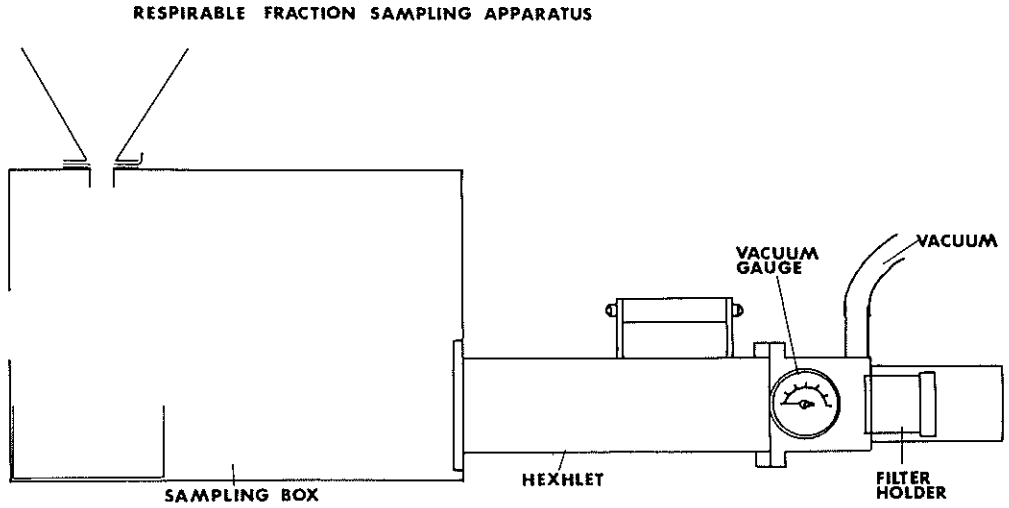



Fig. 3 Apparatus for estimating the dust yield of powders. (Courtesy of Unilever Ltd., Sharnbrook, Bedfordshire)

Dust yield is expressed as mg/100g sample used. WELLS (1982) developed an empirical scale of dustiness categories (Table 1).

Table 1. Empirical dustiness categories (WELLS, 1982).

| Category | Description | Respirable dust mg/100g | Total dust mg/100g |
|---|---|---|---|
| 1 | Non-dusty | $\leqslant$ 0.09 | $<$ 1 |
| 2 | Slightly dusty | 0.10-0.99 | 1-5.99 |
| 3 | Moderately dusty | 1.00-4.99 | 6-12.99 |
| 4 | Considerably dusty | 5.00-12.00 | 13-50 |
| 5 | Extremely dusty | $>$ 12.00 | $>$ 50 |

Wells further comments that the categories given by the total and respirable measurements for the large number of samples tested since this empirical scale was devised are usually the same and rarely differ by more than one category. This is probably because most of the 'fines' are produced by attrition or comminution without any size selection. In the few instances in which there is a marked difference in the categories it is usually found that either a size-selection has been introduced during production or that a different method of manufacture has been used e.g. microcrystallization. An important finding by Wells is that the ranking of the dustiness categories is usually the same whether total or respirable dust samples are measured. This indicates

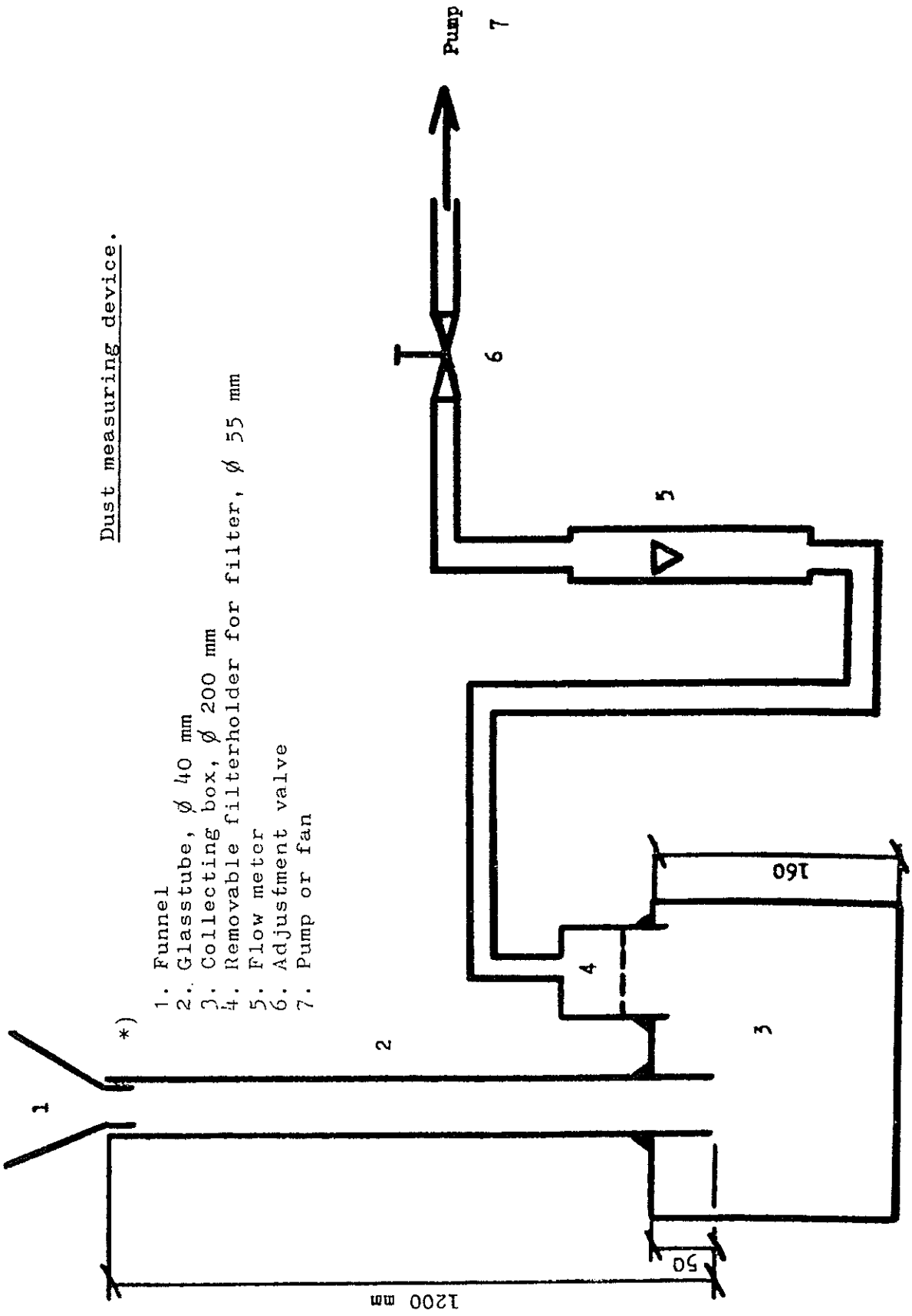



Fig. 4 Dust measuring device (Courtesy of Glasforskningsinstitutet, Sweden)

that the simpler and cheaper expedient of sampling total dust can be reliably used to estimate dustiness.

A similar method to that of WELLS & ALEXANDER (1978) has been developed by the Glass Institute of Sweden (GLASFORSKNINGSINSTITUTET, 1983) and is shown in Fig. 4. In this case 250g of sample is dropped into the chamber via the funnel and glass tube. The sampling rate through the filter is adjusted to 40 l/min. This method is based on an earlier method developed by Verein Deutscher Bleifarbenfabrikanten E.V. (unpublished).

A radically different method to the two preceding methods, but belonging to the mass determination category is that described by ASTM (1975) (originally adopted in 1939). This method was designed specifically for coal and coke and as originally devised the size of the apparatus limits its general applicability. Fifty pounds (about 23kg) of sample is allowed to fall through a distance of about 1.2m in a box. A metal slide is introduced at about mid-point of this box to catch the coarse dust which settles during the period 5 seconds to 2 minutes afterwards. A second slide is introduced for the period 2 to 10 minutes after the sample has fallen to collect the 'fine' dust that settles during this period. The collected dust from each slide is brushed off and weighed separately. WELLS (1982) suggests that this relatively simple method could be modified in a scaled down version incorporating sampling by filter collection and capable of coping with a wide range of types of sample. A prototype has been built along these lines and more details are given in the second paper of this Technical Guide.

Regarding other methods in this category and notably those which involve the tipping of a sample to fall within a chamber, three other methods have been described.

The first was that of SPIVEY (1981). A large chamber (a cube of 3m side i.e. 27$m^3$ capacity) was built to obtain a practical assessment of dust-forming tendencies when tipping the contents of a 25kg bag of product into a hopper steadily over a two minute period (Fig. 5). Total and respirable dust concentrations are sampled for a 30 minute period. The total dust sample is taken by a 25mm open face filter at 2 litres/min to provide a mean mass concentration during the 30 minute period. The respirable dust is sampled at 2 minute intervals to provide incremental and cumulative dust concentrations (mg/$m^3$) over the 30 minute period. In order to achieve reproducible standard conditions the chamber is sealed. This method was devised to simulate a typical product handling situation in a rubber chemicals manufacturer's development of improved dust suppressed products. The purpose was to determine the correlation with a laboratory light obscuration method developed by the same author (SPIVEY, 1981). This latter method is discussed under Section 1.2 below. Whilst a precise correlation between the two methods was not obtained (it is possible that further work could have achieved this), nevertheless the results indicated that the light obscuration method gave a reasonable measure of the dusting tendency of the material as measured by the large chamber method.

The second of the three methods which utilise a tipping technique is that which DU PONT (1981) developed for determining the dusting properties of pigment compositions such as metal chromates. The patent specification describes a vertical cylinder (14cm in diameter and about 30cm high) near to the top of which is mounted a small container (to hold typically 45g of material) which can be inverted by rotating the

supporting horizontal shaft (see Fig. 6). Upon inversion of the internal container the test sample drops 24cm to the bottom of the cylinder. Three seconds after this a vacuum of 60mm Hg is applied for 10 seconds to the top nozzle to draw air through the filter (item 5) in the filter holder (item 3). The amount of dust collected by the filter is assessed by visual inspection, which suffices for the highly coloured pigments for which the method was devised, or by analysis for quantity of metal or other components present.

