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APPLICATION OF THE DETERMINATION OF SIZE GRADING IN THE CONTROL OF A GRANULATION PLANT

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The mechanics of granulation processes are too complex for explanation by any single specific theory.

Furthermore, the phenomena are complicated by interaction between the three parts of the operation - granulation, screening, grinding - and experimental results often lack accuracy and precision.

A new method of study of these operations, founded simply on the determination of size grading of the various materials, in proposed here. Presentation of the results in a form similar to that of statistical distributions is an aid to the formation of firm and quantitative views of the phenomena observed in plant operation; weak points may be identified and guidance obtained in respect of the necessary modifications.

Of all the unifying operations of Chemistry, there is one which has the status of a backward child, when it is not ignored completely : granulation. This is not to say that a number of couragous pioneers have not sought to study the process theoretically and experimentally; and various papers have been published and collected on the problem (1 and 4).

Nevertheless, it may be said, in general, that the theories put forward have been insufficiently developed for industrial application, and the basis for granulation plants remains an antiquated empiricism;

- empiricism in their operation, this partly depending on the skill of the operator, his judgement and expertise;
- empiricism in their design, this depending more on existing facilities, prudently extrapolated, than on precise and objective guidelines.

The main obstacle to the development of an understanding of the theory of granulation is the multiplicity of factors involved and the nature of their interaction. The most obvious ones are :

- crystallization of the solution impregnating the solids;
- forces at the interfaces between solids and between solids and liquids.

The effect of each factor acting individually is far from being determined; the problem becomes even more complicated when all the factors act concurrently. The effects become inextricable if the products for granulation are not of uniform simple composition; this being the case for fertilisers, phosphate fertilisers in particular.

GRANULATION SYSTEMS WITH RE-CYCLE

Granulation is rarely a simple linear system; more often it is included within a loop of the process, the end product which is outside specification being recycled to up-stream of the granulation equipment (Figure 1). In this case, the behaviour of the granulator itself cannot be evaluated in a simple fashion, and study of granulation must take into account the mode of operation of other equipment - the screens and mills.

A first requirement for an overall analysis is that the quantities of material making up the one or more, recycle loops should be measured. This is quite easy to do, but there are two practical difficulties :

- the installation cost of weighing equipment;
- the moment to moment fluctuation of material flows.

The use of a sampling method is of some assistance with the first difficulty but, to overcome the second, such a technique is often inapplicable because of the necessary increase in sample size to take account of the scale of the plant and the required precision. In point of fact, plant managers can obtain accurate information in respect of only two or three of the main flows;

- the rate of production, which is equal to the quantity of inputs less losses;
- the rate of recycle, when it is weighed separately.

When weighing is not carried out, the estimates made are often incorrect, both in respect of the average value and of the variations. The inaccuracy is even greater when an attempt is made to evaluate the recycled material in terms of fines and ground oversize.

As well as direct and indirect measures of material flows which serve for the establishment of an overall materials balance, the collection of other information referring to partial materials balances and the thermal balance might be considered. Since, however, the chemical composition and the temperature of the different flows are usually the same, such data is of no relevance.

On the other hand, the determination of granule size ranges is of great interest. Certainly, it does not provide an operational explanation of the equipment in service, but it does provide a limited amount of insight at the cost of some simple and cheap experimental work which does not interfere with the operation of the plant. This paper deals with the method which may be used and its application.

INTERPRETATION OF GRANULE SIZE RANGES

Choice of size

The representative granule size is an arbitrary choice. It might be held that the real value for each class is the most significant size; however, an actual enumeration of the particles retained on the mesh of a screen is too painstaking an operation for systematic use. Indirect enumeration, carried out by dividing the mass of a class by the "average" for particles of that class, is imprecise; the determination of an "average" volumetric mass and of an "average" volume is a rather risky procedure. Thus, it is preferable to use the classical ponderal fraction to represent the division of the particles into granulometric classes.

Presentation of results

The method chosen for presentation of the results of a granulometric analysis is of great importance if the maximum amount of information is to be obtained. A lack of impact is the drawback of tables showing:"class limits - partial or cumulative quantities included within classes". The curves which correspond with these tables do not suffer from this defect, but it is necessary to establish about six points before they can be drawn. A specially ruled paper simplifies the problem considerably. 'Probability' paper may be used which has, in abscissa, a linear scale and, in ordinate, cumulative frequencies after the law of 'normal' statistical probability, or Gauss's Law (5). The limits of the scale (theoretically infinite) are, for example, 0.5 and 99.5%; i.e. with a sample of 100 gr. one leaves out of account 5 decigrammes of the finest dust and 5 decigrammes of the heaviest particles - the weight of about two granules.

Justification

On this paper, a sample of which the frequency distribution (fractions by weight) between granulometric classes follows a normal law, is represented by a straight line which is increasingly inclined in relation to the X axis as the range of granule sizes of the sample is diminished. The substitution of a logarithmic scale for a linear scale along the abscissa, often recommended in similar cases (6), is not justified because of the limited range which is of interest (0.5 - 5.0 mm approximately).

Basic data

The principal advantage of this method of representing granulometric distribution is to facilitate the interpolation of less accurate results than with the customary curves. It is even possible, if all the distributions are held to be normal distributions, to reduce the number of observations to two per sample (two points to draw a straight line.)

This means, of course, that the value of the sample is lost and that the very characteristics which it is desired to observe are concealed. Three points are sufficient to test the normality of the curve - if they are not in a straight line, it is not a normal curve - but are insufficient to define the true line. Four points give a better indication, and five suffice to bring forth all that the curve has to tell.

The normal distribution (see Figure 2)

Assume that on sample "a", normally distributed, two screenings are carried out to remove all granule size classes except d_1 d_2 ($d_1 < d_2$). It is assumed that the screening is perfect, that is to say that screen (d_1) allows no granules of smaller diameter than d_1 to pass and that screen (d_2) removes all granules greater than d_2. The distribution of the sample which remains will take the form "b", limited by the lines d_1 and d_2 parallel to the abscissa.

Applied industrially, the results obtained will fall short of perfection : within the middle class will be found fines which have not been removed, and over-sized granules which, because of thair attenuated or flattened shapes were able to pass through the rectangular mesh of the screen. If the mesh is deformed or has holes, oversized spherical granules may also be found.

Analysed in the laboratory, such a sample would give the distribution "c" which approaches the theoretical distribution "b" only in its central part. An imperfection of the screening would have the effect of spreading the distribution, giving it a shape closer to that of a normal distribution : the linearity would be well defined with a narrow size range ($d_2 - d_1 = 2$ mm, for example) but even with a wide size range, there would be a good approximation.

By way of illustration, a comparison has been made of the distribution of a sample of granules of particularly wide specification (2.0 - 4.5. mm) with a normal distribution having the same modal value and similar dispersion (3.14 - 0.82 mm). The frequencies are shown in the table below. Without going into the details of tests of statistical significance, it is immediately apparent that there is satisfactory agreement, even in this extreme case of a wide range of granule sizes.

Class limits		Frequency	
Upper	Lower	Observed	Calculated
6	5	24	11
5	4	87	133
4	3.15	329	352
3.15	2.5	352	289
2.5	2.0	161	134
2.0	1.5	42	59
1.5	1.0	4	17
1.0	0.5	0	4
Total		999	999

Superposition of normal distributions (Figure 3)

Other distribution types may be observed, and they may sometimes be capable of interpretation. The most simple example is a doubly inflected line with a horizontal section. This distribution is found whenever two distinct processes form part of a material flow. A typical case is the flow of recycled material up-stream of the granulator, when inadequate grinding produces large fragments of too plastic a material, the fragments being mixed with fines from the screens having a much smaller granule size.