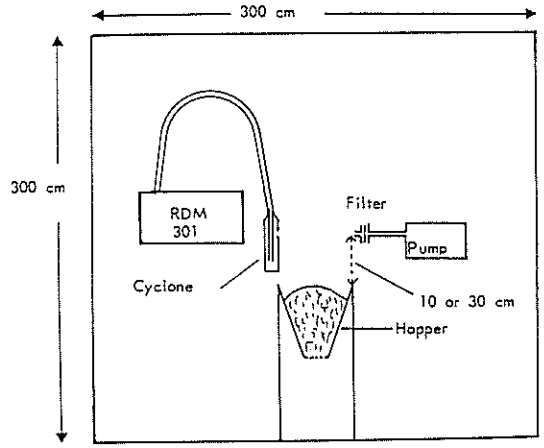



Fig. 5 Dust chamber (side view, dust sampling equipment not to scale). (Courtesy of Monsanto Europe S.A., Louvain-La-Neuve, Belgium)

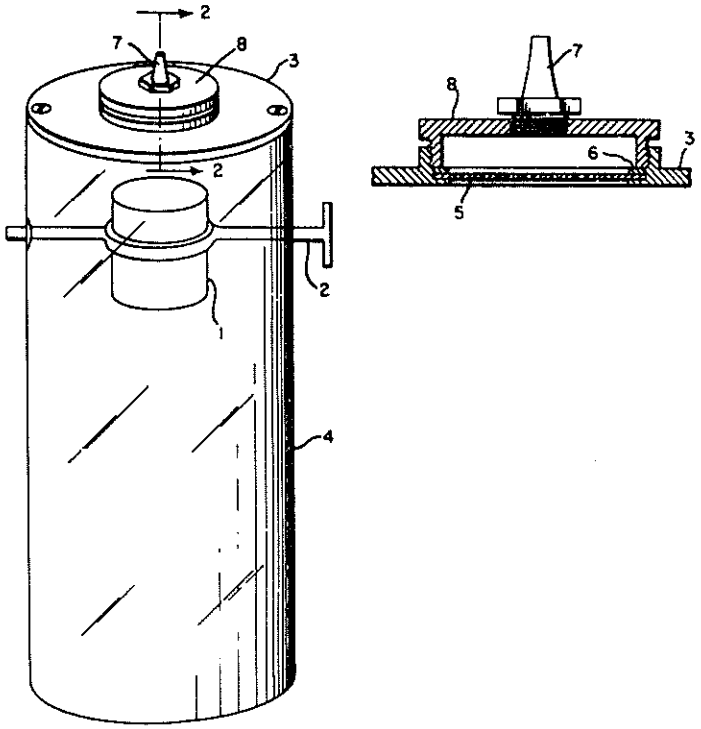



Fig. 6 Dust index apparatus. (Courtesy of Du Pont (UK) Ltd., Stevenage, Hertfordshire)

The third tipping technique which has recently come to our attention certainly has important occupational hygiene connotations and is relevant to our present concern with dustiness estimation. A large experiment is described by SUTTER, JOHNSTON & MISHIMA (1982) which details preliminary studies relating to the environmental impact of

large spills of radioactive materials. The studies were carried out in a stainless steel tank 3m high and 2.9m in diameter (Fig. 7). This was considered to represent the 'lower boundary accidental release event' by allowing the study in static air of different masses dropped from various heights. The variables chosen were drop heights of 1 and 3m and 5 masses ranging from 25 to 1000g. The materials chosen were $TiO_2$ powder traced with uranine, depleted uranium powder, uranine solution and uranium solution. The sample under test is tipped as a single mass from the selected drop height and allowed to drop to the impact area at floor level. The samplers at various locations (see Fig. 7) are then turned on for 30 minutes by which time the samplers have created eight air changes in the tank and 99% of the airborne material is collected.

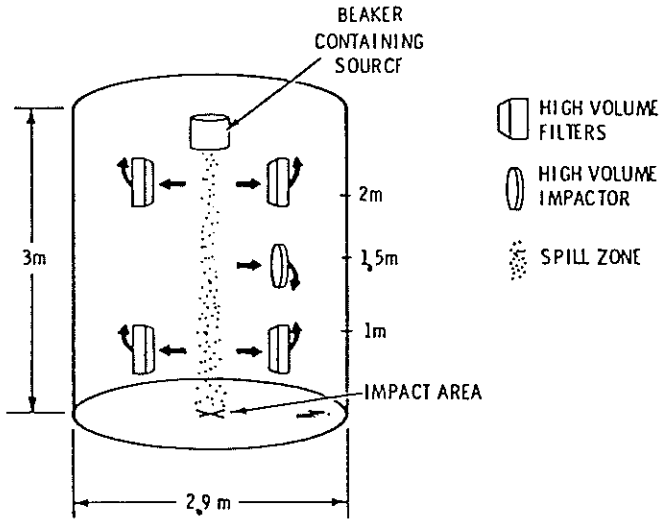



Fig. 7 Sampling from a free fall spill with high volume filters. (Courtesy of the American Industrial Hygiene Association, Akron, Ohio)

It was concluded that source quantity and spill height are both important in aerosol generation and all spills produce a substantial fraction of particles of 10μm aerodynamic diameter and smaller. The results showed that in the case of $TiO_2$ for the 3m spill height the collected weight was consistently about 0.1% of the source weight; the results for 1m spill were more variable but were an order of magnitude less. From a dustiness estimation point of view while this method is relevant to sudden accidental spillages it may not be very typical of industrial powder handling, c.f. the SPIVEY (1981) method already described in which the contents of a 25kg bag were steadily poured over a two minute period. Clearly the aerodynamic conditions and shearing forces, etc. were quite different in the two methods. There are, of course, potentially many further experiments that could be carried out with both methods using a variety of materials and pour rates.

## 1.2 Light Obscuration

There are three known methods in which a dust cloud is created by gravity dispersion and its concentration is assessed by the use of a beam of light. The intensity of the light detected by a photoelectric cell or other light sensitive device is reduced according to the light scattering properties of the dust.

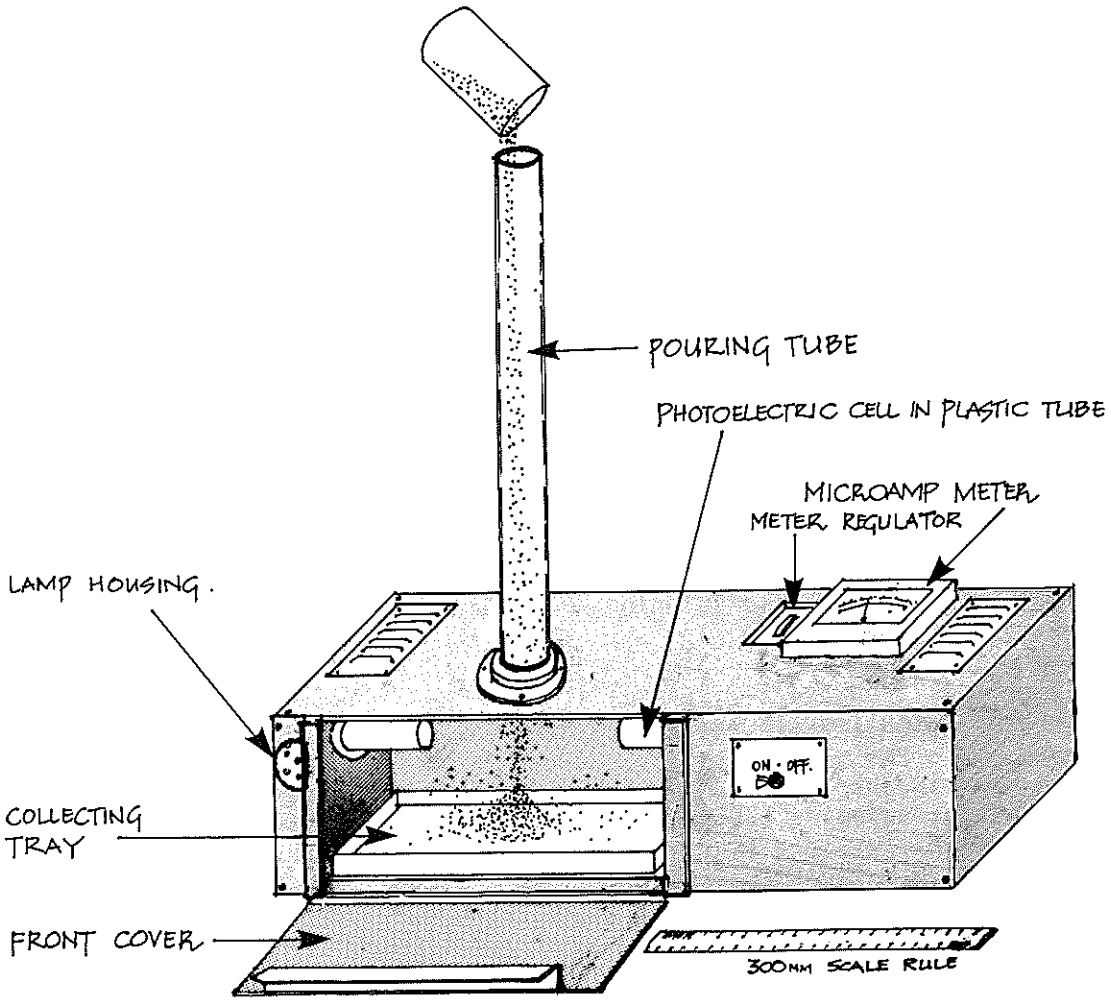



Fig. 8 Dust index apparatus (Courtesy of Monsanto Europe S.A., Louvain-La-Neuve, Belgium)

Two manufacturers of rubber chemicals have developed very similar photometric methods. One of these is described by SPIVEY (1981) as a 'dust index apparatus' (Fig. 8) where 100g of sample (or as little as 20g if dusty) is poured down the 52cm high vertical tube. The light beam is set towards the rear of the chamber and the pouring point towards the front so that the falling material does not directly obscure the light beam. The photoelectric cell current reading is recorded after pouring every 5 seconds for a total of 100 seconds ($I_i$ where i = 0, 5, 10 ... 100) and again at 180 seconds ($I_{180}$). Thus a dust index can be written in the form:

$$\text{Dust index (arbitrary units)} = 5\left(2000 - \sum_{i = 5, 10 \ldots 100} I_i\right) - 100\ \frac{(I_o - I_{180})}{2}$$

where the latter bracketed term is the average blank correction.

Dust indices are assessed in Table 2.

Table 2. Dust index related to subjective assessment (SPIVEY, 1981)

| Dust Index (arbitrary units) | Subjective assessment |
|---|---|
| < 30 | Practically dust free |
| 30 to 100 | Slightly dusty |
| 100 to 300 | Dusty |
| > 300 | Very dusty |

A typical use of this method is in the comparison of original powders and dust reduced forms of the same product (Table 3).

Table 3. Dust indices of untreated and dust suppressed powders (SPIVEY 1981, 1982)

| Product | Dust index (arbitrary units) Normal powder | Dust suppressed powder |
|---|---|---|
| Mercaptobenzothiazole (MBT) | 300 | 40 |
| 2-Mercaptobenzothiazyl disulphide (MBTS) | 220 | 40 |
| Polymerised 1,2-Dihydro-2,2,4-trimethylquinoline (TMQ) | 2500 | 700 |
| N-Cyclohexyl-2-benzo-thiazolesulphenamide (CBS) | 2000 | 100 |

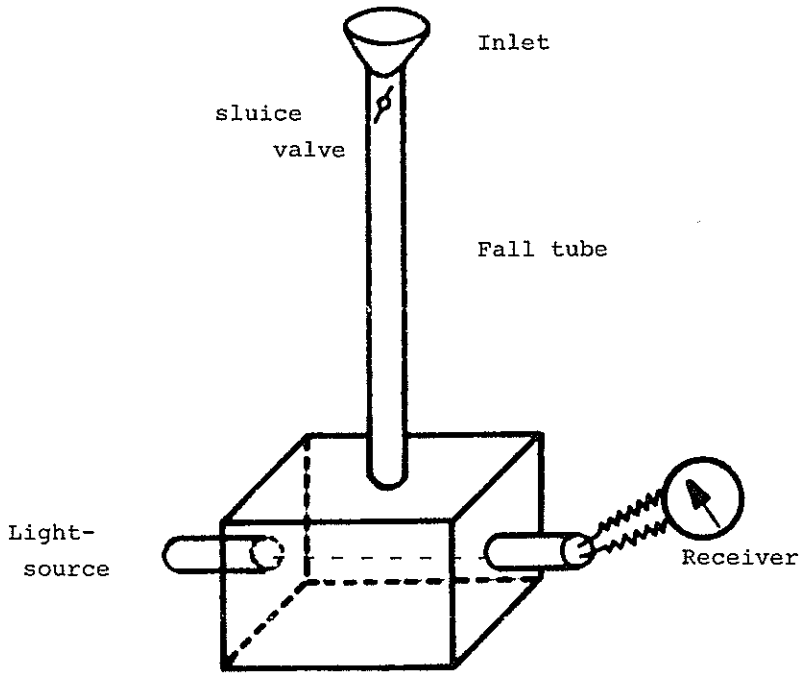



Fig. 9 Dust index apparatus. (Courtesy of Akzo Chemie, Walton-on-Thames, Surrey)

The second apparatus developed by a rubber chemicals' manufacturer is described by RIJNDERS & KATZANEVAS (1979) which appears to be almost identical to the SPIVEY (1981) dust index apparatus described above, but uses a formula for 'dust number' based on just two meter readings, one at zero time and the other after 30 seconds. AKZO CHEMIE GmbH (1982) indicated that the apparatus (Fig. 9) is adjusted initially to give a meter reading of 100. A 30g sample is then dropped down the 'fall tube' and the meter reading is noted immediately; the reading is subtracted from 100. The meter is read again at 30s and this reading is similarly subtracted from 100. The ratio of the initial difference to the final difference in readings is taken as indicating the degree of dustiness. For example 1/0 = dust free; 90/85 = heavily dusty.

The third apparatus (Fig 10) using light obscuration is that of TIOXIDE INTERNATIONAL LTD. (1982) and dates from 1974. A 50g sample of powder is poured down a tapered cylindrical chute within a glass chamber, below which is a collecting beaker. A collimated light beam directed towards the centre of the glass chamber illuminates the dust cloud. A photocell tube is positioned level with and at 90° to the light beam to receive scattered light. The output of the photocell is indicated on a chart-recorder which records the variation in the light scattering properties of the dust with time. Dustiness is assessed either by the time taken for the trace to return to some arbitrary datum or by the area under the voltage-time curve. Light attenuation by dust deposited on the inside of the glass tube during measurement was minimized by painting a white patch on the glass directly opposite and illuminated by the light source. This raised the general level of scattered light received by the photocell and the chart recorder was zeroed before each test drop.

### 1.3 Visual Inspection

Two methods have been found that fit into this category. The first is that of HILL & ROBINSON (1969) which has been used for testing rubber chemicals, pigments and dyestuffs. In this simple and rapid test a Perspex box is placed over a plywood base having a hinged frame which holds a matt black stiff card in place for the test (Fig. 11). A 5g sample of powder is dropped down a central vertical glass tube, and is collected in the glass dish in the centre of the black card. The liberated dust settles to form a halo around the glass dish. This may be photographed or sprayed with adhesive to provide a permanent record. A comparison between two different products may be achieved by bringing half of each card together as shown in Fig 12. HERIOT (1982) comments that a crude numerical index is sometimes used. This involves timing the period over which the airborne dust is visible in the box using a light beam at an angle to give forward light scattering. If no dust is visible after 5 minutes it is given a dustiness index of 1 (non-dusty); if dust is still apparent after 5 minutes a subjective judgement is made scaling the density of the cloud over the range 2 to 5.

The other visual inspection method was that of FORD MOTOR CO (1972) for which only scant information was available at the time of writing. This method was used to test the dustiness of antimony trioxide. A vertical metal shaft guided by a fixed tube is allowed to drop through ¼in (6mm) onto a 0.1g sample of the product placed on a dark vinyl sheet supported by a plywood platform. If the sample spreads less than 3 inches (76mm) from the impact area it would be considered non-dusting and acceptable, whilst if the spread is greater than 3 inches, often with a typical radial striation pattern, it is considered to be dusting.

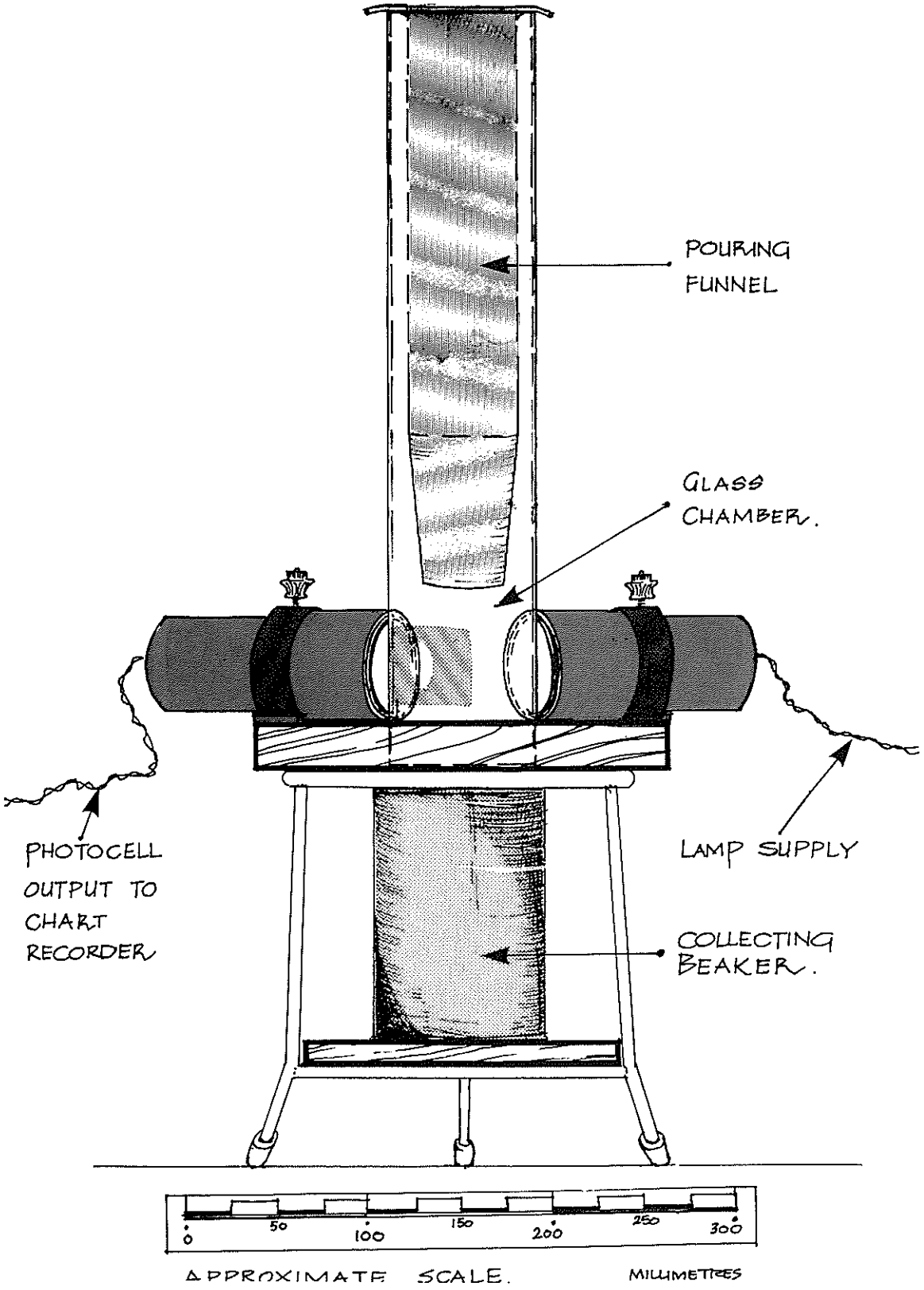



Fig. 10 Dust measurement apparatus. (Courtesy of Tioxide International Ltd., Stockton-on-Tees, Cleveland)

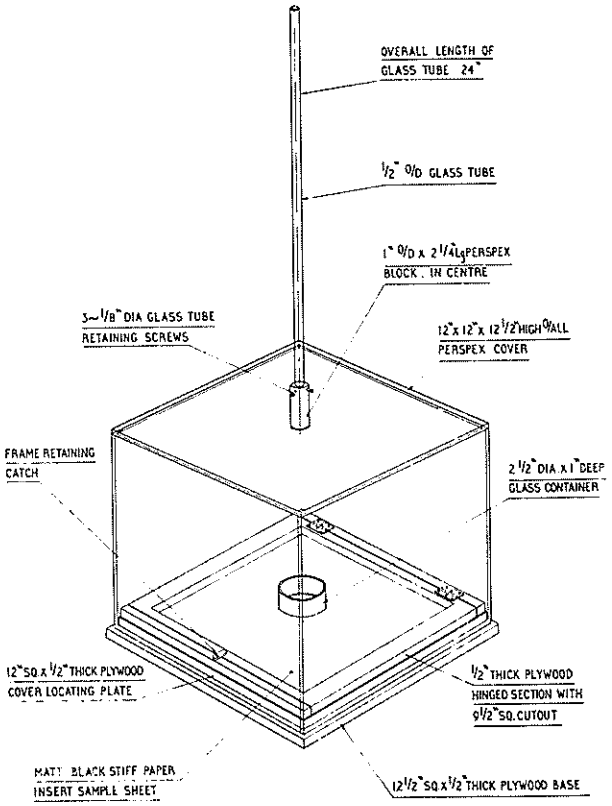
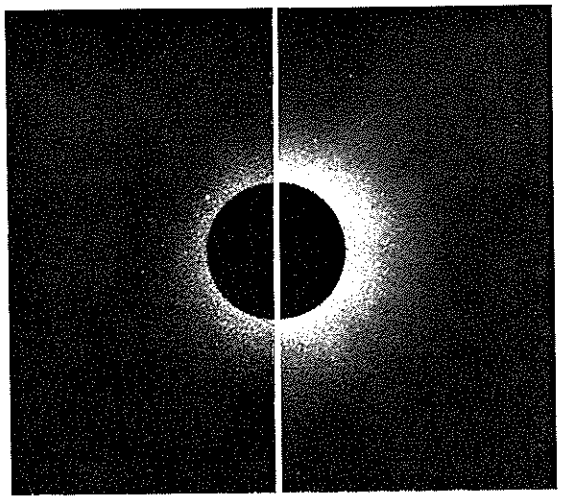



Fig. 11 Dust assessment apparatus. (Courtesy of Vulnax International Ltd., Warrington, Cheshire)



*Comparison of 'Vulcafor' MBTS (SC) [left], with 'Vulcafor' MBTS*

Fig. 12 Example of result using apparatus in Fig 11. (Courtesy of Vulnax International Ltd., Warrington, Cheshire)

## 2. Mechanical dispersion

In this class of method the sample is dispersed either in a rotating drum, of which four methods are known, or, the sample is vibrated above a mesh screen, of which one method is described in the literature. The rotating drum method will be described first.