Abnormal distributions

In addition to the two types described above, there may also occur doubly inflected lines with an inclined portion in the centre, and lines with a single inflection. Neither of these are capable of explanation by the superimposition of a normal curve. Thus, it is only possible to make such statements as, for example, : the granulator is producing "too much fines". The determination of size grading is no more than an experimental method; clearly, it does not indicate how to remedy this problem of too much undersized material. On the other hand, if the plant manager takes steps to find a remedy, it will tell him with precision after each attempt what improvement he has achieved.

In this category of abnormal distribution, as in the two preceding cases, the interpretation of the shape of the distribution has only a limited application. While it may provide an insight into the qualitative aspects of the materials analysed, and perhaps give an understanding of their origin, the study of distributions is unable to provide a quantitative assessment of the phenomena.

Over and above this imprecise but, in itself, significant benefit, the study of distributions has been touched on here in justification of its use as a method of representing analytical data. The visual clarity of the probability chart and the accuracy of linear interpolation thus obtained, justifies the use of the method throughout the rest of this paper.

DETERMINATION OF MATERIAL FLOWS

The most readily available basic information is normally the production figure. Even if this is not measured directly at the outlet of the plant, the plant manager can obtain a reasonably accurate estimate from the consumption of raw materials, the measurement of rates of input avoiding the need for successive stock-taking. The production figure will, in general, be reasonably constant and always more constant than the other values of the "looped" system. Thus, these values are usually quoted in relation to the rate of production; the word "flows" is used to describe these variables.

In the first looped system (Figure I) for example, there is :

- the production flow, by definition equal to 1 (one);
- the recycle flow, varying between 0 and infinity; this is the sum of the two terms:
    - the flow of oversize (B) passing to the mill;
    - the flow of fines (F) returning directly to the head of the granulator.

In the two other systems shown in Figure I, the recycled material is re-introduced at two different points, and B and F remain separate quantities. In all cases, establishing B and F, gives a complete picture of the flows at the various points.

Two size limits must be chosen which will give 3 granule size classes for each sample. The commercial specification (2 - 4 mm, 6 - 14 Tyler mesh, etc.) is most often used, but is not the best. In practice, these limits do not always correspond with the standard laboratory sieve sizes which are used for analytical purposes. Thus, the values for industrial screens must in this case, be determined by interpolation on to the curves, which affects accuracy. It is preferable to choose the two limits from the sieve sizes which are actually used in analysis, for example the AFNOR 32-36 mesh, the 6-14 Tyler mesh, etc.

The following table is then produced:

Flow	Definition of material	Fraction in the upper class	Fraction in the middle class	Fraction in the lower class	Total
T	" Ungraded product" before screening	t1	t2	t3	1
B	Oversize for grinding	b1	b2	(b3)	1
I	Product size	g1	g2	g3	1
F	Fines	(f1)	f2	f3	1

Partial balances are then established on the reasonable assumption that the screening is efficient.

$$Tt1 - Bb1 - Ff1 = g1$$

$$Tt2 - Bb2 - Ff2 = g2$$

$$Tt3 - Bb3 - Ff3 = g3$$

In each case the solution of the linear system is simplified by the relationship between the coefficients (the horizontal totals are in each case equal to 1). It is further simplified if, by a judicious choice of classes, b3 and f1 can be eliminated, and sometimes even f2, b2, g1 or g3.

It should be emphasized that the flow values of the plant (B,F,T) thus determinated, are independent of the class limits which have been fixed - this is clearly desirable. However, accuracy is lost if the choice of limits is such as to produce a class so small that one of the equations relates to an insufficient quantity of material, so small a quantity as to be affected by the sampling error.

The establishment of B and F, the recycle fractions, or of the total recycle B + F = R, is not an end in itself : when the plant manager has observed that this value varies between 3 and 5, for example - often through a wider area than he had supposed - he may put the information to other uses, perhaps to judge the efficiency of the screening and grinding. In the same way, an analysis of the recycle may be made, revealing the unproductive fractions.

SCREEN OUTPUT

Definition

The causes of inefficient screening in industrial practice have been mentioned above :

- a deformed mesh allows oversized granules of all shapes to pass;
- the rectangular mesh allows flattened and attenuated granules to pass;
- some undersized granules may not be able to pass if the screen is over-loaded or the mesh is clogged.

It is therefore useful to define for each screening operation two criteria of its efficiency:

- the quantity retained R or the fraction of the over-size in the input material which does not pass the mesh;
- the quantity passing P, or the fraction of the under-size in the input material which passes the mesh.

Causes of variation

These values are far from being constant; they depend on at least 5 factors:

1. the operating characteristics of the screen affected by the vibratory regime in terms of amplitude and frequency;
2. the state of the mesh, the spacing and regularity of the holes, the type of wire;
3. the moment to moment load on the screen - overloading increases retention and reduces the quantity passing;
4. the shape of the granules - the particular behaviour of non-spherical granules has already been described;
5. the respective granule size ranges of the flow for classification and the flow of product.

The first two factors (the condition and method of operation of the screens) are not relevant to this paper: it is, for example, clear that 1.9 mm mesh, deformed by the screening of superphosphate, cannot be treated in terms of its theoretical dimension but rather of its actual dimension - 1.5, 1.3 or even 1 mm.

The actual load on the screen, that is to say the flow to the apparatus at a given moment, affects the results. Only the plant constructor can predict the relationship between these values, but it remains our concern to stabilise the flow value. If long-term variations are inevitable, the moment to moment variations may be "damped down" by the installation of suitable equipment (a small hopper or a simple vibrating conveyor of reasonable length).

Granule characteristics do no vary greatly over time; in any cases, it is possible to compensate for the effect of non-sphericity by a small reduction in mesh size.

Choice of mesh

The most important factor remains : the respective dimensions of the granules for classification and of the final product. It should be immediately pointed out that to separate out granules of diameter dt (theoretical diameter) a mesh of aperture dt is not always chosen, but rather one of diameter dr which differs from dt (dr = actual diameter).

It has already been noted that dr will be larger than dt if there is a tendency to clogging of the screen and less than dt if the granules depart from the spherical shape. Thus, a mesh larger than the upper limit (dr > dt) is chosen if the material which does not pass the screen is the end product and must be freed from the fines which it contains. On the other hand, dr dt is chosen if the material passing the screen is the end product and must be completely freed of oversize. In these two cases the quality improvements have the effect of increasing the recycle. In fact, by freeing the end-product of granules which are just outside specification, a number of correctly sized granules which are within specification are also removed.

This can be put in another way. In the first case, for example, (lower screen; material retained on screen being the end product) the aim is to retain the maximum of granules larger than dt and to remove the maximum of granules smaller than dt. Choosing a mesh of diameter dr > dt, all, or almost all the undersized granules (smaller than dt) are removed, which increases the quantity which passes the screen and improves the quality of the product. However, some product-size material also passes - the granules of a size between dt and dr - which lowers the quantity retained (a proportion of the granules larger than dt). On the other hand, in the second case the quantity passing is increased to the detriment of the quantity retained.

Thus, for a given specification, outputs vary depending on the mesh actually chosen for the separation. This is shown diagramatically in Figure IV.

The influence of all these causes of variation on a lower screen (separating the fines from the product) is shown in Figure V. The actual separation by screening is shown by an oblique line, leaving on its right all material which finds its way into the product, including fines (shaded black), and on its left all the material which is recycled as fines, including product - sized material (black edged area). It may be seen that there are four factors which have materially the same effect - to lower the quantity passing - and the same remedy - to increase the size of the mesh :

- clogging of the screen;
- a relatively high content of fines in the material fed to the screens;
- over-loading of the screen;
- screen in bad condition.

A quantitative assessment of the values is now necessary.