In the method described by COCKE, PERKINS & GETCHELL (1978) a 68cm by 43cm diameter cylinder with closed ends containing three flights or baffles normal to its inner curved surface is rotated about its horizontal axis at 38 rpm. The flights cause the sample to be repeatedly lifted and dropped to continually liberate dust. The dust is sampled via a filter located within the chamber after running the drum for 2 minutes when it is assumed that equilibrium conditions have been reached. The dust is sampled until it is assumed that sufficient for reliable weighing has been collected, then the filter is replaced and sampling is resumed after a further period of 2 minutes: this sequence is repeated throughout the sampling period. The concentration was found to be fairly constant over 1 to 10 minutes with a 1.8kg sample of grain, and the method was able to show the effect of oil additives in reducing dust yield.

Warren Spring Laboratory (Department of Industry), Stevenage, U.K. found it attractive to develop this idea, because as distinct from the single drop gravity dispersion methods already described, the repeated liberation of dust in the rotating drum method more readily simulates dust liberation in materials handling machinery eg. transfers between conveyors. Hence WARREN SPRING LABORATORY (1981) describes a cylinder as shown in Fig 13. The improvements relate mainly to an increase in the number of internal flights to eight, replacing plain ends of the drum with cones (for better air flow characteristics) and the use of an external size selective sampler, namely an Andersen cascade impactor.

This method has the distinct advantage that lumpy, fine, damp or dry material can just as easily be used. The weighed sample is placed in the drum, the end cone is replaced and the drum set into rotation. This means that long term tests simulating repeated handling can be carried out and all the dust emitted can be collected. This and similar methods can provide information that would require a combination of simpler methods, and takes into account processes such as the tendency to attrition and the breakdown of agglomerates in transfer operations.

The third rotating drum method is that described by NORMAN, PARNELL & GRANT (1977) in which a sample of grain is tumbled at rates up to 85 rpm in a cage fabricated from mesh of 100μm aperture. This is enclosed in a large trapezoidal box from which dust is sampled onto a filter. No further details are given in the reference, but from a general application point of view the method has the insuperable disadvantage that the very fine mesh would clog with many materials.

The fourth method of this type as far as we are aware has not been described in the scientific literature. It is the only commercially available dustiness estimation device known to us and is manufactured by Heubach GmbH of Langelsheim, West Germany. In this device an electric motor rotates a 'dust development vessel' at 30 rpm about a horizontal axis (Fig 14), HEUBACH (1982). Dust liberated from the sample is drawn into a cylinder which separates out the coarse particles, the fine particles being carried through to a filter for subsequent weighing.

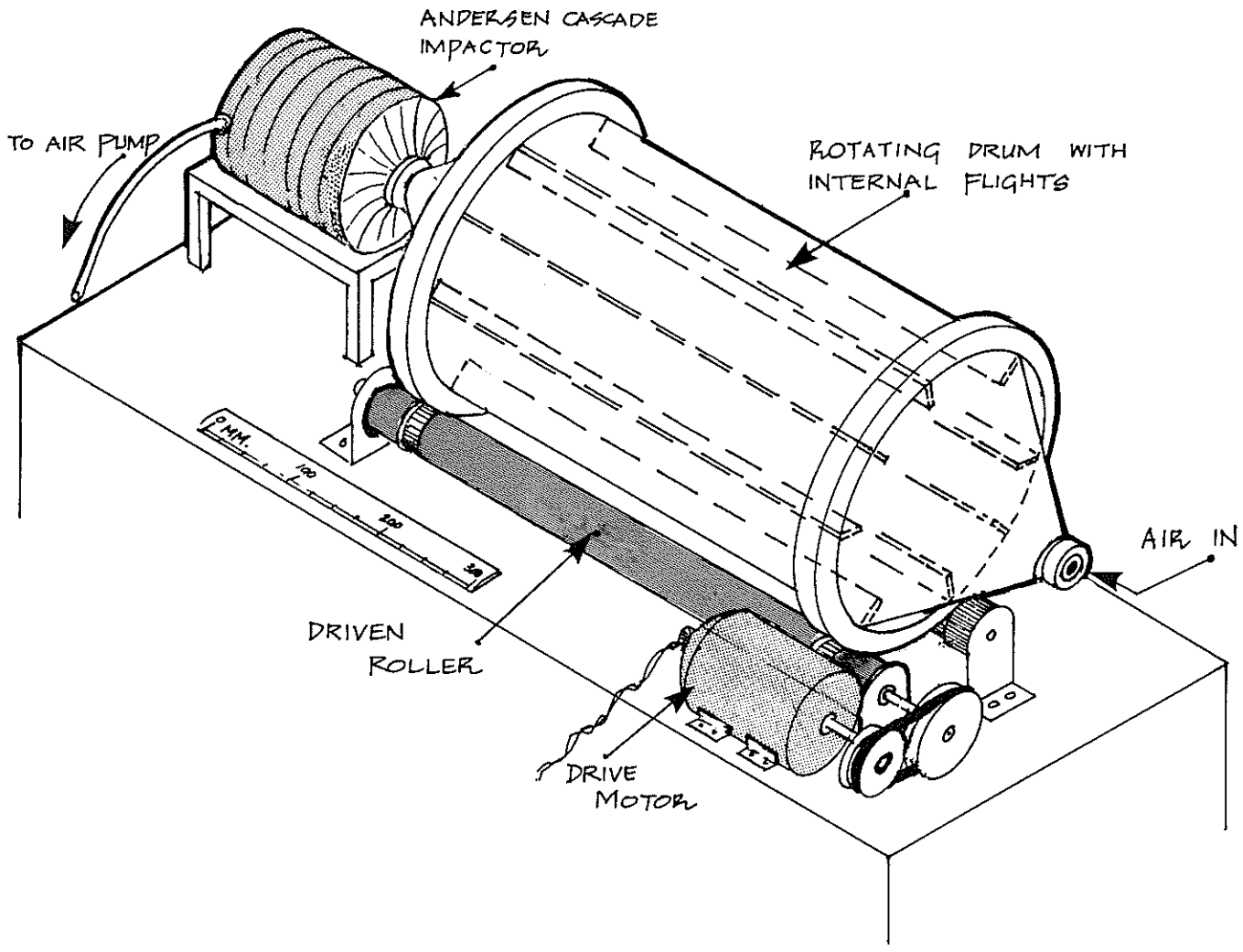



Fig. 13 WSL Rotating drum tester. (Crown Copyright. Courtesy of Warren Spring Laboratory, Stevenage, Hertfordshire)

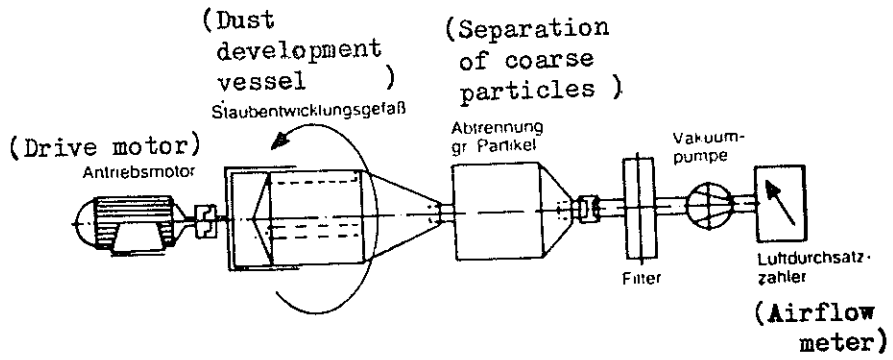



Fig. 14 Dust meter. (Courtesy of Heubach Engineering GmbH, Langelsheim, West Germany)

The only other mechanical dispersion method that we have found is that of DEITZ and PONGPAT (1979) which describes an apparatus designed for testing the mechanical stability of granular activated carbons. The apparatus will collect dust particles which may be too large to become airborne and may therefore be inappropriate for assessing environmental hazards. A cylinder about 50mm diameter and 150mm long with its main axis vertical is supported on a calibrated vibrating table. A 55cm$^3$ sample is placed in the top third of the cylinder to rest on a 60 or 80 mesh stainless steel screen. A glass fibre filter is supported between rubber gaskets at the junction of the middle and lower thirds of the cylinder, and the lower third forms a plenum for a vacuum supply. The cylinder is vibrated for 10 minutes, after which the filter is removed and weighed. Repeats of the test are made with successive filters for the same initial sample. Different samples of the same material are tested at different acceleration rates. The authors found that the quantity of dust in successive 10 minute tests decreased rapidly at first and then became constant, this being attributed to a constant attrition rate once the edges and corners of the granules had become rounded.

## 3. Gas dispersion

In this the third and final class of method, dust is liberated by the action of a gas passing through the sample. Six methods are known, some of which are very similar to each other.

Two of these methods as described by KLEIN & WILCOX (1972) were not primarily concerned with the evaluation of the dustiness of the products involved, but merely characterised them. The primary objective was to determine the effect of blending a hydrophobic silica into preground resorcinol.

The first of these methods refers to the 'elutriation-dispersability' of the blended powders. The apparatus (Fig 15) is a 2ft (610mm) long and 2in (51mm) diameter vertical elutriator at the top of which a filter collects the liberated particles. The sample is dispersed by a 30 msec pulse of dry nitrogen into a winnowing airstream of 1 litre/min. This arrangement liberates 16μm diameter spheres of density 1.3g/cm$^3$. For other densities or shapes, the apparatus must be calibrated each time to

determine the maximum size that would be collected on the filter.

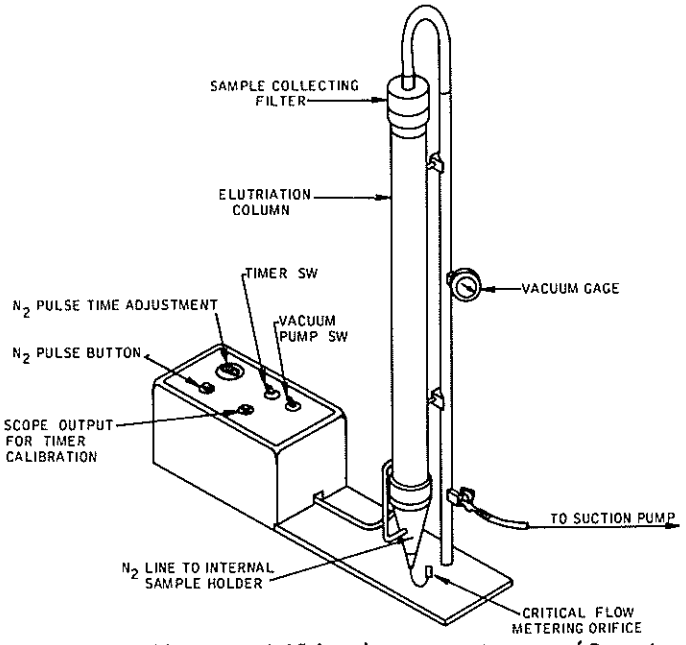



Fig. 15 'Elutriation-dispersability' apparatus. (Courtesy of the U.S. Army Chemical Research & Development Centre, Maryland)

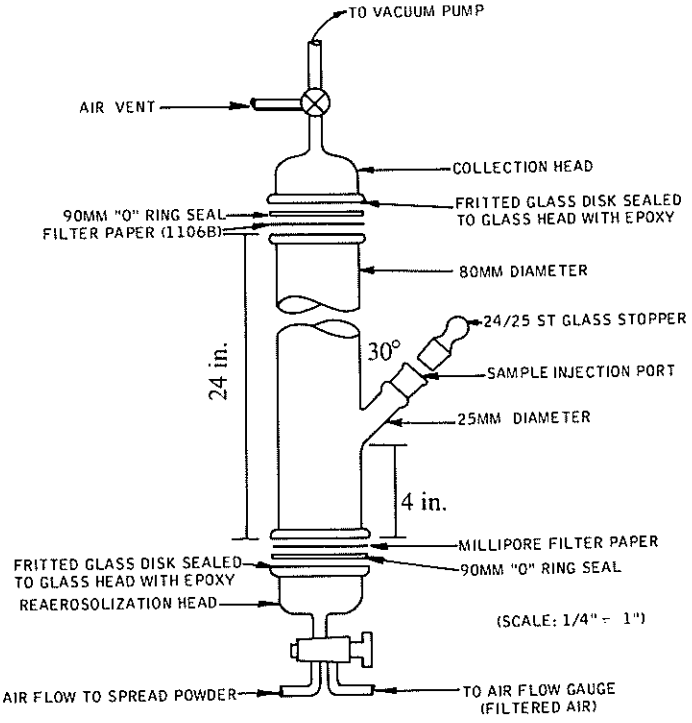



Fig. 16 'Re-aerosolizability' apparatus. (Courtesy of the U.S. Army Chemical Research & Development Centre, Maryland)

The second method referred to a characteristic called 're-aerosolizability'. This describes the capability of a deposited dust layer to become re-entrained into the air when disturbed by relatively weak forces. The sample is injected into the side of a vertical glass tube (Fig 16). It is allowed to settle onto the lower sintered glass disc where it is continuously subjected to an airflow rate of 4.85 l/min. Powder is liberated into the airstream to be deposited on the upper-filter.

Both of these methods yield results in terms of the percentage of collected material weight to sample weight and were used by the authors to rank different blending methods. For the silica-resorcinol blends tested percentages of 25% (dispersability) and 15% (re-aerosolizability) were produced.

Further vertical elutriation work has been carried out at Warren Spring Laboratory using their Fluidized Bed Dust Emission Meter, (SCHOFIELD, SUTTON & WATERS, 1978; WATERS, 1979). In this case a 400g sample is used. This can be the material under test if it is readily fluidized, or, alternatively, dedusted 350 to 500μm sand particles can form up to 90% w/w of the 400g quantity to assist fluidization. The sample is carefully loaded into the bottom of a glass tube (2.05 m long and 70 mm internal diameter) using a special cylindrical container with a remotely controlled bottom trap door. With the sample in place on a porous (sintered) metal bed (Fig 17), air is passed at a fixed rate, 71 l/min (2.5 $ft^3$/min), to fluidize the bed thus liberating dust which passes up the tube. Dust particles are collected isokinetically to pass into an Andersen cascade impactor for particle size distribution analysis. The weights collected in a given time period (usually 60s) are a measure of the test material's propensity to emit dust. Results are usually expressed in terms of $g.kg^{-1}.m^{-2}.min^{-1}$. The area ($m^2$) term in the expression relates to the porous metal bed area.

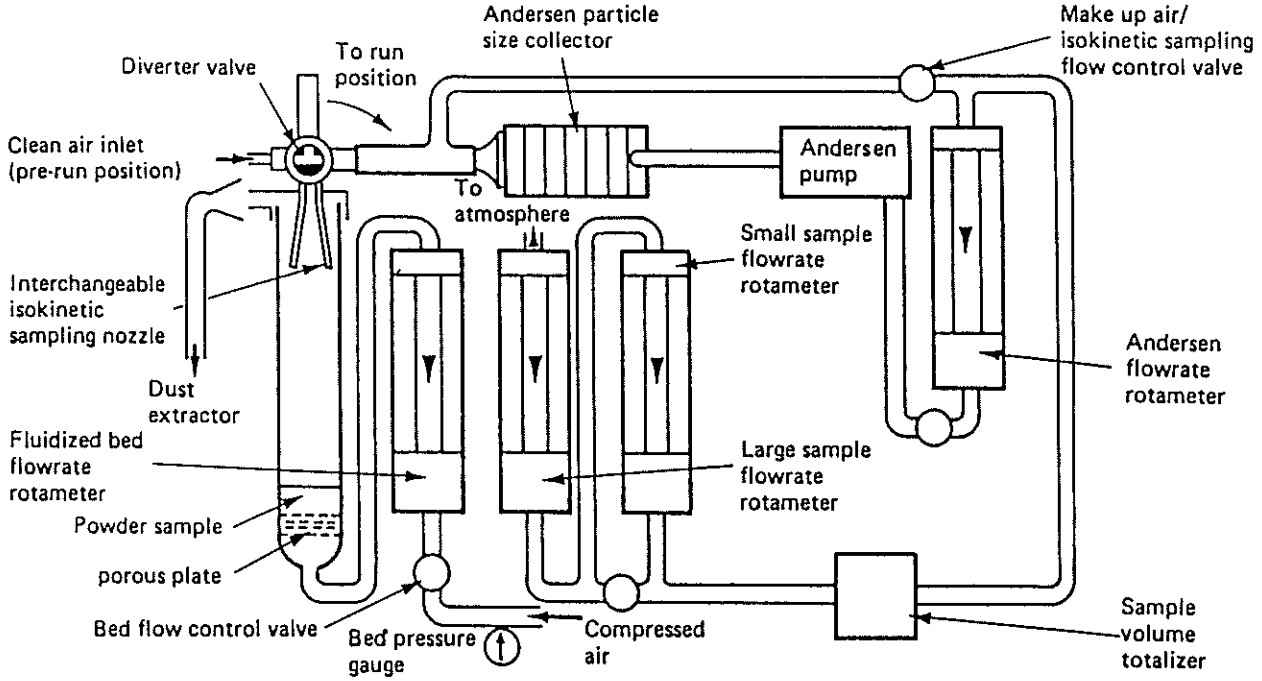



Fig. 17 Fluidized bed dustiness tester. (Crown Copyright. Courtesy of Warren Spring Laboratory, Stevenage, Hertfordshire)

Another gas dispersion method was described by RHODEN (1976). Essentially, a sample of approximately 40g in a glass sample cell is subjected to a controlled air stream entering from the bottom of the cell. The sample is contained in the lower half of the cell (actually a tared glass filter funnel) and weighed before and after the test to determine sample weight loss. The author showed that using Stokes' law a simple equation relating the largest particle size to be elutriated to the square root of the air flow rate, could be derived. For example, for a flow rate of 1 l/s this particle size was estimated to be 55μm. The author demonstrated the feasibility of adding increasing amounts of

a dedusting agent to a product to progressively reduce the percentage of dust liberated.

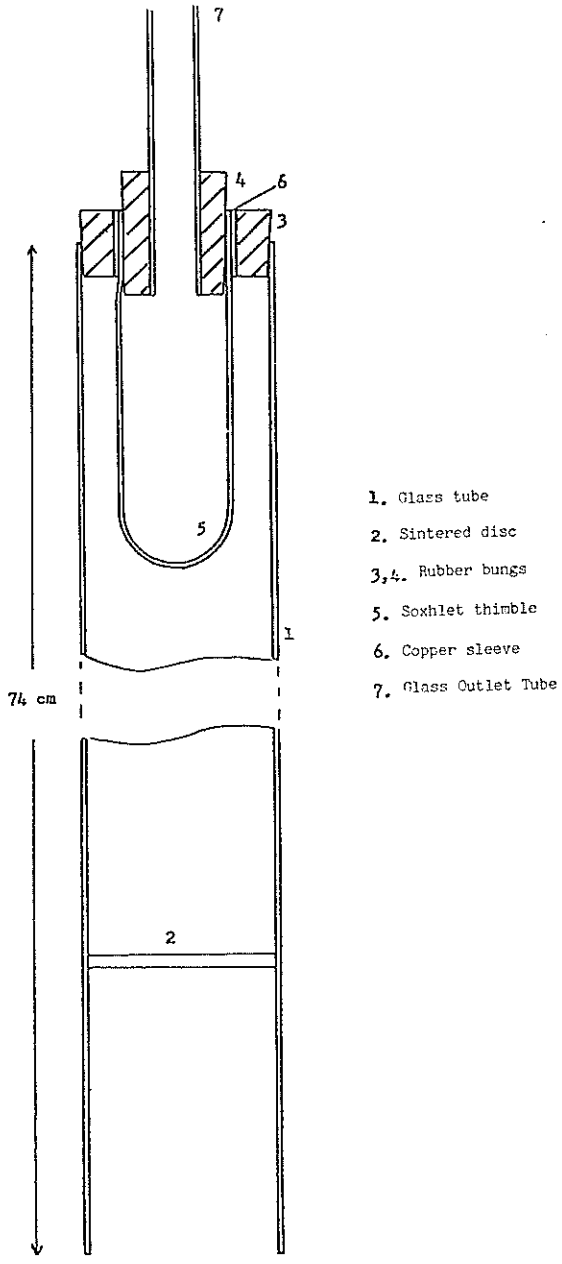



Fig. 18 Dust test apparatus. (Courtesy of Cookson Group plc, Greenford, Middlesex; formerly Lead Industries Group Ltd.)