<u>The calculation of the values</u>

The foregoing section has demonstrated the effect of the choice of mesh on the quantities passing or retained <u>for a predetermined size group</u>. It has been stated earlier that class definitions are a matter of indifference in the calculation of flows. However, it is only in the evaluation of quantity and quality of production that the calculation of screen outputs is of interest; such a calculation must, therefore, be based on the commercial specification. Thus, in the two sections which follow, the index 1 relates to the granules which are larger than the upper limit of the commercial specification, the index 3 to the granules which are smaller than the lower specification, and the index 2 to the granules which are within specification.

Evaluation of the various qualities on the upper and lower screens allows calculation of the four outputs Rs, Ps, Ri and Pi (Figure VI)

The results are shown in the following table.

	Upper Screen	Lower Screen
Retained	$Rs = \frac{Bb1}{Tt1}$	$Ri = \frac{1 - g3}{T(1-t3)-B(1-b3)}$
Passing	$Ps = \frac{1-g1 + F(1-F1)}{T(1-t1)}$	$Pi = \frac{Ff3}{Tt3 - Bb3}$

In general, at least b_3 and f_1 will be equal to O, which simplifies the expressions to the following :

$$Rs = \frac{Bb1}{Tt1} \qquad Ri = \frac{1 - g3}{T(1-t3)-B}$$

$$Ps = \frac{1 + F - g1}{T(1 - t1)} \qquad Pi = \frac{Ff3}{Tt3}$$

These outputs are in direct relationship with the progress of granulation and allow it to be judged quantitatively and qualitatively.

The quality of the production is bound up with the upper output of quantity retained, Rs, and the lower output of quantity passing, Pi. If, in commercial operation, a proportion Γ of oversize and a proportion φ of fines is accepted in the product, the following inequalities must be satisfied

$$g1 = \frac{Tt1 - Bb1\ (-Ff1)}{1} > \Gamma$$

$$g3 = \frac{Tt3 - Ff3\ (-Bb3)}{1} \quad \varphi$$

which are expressed as a function of the values Rs and Pi (ignoring f1 and b3)

$$Rs > 1 - \frac{\Gamma}{Tt1}$$

$$Pi > 1 - \frac{\varphi}{Tt3}$$

The quantity of production is also related to the outputs which have been defined. The effect of the outputs Ps (passing the upper screen) and Ri (retained by the lower screen) is predominant in this case; however, the two other outputs also play a part and no mathematical expression as simple as that for the quality criteria can be put forward. Such an expression would have to include as indicative values the relative increases $\frac{\Delta T}{T}$ and $\frac{\Delta' T}{T}$ of the flow of ungraded material T as a function of the yields (Ps) and (Ri). It should be remembered that, in the expressions $\Delta T/T$ and $\Delta' T/T$ the predominant terms are respectively :

$$\frac{1 - Ps}{Ps} \quad \text{and} \quad \frac{1 - Ri}{Ri}$$

The other terms are no more than corrections which depend on the tolerance limits Γ and φ of the quality of the graded product. The increases $\Delta T/T$ and $\Delta' T/T$ would affect the theoretical value of T if the two screenings were perfect. On this hypothesis the fraction Tt_2 of T, and this alone, would form the unitary production flow, and one would have :

$$Tt_2 = 1 \text{ or } T = \frac{1}{t_2}$$

Example of application (details in Appendix)

the ungraded material leaving the granulator represents 3.2 times the production (recycle rate of 2.2 : 1). Its granulometric analysis gives 50% oversize, 45% to specification and 5% of undersize and 2% of fines is tolerated within the product. The upper output of quantity retained Rs must satisfy the inequality

$$R_s \geq 1 - \frac{\rho}{Tt1}$$

$$Rs \geq 1 - \frac{0.01}{3.20 \times 0.50} = 99.4\%$$

similarly for the output of material passing the lower screen

$$Pi \geq 1 - \frac{\varphi}{Tt3}$$

$$Pi \geq 1 - \frac{0.02}{3.20 \times 0.05} = 87.5\%$$

Referring to Figure 4, it may quickly be seen that if the first inequality is satisfied it is at the expense of the quantity of material passing the upper screen; that is to say that a good proportion of product-sized material is taken on to the mill. The second inequality gives much more latitude, and allows the quantity retained on the lower screen to take quite a high value; the recycle is therefore kept to a minimum. Complete calculation of the flows also allows the four screening outputs to be obtained :

$R_s = 99.7\%$ $\qquad$ $Ri = 94\%$

$\rho_s = 74.9\%$ $\qquad$ $P_i = 89,6\%$

The theoretical value of the ungraded product is : $\frac{1}{t_2} = \frac{1}{0,45} = 2,22$

The effect of the upper screen is a relative increase of the order of :

$$\frac{\Delta T}{T} = \frac{1 - Ps}{Ps}, \text{ i.e. } \frac{25,1}{74,9} = 33,5\%$$

or an absolute increase close to 0,335 x 2,22= 0,75
The lower screen has a small effect :

$$\frac{\Delta' T}{T} = \frac{1-Ri}{Ri} = \frac{6}{94} = 6.4\% \qquad \Delta' T = 0.14$$

The ungraded product may therefore be broken down as follows :

theoretical value determined by the granulation process 2,22
increase from the upper screen 0,75

- increase from the lower screen	0,14
total by approximate calculation	3,11
actual ungraded product	3,20

Clearly, the recycle may be broken down in the same way:

- theoretical recycle	1,22
- increase from the upper screen	0,75
- increase from the lower screen	0,14
total by approximation	2,11
actual recycle	2,20

Limitations of the analytical method

It has been stated in the previous example that 0.75/2,11, i.e. about 35% of the recycle, is due not to the granulation process but to the inefficiency of the upper screen. The bottle-neck of the system is thus clearly exposed and it may be said that the method of analysis has borne fruit. No more need be asked from the method at the present state of its development.

What, in practice, would the plant manager do, who had followed this investigation through to its final conclusion ? Probably seek to break out of the bottle-neck, improving the screening operation by stabilising the input in time and space, if necessary by changing the screen. At best, he could make an approach to perfect separation, increasing relevant output of material passing, Ps, from 74.9 to 99.5%, the output retained, Rs, remaining at the same figure of 99.7%. By such an improvement, he could achieve a reduction of recycle with the same level of production, (or an increase in production with constant recycle). Would a reduction, of recycle equal to T calculated above, that is to say 0.78, be possible ? The answer can not be specific; it varies from one process to another.

If one considers the actual nature of the recycle, it is known that before the modification 35% was made up of product-size granules, retained on the upper screen, ground and returned to upstream of the granulator. One of two conditions obtain:

- either this portion of the recycle is simply a load on the granulator, or, worse, hinders granulation by its dusty or heterogeneous character (see following section); in this case granulation will be improved after the modification and the beneficial effect will be greater than expected;
- or this portion of the recycle acts as a "base" for subsequent granulation, a base which is indispensible for part or all of the granulation process; in this case the beneficial effect of the improvement will be partially nullified by the absence of these particles.

No attempt will be made here to go more deeply into the question, the answer is too bound up with the particular granulation process in use. For a given process, some answer may be forthcoming but there will still be some uncertainties.

GRINDING EFFICIENCY

Grinding is one of the three operations which make up the granulation system; in contrast to the screening operation, it does not modify the flows in quantitative terms but affects their granule size distribution. One begins, therefore, by establishing the distribution S, representing the ground material, and by calculating B, the corresponding flow.

This section is made up of two paragraphs of different emphasis:

- the use of the method for the development of the process or for modification of the characteristics of the product; this implies a study of the variations of the S distribution (of the ground material) together with the flow B passing to the mill; determination of the influence of grinding on the granulation operation; study of the variations of flow of the recycle R together with the granule size distribution of the ground material.

Production development

The first study consists of examining the evolution of the curve S when the flow B increases. In particular, this permits the establishment of whether the mill is able to handle a greater load, grinding it to the desired fineness, or whether it is working too close to its maximum capacity to permit such an increase. A similar problem is encountered when consideration is given to the production of granules of smaller average size to satisfy a particular requirement of a customer.

As an example, Figure 7A shows various S distributions corresponding to different B flows. The case illustrated is intermediate between the two extremes : when B increases, the distribution S is displaced towards the larger granule sizes although without reaching the maximum. However, the lines sketched in Figure 7B indicate that saturation is not far away.

The first family of curves shows clearly that the modification of granule size is mainly felt in the fraction 1 to 2 mm which declines from 50 to 30% when the flow of ground material increases from 0.28 to 1.01. In some cases, this change is so great that the fraction 1 to 2 mm may be considered to be the active fraction, acting as a base for granulation; agglomerated with the input material, granules within the specified fraction 1.5 to 3.5 mm are obtained. The increase in flow of ground material thus introduces into the head of the granulator a considerable unnecessary load:
$1.01 \times (1.00 - 0.30) = 0.71$
instead of $0.28 \times (1.00 - 0.50) = 0.14$.

With the inf rmation provided by such curves, a modification of the process may be envisaged; from the flow of ground material the fraction which is within specification may be retained instead of being recycled to the granulator (the middle diagram of Figure 1 instead of the upper diagram). Three preconditions must be met:

- the irregular granules must not affect the appearance of the product;
- in spite of their initial size, the granules must attain the required dryness;
- the fines contained in the same flow, returned to the screening stage, must not overload the lower screen.
  Once again, the full exploitation of the analytical results depends on the exact nature of the process and will not be discussed here in any further detail.

Optimisation of actual production

Other information may be gathered at the same time, among other things the effect of the granule size range of the ground material on granulation (characterised for example by the recycle flow).

Some processes tolerate the recycle of very fine material (less than 0.5 mm) while other processes better accomodate, as a base for granulation, a material closer in size to the finished product, that is to say slightly undersized granules. In the second case, a periodic check of the quantity of material which does not pass the lower screen provides valuable information; in such a process, a minimum value of 35% may be ascribed to this material, which means that more than a third of the recycle is reintroduced to the head of thegranulator at product size. When the product is finer (75 to 80% of fines instead of 65%), granulation is affected. It has been stated a posteriori that entry into the granulator the fine particles tend to agglomerate together giving "pralines" or friable "pop-corns" rather than acting as individual nuclei for "onion skin" growth, which is the desirable process.

However, the datum of a single point on the curve, the material which does notpass this or that screen, fails to take into account an important factor - the narrow size range of the ground material. This is a beneficial factor in granulation, although assuming, of course, that the average size has been carefully chosen. This choice may be considerably facilitated by the further development of the analytical method. Thus, it is necessary to follow the evolution of the re-cycle R, characterising the granulation procedure, the distribution of the ground material being more or less dispersed or, with equal width, being displaced towards greater or smaller granule sizes.

The following are two examples of the types of conclusions which may be drawn from this study.

1. Identification of a falling away in granulation quality when a mill departs from optimum operating conditions. This may be due to wear, to faults in the mill itself, or to out-of-adjustment equipment for self-cleaning or grading (scraper blades, grills).

2. The presence of particles which are too fine upsets granulation by the mechanism described above. In this case, if the displacement of the S distribution towards a larger granule size range is impossible because of the danger of producing an excess of over-sized granules, a possible solution may be to maintain the same distribution while removing the finest particle fraction from the flow. This separation may sometimes be achieved at little cost, without introducing a further screening step into the process, by the following techniques:

- by recycling the fines from the screens to the head of the granulator, treating the fines from the mill separately (Figure 1, lower diagram);
- by recycling to the head of the granulator the fines from the screens and the mill, treating separately the fines recovered by the de-dusting equipment (cyclones, filters, gas scrubbers).

In the two cases, the "separate treatment" consists of recycling the material further upstream, to the reaction stage of the manufacturing process, that is to say, introducing it together with the raw materials. When this recycled material again reaches the granulation stage, it no longer has the powdery nature which causes trouble at this stage, and merely forms an addition indistinguishable from the main input. Assimilation is much improved, and the only point to watch is overloading of the reactors. In general, this stage is not a bottle-neck in the production process and is able to absorb a somewhat higher input of material without difficulty.

CONCLUSION

To sum up, this method of study has many possibilities and the user should ask himself to what uses it could be put. To familiarise himself with it, he might use it in its most simple applications - to represent product size ranges in the form of statistical distributions. By this means, quality variations may be identified which are otherwise only suspected until complaints are received from customers. The comparison of size ranges from one product to another is also very instructive.

Subsequently, the determination of size grading may be applied to the different flows in the plant and, carried out regularly, may become the basis for a method of investigation too valuable to be ignored.

If the studies of the use of this analytical method, which have been briefly sketched here, have not been carried further in the context of a general exposition, this is merely a reflection of the fact that each process has its own peculiarities and that the points of interest to be taken into account are many and diverse.

GLOSSARY

b. Granulometric fractions of the flow to the mill.

B. The flow to the mill; ratio of the flow to the mill and the production flow.

d. Mesh size or limit of granule class.

f. Granulometric fractions of the flow from the lower screen (fines).

F. Flow of fines; ratio of the fines from the lower screen and recycle and the production flow.

g. Granulometric fractions of the production flow.

I. One.

r. Index: see below.

R. Recycle flow : ratio of the flow of material recycled to granulation and the production flow.

S. Granulometric distribution of ground material.

t. Granulometric fractions of the flow from granulation (ungraded).

t. Index: see below.

T. Flow of ungraded material : ratio of the flow from granulation and the production flow.

ΔT. (Δ'T) Increase in ungraded product in relation to its theoretical value, due to the inefficiency of the upper screen (lower)

Γ Proportion of oversize admitted in the final product

γ Proportion of undersize admitted in the final product

Indices

r. relative to the actual dimension of the screen mesh,

t relative to the limits of the class chosen, 1 relative to the upper granule class,

2 relative to the middle granule class,

3 relative to the lower granule class,

The limits of classes 1 2 and 3 may be the commercial limits (section on screening) or may be of no consequence (calculation of flows).

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Figure I Granulation systems - three alternatives