Another fluidised bed method is used by LEAD INDUSTRIES GROUP LTD (1982). This method (Fig. 18) was developed to test batches of red lead used in glass making and uses a 50g sample placed at the bottom of a glass tube 74cm long and 42mm internal diameter. The sample rests on a sintered metal disc through which compressed air is passed from below to fluidise the bed to carry liberated dust upwards to be collected on a Soxhlet thimble (80mm long and 25mm diameter). The whole assembly contained by the outer bung is preweighed before a test and reweighed

afterwards to determine the weight of dust collected by the thimble. This is expressed as a percentage weight loss (of the test sample) per minute. The test normally lasts one minute during which time the air flow rate is kept constant at 20 l/min, except in the case of very dusty substances when 10 l/min is used. Typical weight losses range from 0.5 to 2%, but can range from 0.1 to 7% in extreme cases.

The British Standards Institution, BSI (1963) have described three methods in BS 3406, Part 3 which while designed for particle size determination are nevertheless relevant to dustiness estimation. Method E2 uses a vertical elutriator in which liberated dust is collected on a thimble filter. The sample is contained in a U-shaped reservoir at the base of the glass tube. Four different sizes of elutriator and 10 injection nozzles give a wide particle size range capacity.

In the other two methods, E1 and E3, a vertical elutriator is again used, but the liberated dust is assessed by sample weight loss. Whilst these methods could be used to estimate dustiness they are subject to small sample sizes, the dehydration of the sample (when wetting may be used as a dedusting technique) and insensitivity to a small weight loss of very fine powder which would be significant in relation to the dustiness potential of the powder.

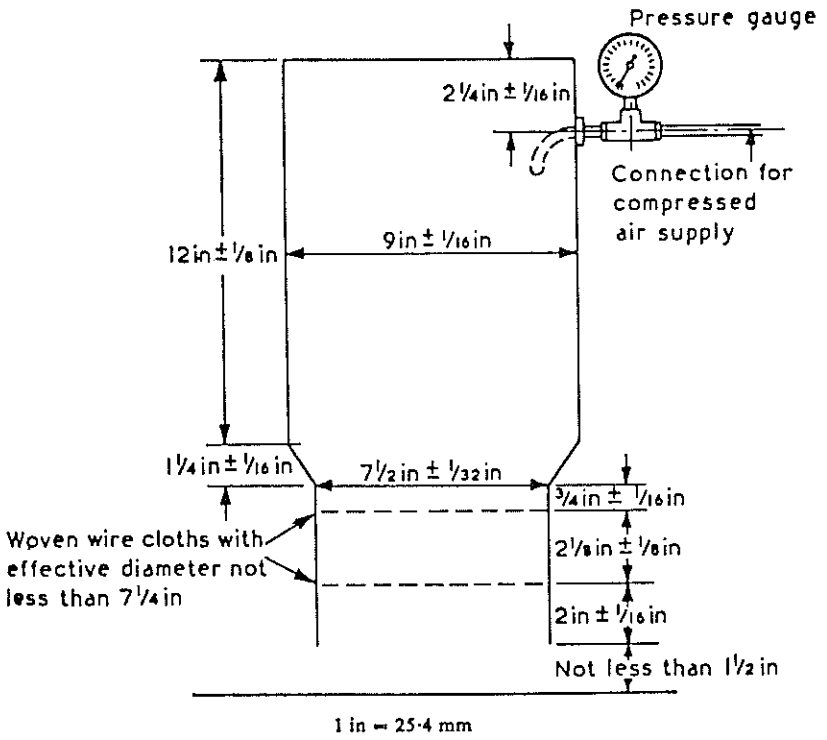



Fig. 19 Apparatus for dust index test. (Reproduced by permission of the British Standards Institution. Copies of the British Standards are available from BSI, Linford Wood, Milton Keynes, UK)

Finally, a rather novel but simple method which was developed for use on mattress filling materials such as flock, jute, kapok, vegetable fibre and man made fibre is described by BSI (1967) as a Dust Index Test (Method 1 - Dry Method). The dust index apparatus (Fig 19) is

cylindrical with a removable top cover which includes a viewing window.

Near the bottom of the apparatus and filling the whole of its area are two woven wire cloths one above the other, the upper cloth being of No 100 mesh and the lower cloth being of No 170 mesh. The wire cloths are contained within a removable cylinder. The joints between all parts when assembled are airtight. A weighed test sample of either 25 or 50g as specified in the Standard is carefully spread out on top of the upper wire mesh and the lid is replaced. Compressed air is injected at a pressure of 15 $lbf/in^2$ (1.06 $kgf/cm^2$) for a period of 60 s. The material which passes through the upper mesh and is retained by the lower mesh is collected and weighed. The dust index of the tested material is expressed as the percentage of collected material relative to the initial sample weight.

## CONCLUSIONS

A large variety of dustiness estimation methods are described in the literature and these have been reviewed and categorized according to whether gravity, mechanical or gas dispersion techniques are employed. The Working Group have decided to eliminate gas dispersion methods as impracticable in relation to a standardization method for general application. There are three main reasons for this decision: i) gas dispersion is not generally representative of materials handling methods used in industry; ii) conditions for efficient gas dispersion vary considerably according to bulk density, form and the particle size distribution of the material; and iii) gas dispersion does not lend itself readily to testing dust reduced chemical forms in which the reduction is achieved by the use of wetting agents.

The remaining gravity and mechanical dispersion methods offer the basis for potential standard methods of dustiness estimation. It is unlikely that any single standard method would be suitable for application to the whole industrial product range for which such tests may be needed. However, a considerable range of product types could be checked by a device capable of handling a sample size of 500g or less. Certain methods of dustiness estimation could be established as laboratory standards and other simpler methods as routine non-laboratory standards. In order to simulate manual operations, a laboratory standard could be developed from a single drop mass determination method, preferably in which the sample is dropped within a chamber (as described by WELLS (1982) referring to a modified ASTM (1975) method) rather than poured into a chamber. For materials handling machinery, a suitable standard could be developed from a rotating drum method which provides multiple drops of the sample (with mass determination by a simple particle size-selective device) as described by WARREN SPRING LABORATORY (1981). For more rapid routine checks, possibly in factory conditions, the single drop light obscuration technique described by SPIVEY (1981) may be suitable. The correlation of such a method with an adopted laboratory method for the materials involved would need to be understood. Calibration of the laboratory method using standard dust samples could be carried out in order to express results against a standardized dustiness index scale. Four dustiness estimation methods have been studied, developed and compared by members of the Working Group. Details of this work and further comments on standardization and calibration are presented in the second part of this Technical Guide.

# PART 2. TOWARDS A STANDARD METHOD

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(A revised form of a paper presented at the BOHS Annual Conference, Edinburgh, 1983 on behalf of the BOHS Technology Committee Working Group on Dustiness Estimation.)

Abstract.

The principles of dustiness estimation by the use of gravity and mechanical dispersion methods are reviewed and an inter-laboratory comparison of four methods is made. Two of these showed high correlation and a third was consistent with these within the experimental error. The fourth method showed some discrepancies which can be attributed to differences in friability. This study suggests that a simple optical method or a gravimetric method could be equally valid for dustiness ranking purposes. Modifications of one of the gravimetric methods to improve its versatility and to reduce the possibility of operator bias are described. This work demonstrated the need for standard materials for calibration. Future work is discussed.

## INTRODUCTION

The first objective of the Working Group was to review the existing techniques for dustiness estimation, together with a review of the benefits accruing from the control of the dustiness of chemicals and details of possible application for industry and industrial research. This task has been completed and the review comprises the first part of this Technical Guide.

The second objective of the Working Group was 'The development of the most practicable and relevant form of estimating dustiness and the development of a dustiness index scale'. This paper is an account of work in progress towards meeting this objective. It comprises a review of the principles of methods for estimating dustiness, an account of an inter-laboratory comparison of four different methods for estimating dustiness, a description of modifications to one of these methods intended to improve its versatility and a discussion of the problem of selecting suitable materials for evaluation of test methods. The paper concludes with an outline of plans for further work towards meeting the second objective and also the final objective which is 'To determine the practical relationship between the measured dustiness and the actual airborne dust concentration which can ensue'.

## THE PRINCIPLES OF DUSTINESS ESTIMATION

### 1. Dust dispersion

Dustiness measurement comprises three operations. First, the dust is dispersed into air. This is frequently done by allowing a weighed amount of the material to fall under standard conditions in a test chamber; further dispersion will occur in most cases when the sample hits the floor of the chamber. The material may be simply dropped or poured once from outside or within the chamber, or it may be tumbled to produce repeated drops as in a rotating drum. In other methods air can be blown at or through a known amount of the material in a chamber or in a tube.

### 2. Particle size selection

The second operation is to collect a sample of the airborne particles. Size selection even if not intentional will nevertheless nearly always occur as it is highly probable that some particles will be dispersed which do not remain airborne for sufficient time to reach the sampling device. Conversely, in some simple techniques in which the sample is dropped onto a plate and the pattern of the scattered dust is examined by visual inspection it is probable that particles of respirable size will either not settle or will be deposited on the walls of the chamber and they will not be considered in the assessment. Thus the dimensions of the apparatus and the dispersion technique become critical in determining what size fraction is collected. Deliberate size selection may be achieved by provision of a horizontal elutriator which allows 'non-respirable' particles to separate before the airborne dust is collected, or by use of a given upward air velocity for sampling dust dispersed in a vertical cylinder which thus becomes a vertical elutriator.

It is appropriate here to note that the presence of fine particles does not necessarily mean that a material will be dusty. Obviously, particle size is a factor in dustiness; if no fine particles are present and

cannot be generated when the material is handled, dust cannot be released. However, fine particles tend to aggregate and van der Waals' and other forces of interparticle attraction may resist the shearing forces in handling which would tend to disrupt the aggregates. These factors vary with the material and the process. It can be seen, therefore, that the particle size distribution of a powder is not on its own a guide to potential dustiness.

### 3. Dust measurement

The final stage is collection and/or assessment of the dust. The most common technique is to collect the airborne dust on a filter which may be weighed or analysed by physical or chemical methods. The airborne dust concentration may also be measured by optical methods. Visual inspection and comparison with standards may suffice for estimating settled dust in some cases, e.g. dyestuffs. Some methods which have been or could be used to collect and measure the dust are listed in Table 4. A method which should be added uses the Coulter counter (SCHUMACHER, 1978) or any similar device which enables the total volume (and hence mass) of the collected particles to be measured. Even a device which indicates particle numbers only may be of value in this work, since although the number distribution is heavily biased towards the smallest particle sizes, which may represent a very small fraction of the total mass, it is these particles that will be carried furthest on air currents and cause more widespread contamination in an industrial environment. MACHADO et al (1983) used a Royco particle counter to count airborne dust particles in three size classes in a simple comparative test of the dustiness of an allergenic medicinal preparation and a dust-reduced form.