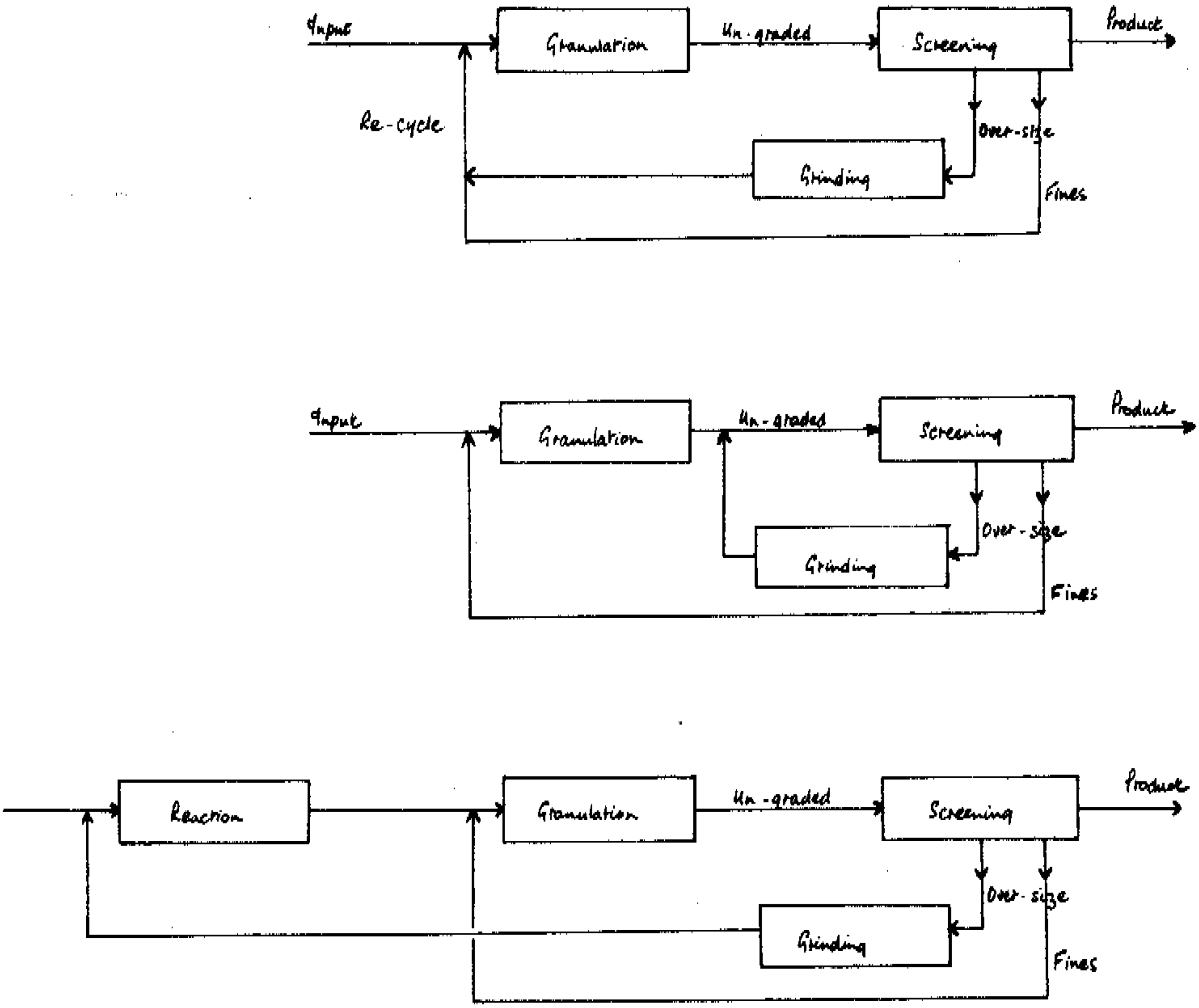


Figure 2 Normal Distribution of Product

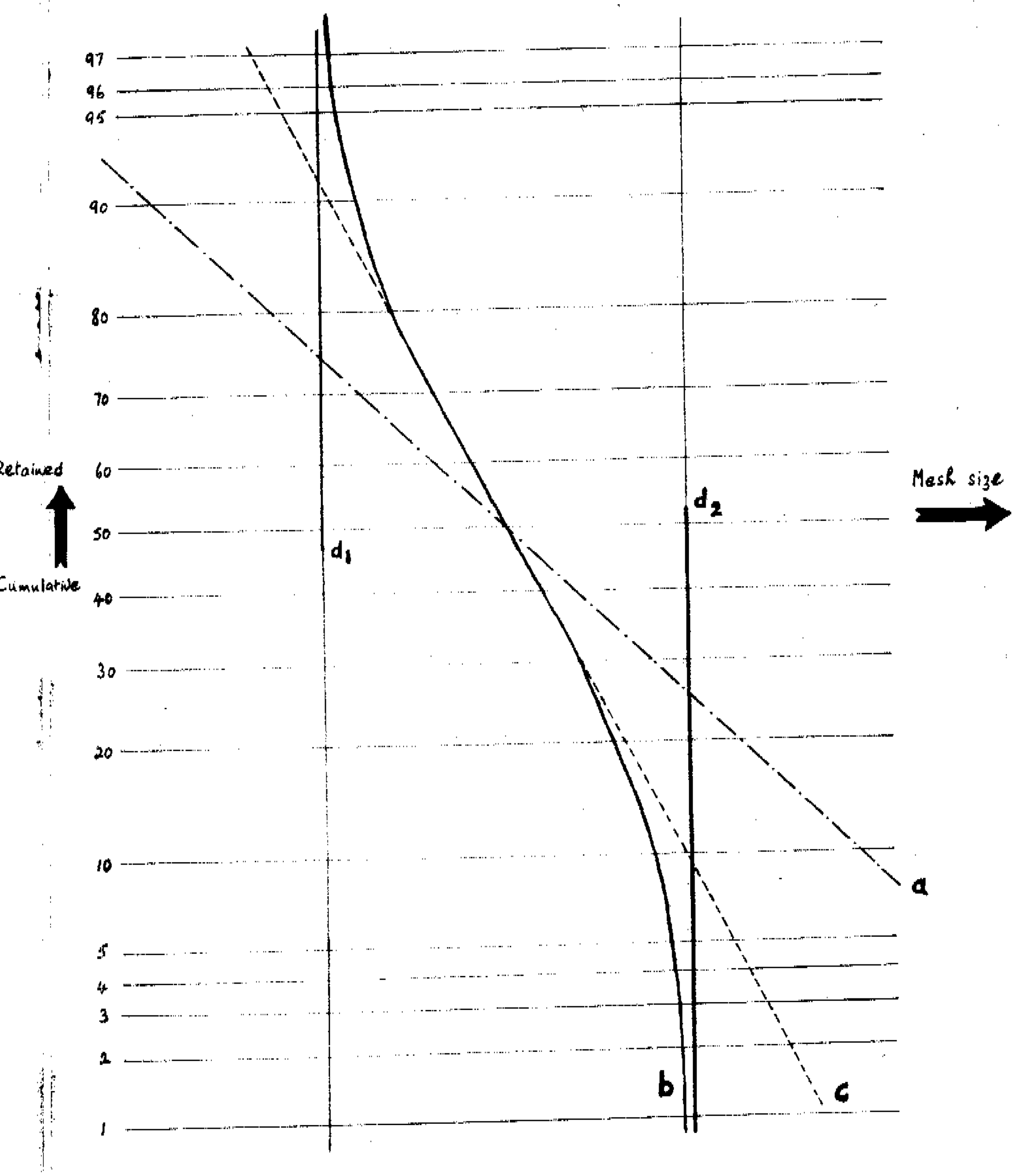


Figure 3 Abnormal Distributions

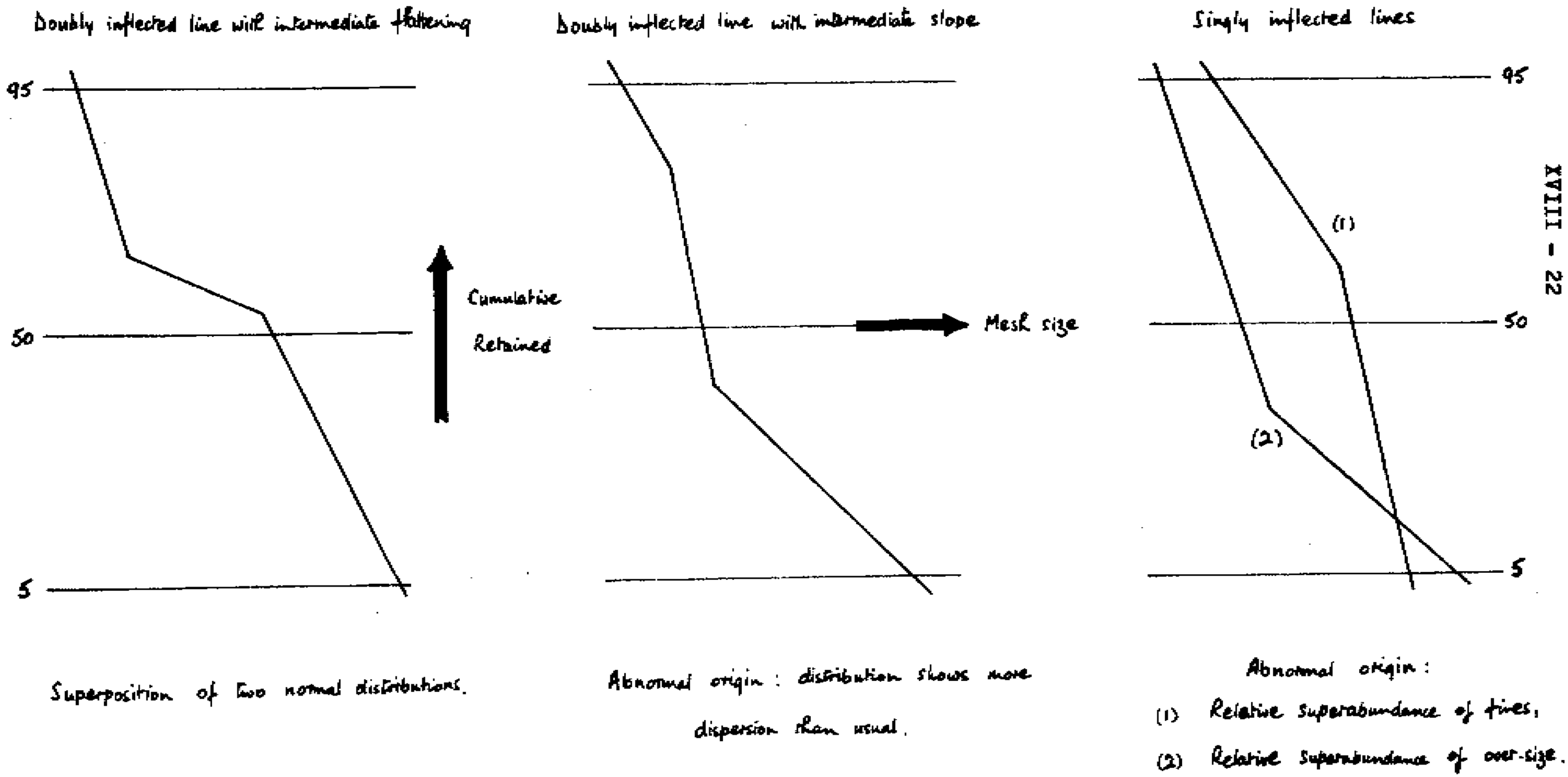
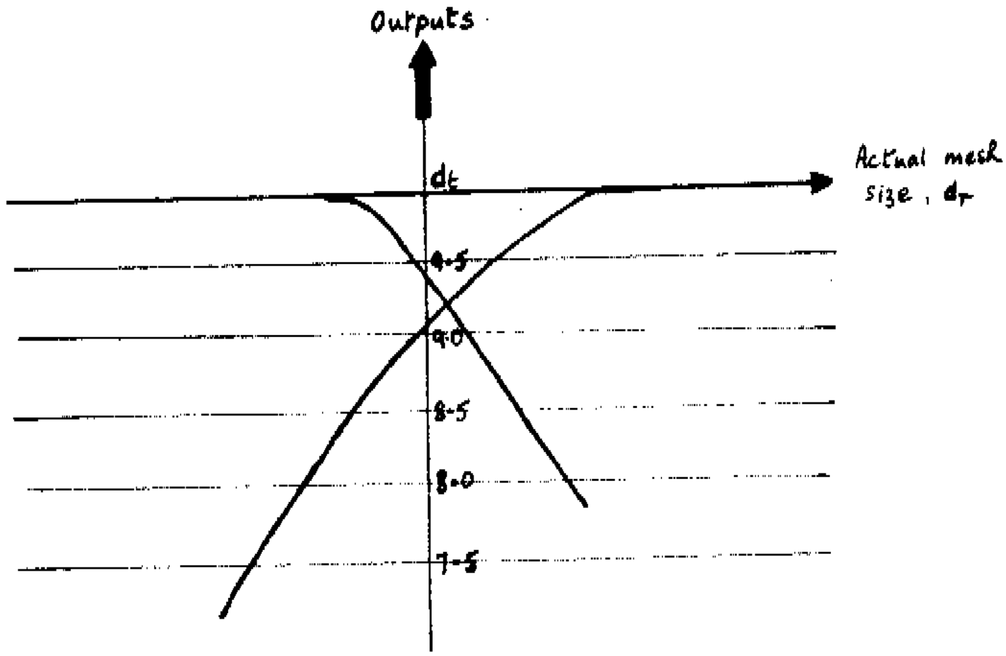


Figure 4 The Influence of Mesh Size on Screen Output



APPENDIX : EXAMPLE OF SCREENING.

Basic data :

	t	b	g	F
1	0.50	0.80	0.008	—
2	0.45	0.20	0.976	0.30
3	0.05	—	0.016	0.70

From these values, the flows and outputs are calculated.

$$\begin{cases} F = 0.205 \\ T = 3.200 \\ B = 1.995 \end{cases} \qquad \begin{cases} R_s = 99.7\,\% \\ P_s = 74.9\,\% \end{cases} \qquad \begin{cases} R_i = 94.0\,\% \\ P_i = 89.6\,\% \end{cases}$$

Figure 5 The Effect at the Lower Screen (removal of fines) of Variations in Input Conditions