### 4. Expression of results

The results may be expressed in absolute units such as the weight of dust collected under specified conditions from a given weight of material tested (mg/100g) or when a rapid result is needed for a quality control check on a production line, a result expressed in arbitrary units will suffice. For example, the dust from a known amount of product may be dispersed in a chamber of specified size and the dust concentration measured by obscuration of a light beam. In this case the reading on a photometer may be used. In some cases a comparative result may suffice, for example the ratio of the dust yield from a sample of the test material to that of a reference sample tested under the same conditions. Thus dustiness index scales that rank powders against subjective assessments, e.g. 'very dusty', 'practically non-dusty', etc. have been devised and found to be useful.

## INTER-LABORATORY COMPARISON OF FOUR METHODS OF ESTIMATING DUSTINESS

### 1. Selection of methods

For general use an essential requirement for a standard test method is that the results should be reproducible in different laboratories and for this to be possible the equipment and procedure needs to be described in sufficient detail. Four methods which could be specified were chosen for detailed study. These methods met certain criteria, namely that the equipment should be relatively simple to construct and use, it should be able to cope with a variety of test materials and that the dust dispersion procedure was relevant to most handling situations. It was decided that although dispersion of dust by an air blast or a

continuous flow of dry air through the sample might be relevant to specific processes it was not of sufficiently general applicability and it imposed many restrictions. This eliminated many of the methods reviewed in the first part of this Technical Guide.

The four methods selected for further study by the Working Party were:

1) The Unilever gravimetric method for measuring respirable dust (WELLS AND ALEXANDER, 1978).
2) The Unilever gravimetric method for measuring 'total' dust (WELLS AND ALEXANDER, 1978).
3) The Monsanto optical method. (SPIVEY, 1981)
4) The Warren Spring Laboratory rotating drum gravimetric method. (WARREN SPRING LABORATORY, 1981)

The apparatus used in the four methods are shown in Figures 3, 8 and 13 and the essential characteristics of the methods are summarised in Table 5. There are two main differences between them. In the Warren Spring method the dust is generated by allowing the material to fall repeatedly in a rotating drum fitted with 'flights', whereas in the other three methods the sample is simply allowed to fall into a test chamber. The second difference is that the Monsanto method uses an optical device for measuring the amount of airborne dust while the other methods use a gravimetric method.

## 2. Outline of test design

The Working Party were not aware of any published comparison between different techniques for dustiness estimation and this investigation was considered to be an essential preliminary to development of a standard method. A number of materials were selected which were readily available and covered a wide range of degrees of dustiness and physical properties. They comprised five 'off the shelf' materials and three rubber additives in dust-reduced form. Samples of the test materials were distributed between the three laboratories concerned and each measured the dustiness by its own technique.

## 3. Results

The results shown in Table 6 are the means of duplicate measurements on each sample. (The results are usually so close that two replicates are sufficient, at least for ranking purposes). It is impossible to compare results directly when the units in which they are expressed are quite different, so the materials were ranked in increasing order of dustiness as shown by each method. These results, shown in Table 7, indicate close agreement between the Unilever 'total' dust rankings and the Monsanto dust index rankings, and also the ranking by the Unilever respirable dust results was consistent with these within the experimental error. Only the ranking by the rotating drum method showed significant discrepancies; these will be discussed in Section 5.

## 4. Correlation between optical and gravimetric methods

These results show that a simple optical method can give results which are consistent with those given by weighing. Thus it may be possible to standardize on two devices. The first would be an optical device, which gives rapid results and which can be used on a production line or in laboratories without accurate weighing facilities and where a quick evaluation is required. The second would be a gravimetric device which

could be used alone and also as a reference method against which the optical device could be calibrated. Alternatively the optical and gravimetric systems could be incorporated into one unit, as is the case with the SIMSLIN respirable dust measuring instrument (BLACKFORD & HARRIS, 1978) now used in British coal mines for research purposes. Clearly, the limited number of results obtained in our test do no more than suggest the possibilities for future development and the method(s) finally chosen must be fully evaluated in order to establish the reproducibility of results given by one instrument and by replicates of the same instrument.

## 5. Anomalous results

After allowing for experimental error there were in fact only two anomalous results, both obtained by use of the rotating drum method. If the materials are ranked in order of increasing dustiness, the Unilever 'total' dust method and the Monsanto dust index method give the same rank order and this is consistent within the experimental error with the rank order given by the third single drop method, the Unilever respirable dust method. It is possible to calculate approximately what the dust yield would have been by the rotating drum method if this had given the same rank order. On this basis the dust yield for sulphur was five times higher than that calculated from the ranking given by the single drop methods. This sample contained many large but relatively friable lumps which probably broke up in the rotating drum giving rise to a more dusty sample. Conversely the oil absorber, which gave a lower dust yield than that calculated from the single drop results, comprised large, very hard pieces of material coated with a thin film of dust. It is probable that this dust was readily removed but the crushing and shearing forces in the rotating drum were not sufficient to generate new dust, so the yield was low relative to the weight of sample tested.

## 6. Friability

The anomalous results from the rotating drum method show that different methods of dust dispersion can give consistent results for homogeneous samples but apparently anomalous results for heterogeneous materials, because the result given by a repeated drop method measures not only of dustiness but also of friability.

Such a finding may be more appropriate to certain processes than the result from a single drop method and it may be of interest that the only commercially marketed device for measuring dustiness of which the Working Group is aware (the Heubach Dust Meter, Heubach, Langelsheim, Germany) is of the rotating drum type. However, there are advantages in measuring friability separately from dustiness and this can be done by use of the Roche Friabilator (SHAFER, WOLLISH & ENGEL, 1956).

## 7. Relative humidity

If the sample is hydrophilic or contains hydrophilic ingredients and is not in equilibrium with air at the relative humidity of the laboratory the result will be affected by the uptake or loss of moisture during the period of measurement.

No attempt was made to control relative humidity in the study described. It might be expected that a continuous dust release procedure such as the rotating drum would be more susceptible to the effects of relative

humidity but recent work suggests that the effect is so rapid that even single drop methods might be vulnerable (HIGMAN, 1983). This factor will be considered in future work.

## DEVELOPMENT OF ONE METHOD OF ESTIMATING DUSTINESS

The problems arising in developing a standard method are illustrated by the results of attempts to improve one method. For example, a valid criticism of the Unilever apparatus is that the sample is dispensed into the test chamber via a funnel. Although this funnel has a short wide stem, few samples will flow through the stem without assistance and the rate varies considerably from one material to another. No evidence is available that the dustiness varies with the feed rate but this factor introduces the possibility of operator bias. Various methods of controlling the feed rate have been tried such as vibratory feeders, but they are an unnecessary complication of what was intended to be a simple technique. The funnel also restricts the use of the test to materials of particle size up to about 10 mm: materials used in heavy manufacturing industries often contain much larger particles. Replacing the funnel with a cylindrical or rectangular hopper having parallel sides was considered but it was decided that the more shallow the layer of material prior to dispersion the less risk there was of compaction.

A scaled-down version of an apparatus described by the American Society for Testing and Materials (ASTM, 1975) for testing coal and coke seemed to overcome these problems. The original apparatus was designed for samples weighing about 23 kg but as samples of less than 1 kg are typical of existing methods and quite suitable for a very wide range of industrial products, a much smaller apparatus could be designed. Also, the original apparatus allowed dust to settle onto a plate (introduced after the material had been dropped). A scaled-down version was constructed and air was drawn from the chamber through a filter mounted in the side of the cabinet near the base. This had the advantage of collecting most of the airborne dust within a reasonable time. The apparatus, which is shown in Figure 20, was constructed first of aluminium and later of Perspex so that the behaviour of the dust could be seen. (The Perspex model was treated with an antistatic fluid and allowed to dry before each use). The optimum sample size was about 200g.

To use the device (shown on the left in Figure 20) the sample is released by withdrawing the slide. Air entering the chamber via the two rows of holes on the left carries airborne dust to the filter housed in the holder shown on the right of the device. The filter holder is connected to a vacuum line via a flow gauge and tap. Non-airborne particles are trapped in a short cylindrical extension of the filter holder.

The Perspex model showed that most of the dust was released when the test sample reached the floor of the chamber and the cloud of dust was ejected towards the top of the chamber, possibly as a consequence of the release of entrained air. It was necessary to remove the sample tray completely otherwise it would trap some of the dust. Removal of the tray also provided an air inlet near the top of the chamber which supplemented the inlets which had been provided opposite the filter holder and thus probably improved the flushing of airborne dust down to the filter.

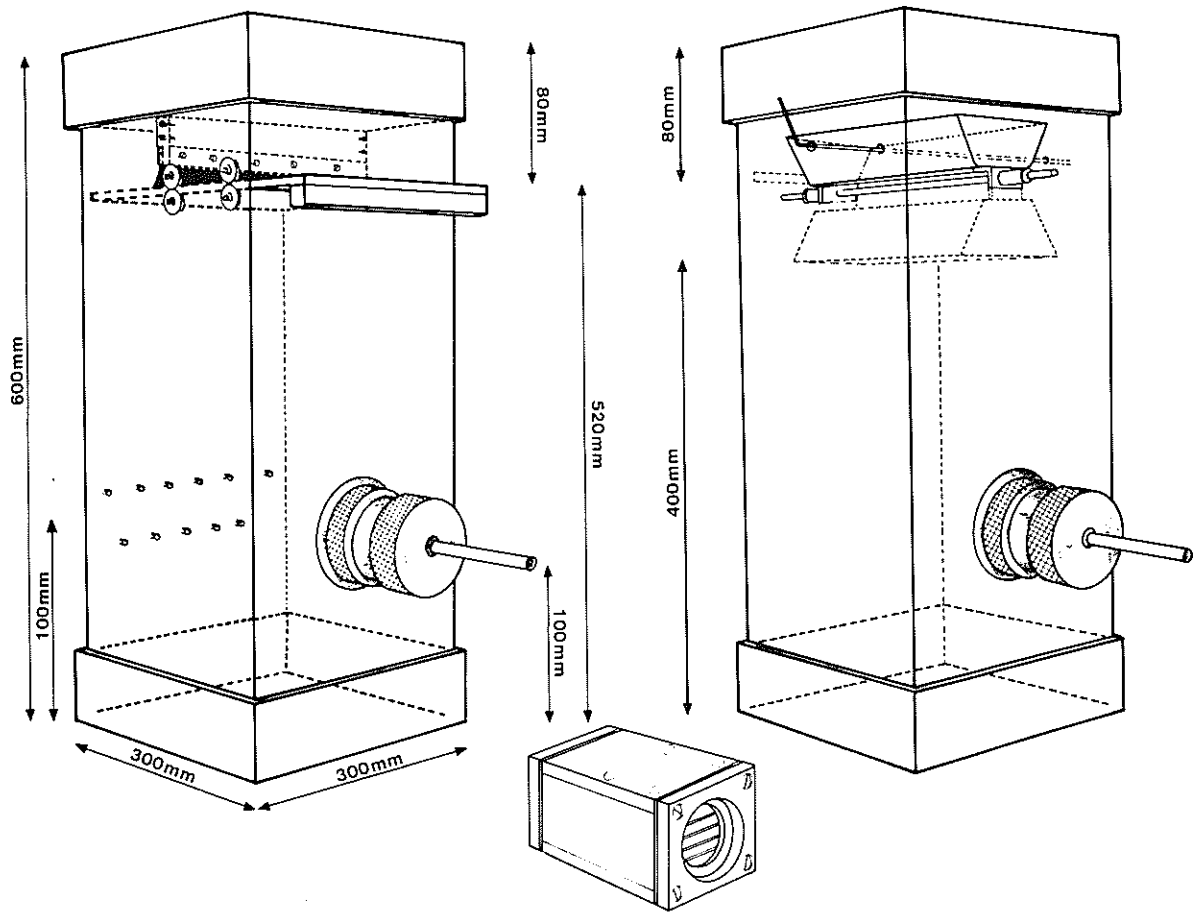


Fig. 20 Prototype devices developed from the A.S.T.M. apparatus for dustiness estimation. See text page 29 for method of use. (Courtesy of Unilever Ltd., Sharnbrook, Bedfordshire)

Several tests have been performed with this apparatus during the course of which it was realised that the withdrawal of the sample tray might itself compress the sample against the wiper and thus again introduce operator variability.