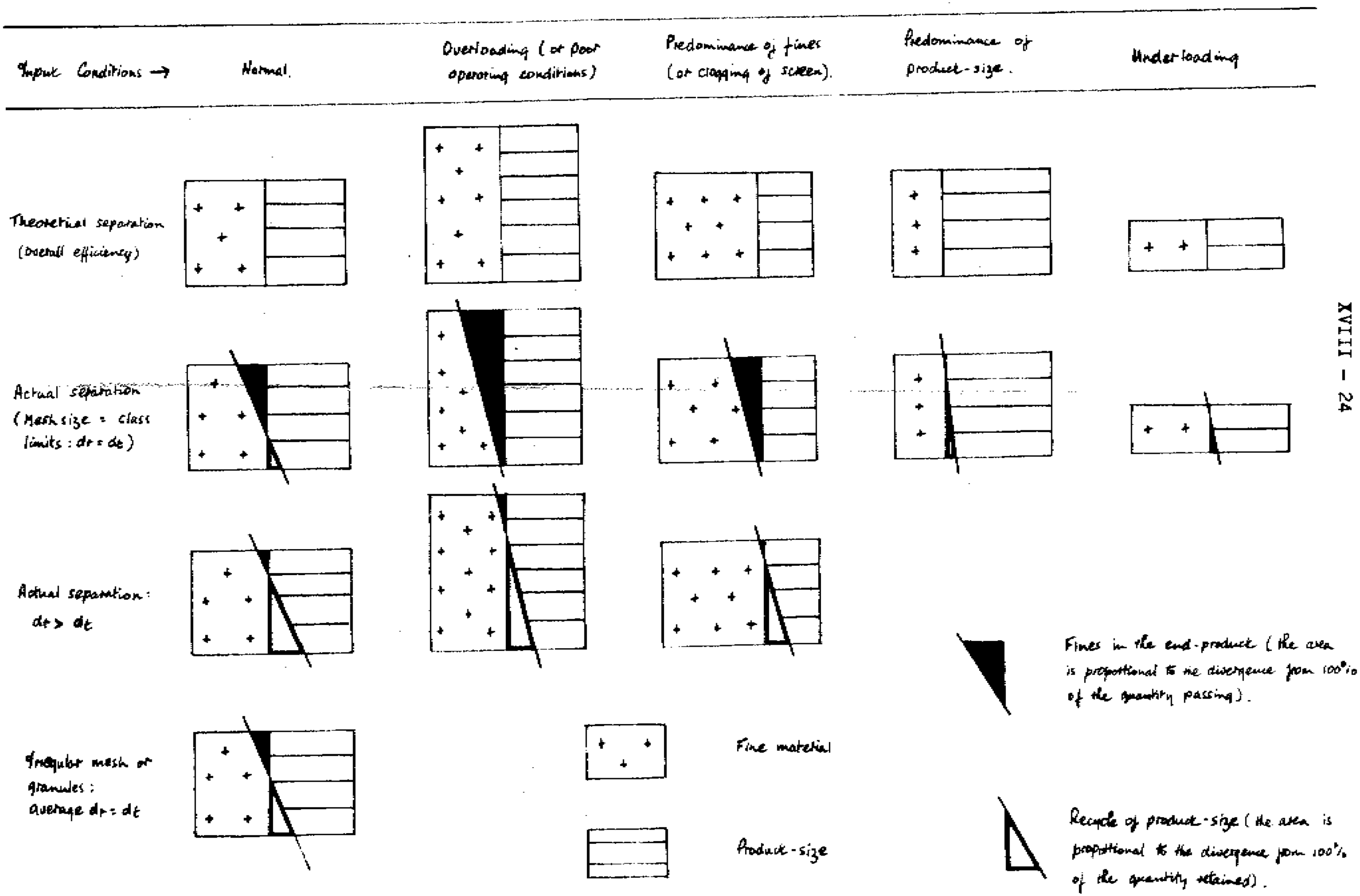


Figure 6 Calculation of Screening Outputs

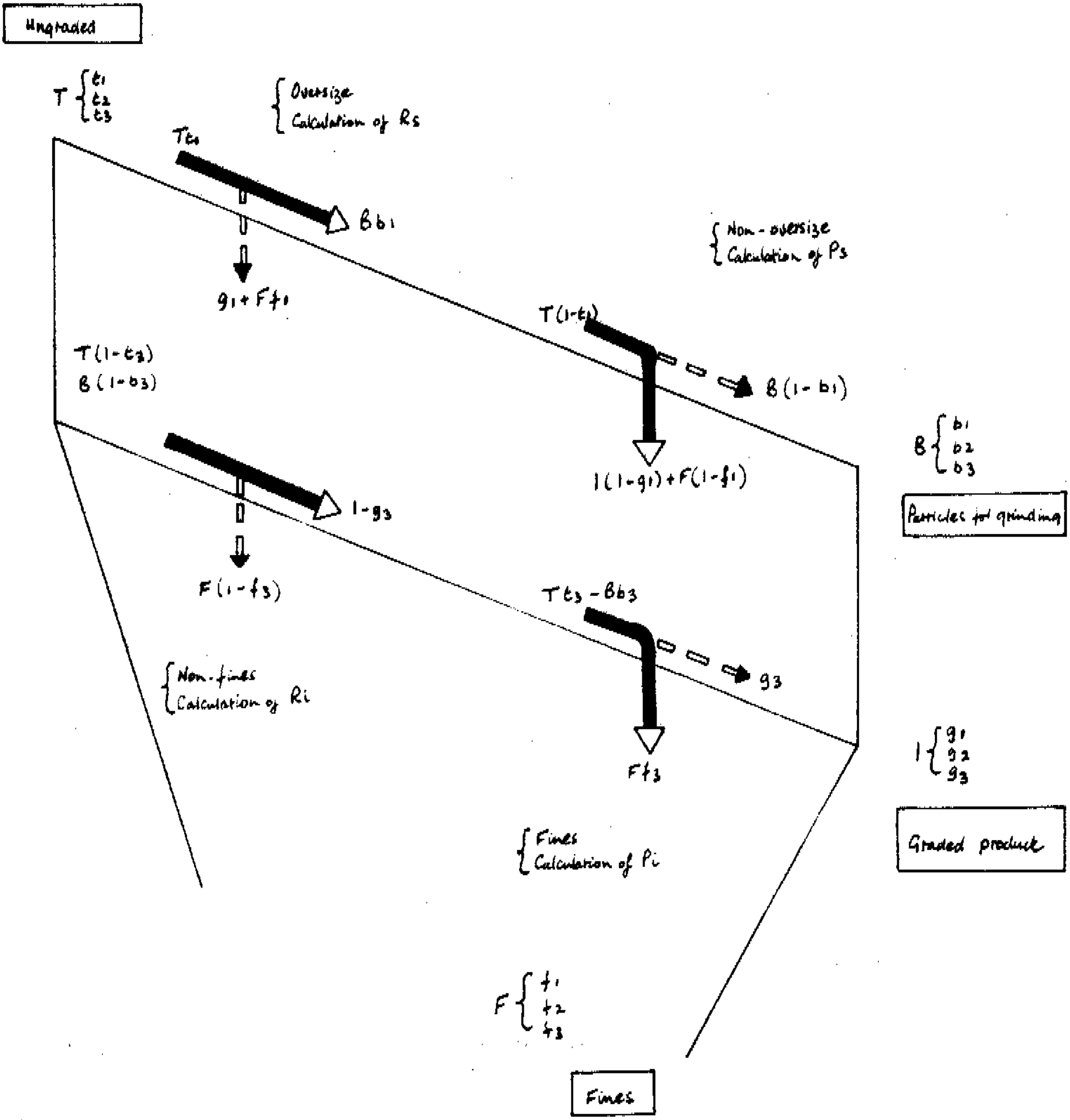
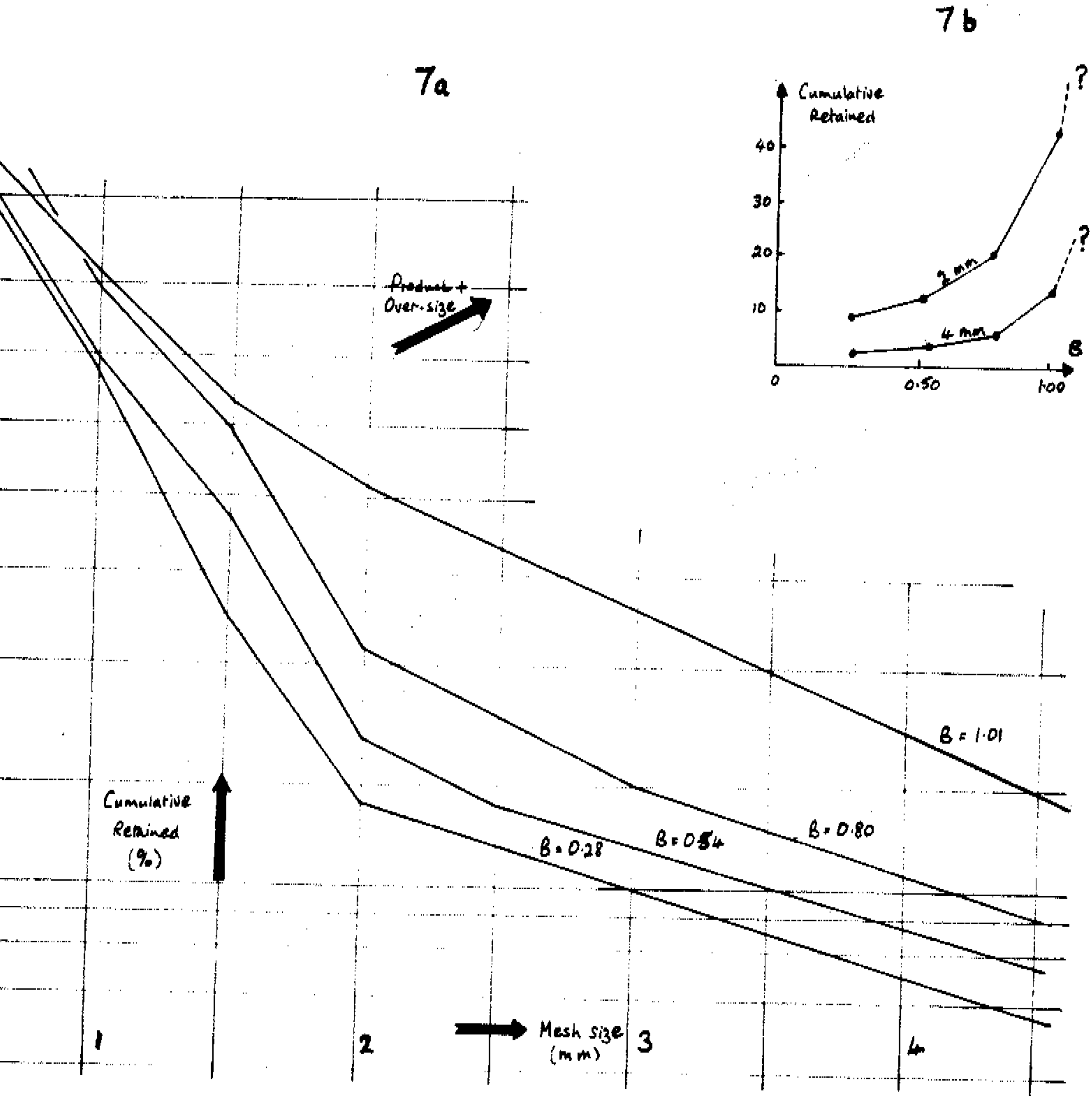