Therefore a second device (shown on the right in Figure 20) was constructed in which the sample is contained in a shallow layer in a hopper which has a fairly large surface area. This hopper pivots on an axis at its base so that when the retaining rod is removed it tips the sample into the chamber. This method should be free from operator variability. The apparatus is not airtight but the aperture now provided near the top of the chamber is large relative to any gaps between the chamber and the lid or the bottom tray, so most of the air entering the chamber should flush airborne dust downwards to the sampling filter.

In this type of instrument there is a much higher probability of large particles reaching the sampling zone than in the three instruments previously described. Indeed, when the filter was mounted close to the internal wall, particles which would not normally have been airborne, were deposited on the lower half of the filter and in the filter holder. Such particles could have bounced off the floor of the chamber or could have been ejected by the release of entrained air on the impact of the material with the floor of the chamber. Therefore a simple horizontal

elutriator was constructed which may be inserted between the filter holder and the sampling port. The elutriator comprises 5 channels 7mm high x 50mm wide x 94mm long. It is fitted with a circular flange at each end, one of which accepts the filter holder, the other fits the sampling port on the chamber. The elutriator was designed to have zero penetration above 20μm at a sampling rate of 10 1/min. This elutriator will permit a study of the effect of restricting the aerodynamic size of the particles collected to that fraction which if inhaled is capable of penetrating below the human larynx i.e. the 'Thoracic' fraction as defined in the 1983 recommendations of the ad hoc Working Group of ISO Committee TC 146 (ISO 1983).

## STANDARD TEST MATERIALS FOR EVALUATION OF METHODS

Many of the problems encountered in this work would be common to the development of the optical and the rotating drum methods. For example, one problem that was foreseen and which is becoming increasingly important as this work progresses is the standardization of the materials used to evaluate the instruments. The variability of results for some powders such as talc suggests that although apparently homogeneous they may be heterogeneous in the distribution of free dust within the sample. In order to determine the precision of the method of measuring dustiness the variation between aliquots of test material must be minimized. At present each sample is prepared by dispersing a weighed amount of very dusty material, sieved charcoal, in a weighed amount of non-dusty carrier, coarse sand. This has itself produced some interesting results but it is a time-consuming operation. A commercial source of readily dispersible dust which promises to provide a more convenient standard is Brown Aloxite (The Electro-Minerals Company Ltd., Manchester) which is available in several different grades with narrow particle size distributions. Work is in progress using this material and the results will be reported in future publications.

## FUTURE DEVELOPMENTS

### 1. Basic studies and selection criteria

Clearly, some basic studies are required before the second objective of the Working Group can be met, ie. the dependence of the dustiness estimate on drop height and sample size for different materials in the single drop methods and the sensitivity of single and repeated drop methods to changes of relative humidity. Issues to be decided that are relevant to industrial needs relate to the various methods of dust dispersion, the desirability of including or excluding friability as a factor in a dustiness estimate and the relative importance of the various criteria to be considered in the selection of a standard method. Consideration needs to be given to the advantages of a cheap and simple device that gives purely empirical results as well as the precision of a more sophisticated instrument designed so that the aerodynamic behaviour of the dust particles can be predicted.

An important question that must be addressed is that of sample size. Many important industrial materials such as coals and iron ores contain very large lumps. In order to obtain a representative sample, several kilograms would have to be taken (e.g. BS 1017:Pt1 (1960)). It will be necessary to determine whether removal of the largest particles by a specified procedure will allow meaningful dustiness tests to be made in a bench-scale apparatus or whether useful results are obtained only if a sample representative of the complete range of particle size in the sample is tested in an apparatus of the size prescribed by the ASTM for coal and coke (ASTM, 1975).

### 2. The relationship between dustiness and dust levels in the workplace

The only objective of the Working Group then remaining before final development of the standard is 'To determine the practical relationship between the measured dustiness and the actual airborne dust concentration which can ensue'. An example of such a relationship has been published (SCHUMACHER, 1978). Laboratory dustiness measurements were used to determine the amount of water to be added to casting sand in order to reduce the airborne dust in a foundry to compliance level: the dustiness measurements were predictive of the workplace concentrations. In order to extend this, a relationship of this kind would have to be established for each material and for defined sampling points in the working environment. However, a dustiness estimate could enable a material to be ranked as, for example, 'very dusty' or 'comparatively dust free' provided a sufficient number of 'bench marks' had been established by testing materials whose behaviour in the workplace was well known by general experience. A recent report by the British Materials Handling Board (BMHB, 1983) classifies several such materials in a similar way and a preliminary study by use of the WSL rotating drum apparatus shows an excellent correlation with the ranking given by this laboratory method (idem). These materials, many of vegetable origin, are unsuitable for use as permanent calibration standards but suitable materials of the type previously described could be matched with these bench marks to provide permanent standards. Such standard materials could be included in the final test standard.

## CONCLUSIONS

1. An inter-laboratory comparison of four methods of dustiness estimation was made. Two of these showed a high correlation and a

third was consistent with these within the experimental error. The fourth method showed some discrepancies which can be attributed to differences in friability of the materials. This study suggests that a simple optical method and a gravimetric method could be equally valid for dustiness ranking purposes.

2. Future work should include studies of the effect of variables such as sample size, drop height, number of drops and relative humidity. There is a need for standard materials for calibration and reference.

3. Issues to be addressed are the relevance to industrial needs of the various methods of dust dispersion, the relationship between dustiness measurements and dust concentrations in the workplace, the relative importance of various design criteria of the methods, and the best procedure for bulk materials which contain large particles.

Table 4. Dustiness Ranking Methods

| Dust Dispersion | Size Classification | Collection/Detection/Measurement |
|---|---|---|
| 1. Dust into air ) Single<br>) Repeated<br>2. Air into dust ) Continuous | 1. (None) | 1. Light obscuration |
| | 2. (Light scattering) | 2. Light scattering |
| | 3. Vertical elutriator | 3. Visual - comparison with standards |
| | 4. Horizontal elutriator | 4. Filter - weighing |
| | 5. Impactor | 5. Filter - extraction & analysis, colorimetry etc. |
| | 6. Cyclone | 6. Impaction - mass measured by piezo-electric sensor |
| | | 7. Impaction - mass measured by $\beta$-particle attenuation |
| | | 8. Weight loss of sample |

Table 5 Characteristics of the four test methods chosen for comparison by the Working Group

| Laboratory in which method was developed | Method of dispersion | Drop height (mm) | Single or multiple drops | Weight of sample | Size selection | Duration of sampling | Method of dust measurement |
|---|---|---|---|---|---|---|---|
| Unilever Research (1) and (2) | Fall into air plus impact with surface | 300 | Single | About 200g | (1)None ('total' dust) (2)Hexhlet (respirable dust) | Duration of sample feed + 5 mins. | Collection of airborne dust on filter, followed by weighing or analysis. |
| Monsanto (3) | Ditto | 700 | Single | 100g | None | 1.6 min. | Obscuration of light beam. Integrated over sampling period and corrected for change in blank reading. |
| Warren Spring (4) | Ditto but in rotating drum | 300 | Multiple | 100g | Andersen cascade impactor | 1 min. | Collection in preseparator and on filter, followed by weighing. (Could be analysed). |

(1) and (2) WELLS and ALEXANDER, 1978
(3) SPIVEY, 1981
(4) WARREN SPRING LABORATORY, 1981

Table 6 Comparison of dust indices of eight materials estimated by various methods

| MATERIAL | UR | UT | MDI | WS |
|---|---|---|---|---|
| | mg/100g | mg/100g | Arbitrary Units | mg/kg.min |
| RUBBER CHEMICAL 'E' | 0.00 | 0.30 | 63 | 0.52 |
| SULPHUR | 0.01 | 0.31 | 328 | 6.40 |
| RUBBER CHEMICAL 'F' | 0.06 | 1.36 | 593 | 1.17 |
| RUBBER CHEMICAL 'G' | 0.55 | 2.01 | 1175 | 4.37 |
| OIL ABSORBER | 0.50 | 2.35 | 1548 | 1.40 |
| CHALK | 0.36 | 9.41 | 2260 | 6.87 |
| SILICA | 4.01 | 14.03 | 2288 | 89.70 |
| CHARCOAL | 13.35 | 63.19 | 4750 | 145.00 |

UR - Unilever, respirable dust, single drop.

UT - Unilever, 'total' dust, single drop.

MDI - Monsanto Dust Index, single drop.

WS - Warren Spring rotating drum method, repeated drops.

Table 7 Ranking of eight materials by dust indices estimated by various methods

| MATERIAL | UR | UT | MDI | WS | COMMENT |
|---|---|---|---|---|---|
| RUBBER CHEMICAL 'E' | 1 | 1 | 1 | 1 | |
| SULPHUR | 2 | 2 | 2 | 5 | 5 times more than expected result. |
| RUBBER CHEMICAL 'F' | 3 | 3 | 3 | 2 | Attrition? |
| RUBBER CHEMICAL 'G' | 6 | 4 | 4 | 4 | |
| OIL ABSORBER | 5 | 5 | 5 | 3 | 1/5 of expected result. Dust depleted? |
| CHALK | 4 | 6 | 6 | 6 | |
| SILICA | 7 | 7 | 7 | 7 | |
| CHARCOAL | 8 | 8 | 8 | 8 | |

UR - Unilever, respirable dust, single drop.

UT - Unilever, 'total' dust, single drop.

MDI - Monsanto Dust Index, single drop.

WS - Warren Spring rotating drum method, repeated drops.

The heavy line encloses rankings which are the same by all the methods: the broken lines exclude rankings which are not the same but for which the differences are small.

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