Figure 7 Influence of Input on the Granule Size Range of Ground Material



DISCUSSION

MR. Y. Le MENESTREL (Ugine Kuhlmann - France): Before presenting this paper to you I should like to say that it represents neither a unitary theory nor a fundamental study of granulation. Am I therefore offering you a trick to stabilise the operation of a unit with two operators less? Not yet. On the contrary, if a manager wanted to apply the method in question he would have to start by using additional workers to screen scores of samples. So, what use is it? It is above all a method of investigation or a means of knowing precisely the operation of a granulation chain, i.e. a group of apparatus: the granulator, the screen, and the mill. The demonstration of the weaknesses and limitations of the existing plant constitutes the point of departure for improvements and modifications. It offers the possibility of testing and enumerating variations in operation caused by modifications which should have been carried out on a day to day basis because of the fluctuating feed parameters. For example, one is able to see if the variation of the sulphate ion in the basic acid affects the granulation. In short, it is a tool which is available to the plant manager in the same way as a control apparatus or a chemical production test.

How is this tool constructed? Its "handle" is the graphic representation of granule size ranges on special paper - gausso-arithmetic paper which is usually used to trace normal statistical distributions. Why this curious choice? The first part of the paper shows that many curves or granulometric spectra can give straight lines, particularly the spectra of commercial products. Even for other samples, this kind of paper offers the advantage of affording a linear interpollation between the points corresponding to the various screen sizes of the analysis. The "head" of the tool is the calculation of the balance of the chain, i.e. the determination of all the flow rates of the granulation plant by means of granulometric analysis of these flows. This is expressed as the rate of recycle in relation to the rate of production, e.g. 4 to 1 for the recycle, or 2 to 1 or 3 to 1 for the oversize to be ground.

What is done with all this? In other words, how is this tool handled? Each of the elements in the chain-granulation, screening or grinding - is attacked item by item in an effort to evaluate its efficiency. For example, in the case of a screen, one might conclude that it works well as far as retention is concerned, i.e. it lets only a minimum of oversized granules pass through, but that it lets through an inadequate proportion of undersized granules. Thus, in the case of the upper screen which has to separate the granules to be ground, this would mean that production would not be loaded with granules larger than the normal which is an advantage - but that nevertheless a saleable proportion of granules would in fact be ground and recycled, thus constituting, in general, a loss of production.

In the case of grinding, it is interesting to know if a unit is working close to its maximum capacity or whether it can take a greater load. The practical limit of use is too often considered to be the choking of the mill by overloading, whilst in fact the mill ceases to carry out its function of reducing the size of the particles well before such a stoppage.

Here, then, are two aspects of the possibilities of the method, each capable of being used or developed in the way which is of most interest. Finally, I should like to correct certain errors in the text. On page 7, for "partial balances are then established on the reasonable assumption that the screening is efficient" please read "partial balances are then established on the reasonable assumption that the screening does not affect the size of granules". On page 13, in the middle, the value 0.78 should be 0.75. On figure 4, the curve ascending from the left represents "passing efficiency" and the descending curve represents "retaining efficiency".

MR. I. WENNBERG (A.B. Svenska Salpeterverken, Sweden): It is always a pleasure to read a paper concerning an old process in which someone has tried to bring some order and principles, especially an old process burdened with a lot of empirical ballast.

According to our experience the sieve analysis is a rather poor instrument. We have found that if we put a sieve analysis on a probability chart, now and then there is quite a difference with the same screens, especially if we use plant materials in the screens, and the curve is not a straight line. When they are used, it tends to flatten out at the bottom. Consequently, can you give us some idea about the reproducibility of your screen analysis? Secondly, for the probability chart you suggest that we can use 2 to 5 points to have more indicative and more detailed information about distribution. If we use 2 points, can you give us an idea where to put these points on the probability chart to have the optimum information? I mean, for instance, should we put them at 20 or 80% or 10 or 90% for 2 points, and for 3 points for instance 4, 50, 95, or is it better to put them at 10, 50, 90? My third question concerns Figure No. 1 about the various granulation systems. According to my opinion there is one flow missing in these alternatives: the recycle of a small amount of the product to keep input and output equal. Otherwise the pulsation in the plant can upset the whole system. I would like very much to have your comments about this. Fourthly, on page 13 you conclude that the bottleneck is the upper screen. I interpret the excellent calculation in another way. I find that the upper screen in this system functions as its regulator - a rather poor regulator but still a regulator to keep the input and the output of this system in some balance.

MR. Y. Le MENESTREL: Your first question concerns the reproducibility of the granulometric analysis. I think this reproducibility depends on the origin of the sample. With regard to the internal process flows, the flows comprising the recycle, the reproducibility is certainly doubtful, to say the least. This is why there is no question of constructing a balance on one single set of samples, and in the actual case which I am thinking of, I certainly had to take at least ten samples before obtaining a balance which could give me not only suitable average figures but also an idea of the variation in time of these granule size ranges. However to defend granulometric analysis, I think one can reply that in the case of the production flow, i.e. the flow of commercial product, the reproducibility is much better. I am sorry I don't have figures here, but in our works laboratory we have taken ten samples of a product ready for sale and of a specific granulometric category and the variation of the partial rejection of the particular screen did not exceed 3-4%.

The second question relates to the number of points or, more practically, the choice of screen cuts in order to use the method, and you ask me whether one should place these cuts in order to obtain 10 or 20% rejection and, on the other side, 70 80 and 90%. But I cannot reply like that, because what counts, in fact, is to choose a cut which is the same for each of the different flows. Thus, for example, if you choose a 4mm mesh for the upper screen, the determination of the upper category for the oversize flow, it will retain perhaps 80%, whilst for the flow of fines returning to granulation, the retention will be practically nil. Thus, since one must choose two limits, and only two, what counts is to place them in the centre of not only one flow but all the flows. In practice, the limits which I have chosen are generally 2-4 mm.

Your third and fourth questions are to some extent related to each other. They both concern the value of recycling part of the production to ensure a constant quantity of recycle. This can, of course be done by the upper screen, as you say. However, the value of doing this is not common to all granulation processes. For example, in the case of triple superphosphate, granulated in a drum granulator, we believe that as much of the product fraction as possible should be evacuated and the recycle should be kept as low as possible. But in another process, which we use for granulating diammonium phosphate, we voluntarily recycle a part of the production back to the granulator. Even in this kind of process, I think there is considerable value in measuring the production: do not simply trust to the regulatory action of the upper screen, but use a weigher to control the recycle. Thus we recommend the use of an accurate measurement device rather than a natural, inevitably inadequate action.