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**In 1982, the name of the International Superphosphate Manufacturers' Associations (ISMA) was changed to International Fertilizer Industry Association (IFA).*

GRANULAR COMPOUND FERTILIZERS

Some Investigations into the Control of
Granule Size

INTRODUCTION

As its title indicates, this paper is not intended to be a symposium on granulation. It is an account of some of the experimental work carried out on a small scale experimental batch granulating unit over the last few years, but re-arranged in order to bring out what we now consider to be points of major interest in producing granules of the required size under varying conditions.

An attempt will be made to relate the work on the batch unit with continuous granulation in full-scale tube-type rotary granulators, employing mixtures based on superphosphate, triple superphosphate, sulphate of ammonia, and muriate of potash. The effect of the subsequent drying process will also be referred to.

The experimental batch granulating unit was built for the express purpose of studying granulation in the manner carried out on our full-scale granulating plants, as it was felt that investigational work on all the many variables concerned in granulation was not practicable on production units.

We have since found that characteristics of full-scale continuous units vary widely from the batch units. At first this appeared to render the small-scale experiments of little value, because we could frequently granulate "difficult" mixtures on the batch unit with ease. Investigation into this fact alone has helped to bring out a number of interesting points, and has established the value of the small-scale work.

All the work to be described was on materials which require the addition of water for granulation, and which therefore require subsequent drying to produce a hard granule.

FIRST ESSENTIALS

The aim of all continuous granulating processes is to produce a uniform product, i.e. uniform in size and in chemical constitution. The obvious first step in getting this result is to present the raw materials to the granulator in as uniform a state as possible.

This necessitates -

- (a) Individual raw materials of constant size and quality, e.g. moisture content.
- (b) A uniform mixture of the above raw materials.
- (c) Uniform rates of flow of materials and water.
- (d) Uniform wetting of the materials.

(a) & (b) Raw Materials and Mixing

We find frequently that production works expect to get a good quality product from a mixture which varies in constitution from minute to minute.

In the batch unit, raw materials are prepared by hand, and thoroughly mixed, thus ensuring uniformity.

Furthermore, granulation by addition of water commences immediately the mix is placed in the unit, whereas on full scale, it is frequently found that after mixing, subsequent handling of the materials before they reach the granulator results in some segregation, particularly if the size range of raw materials is wide. Segregation can also occur in the first part of the granulator shell before addition of the water - due to the tumbling action.

(c) Uniform Rates of Flow

With regard to uniformity of rates of flow, the problem does not arise in batch units, particularly small ones, as sufficient water can be added and uniformly distributed, to suit the weight of material in the batch. In a continuous process, water is added at a nominally uniform rate at one or more fixed points, and in order that the material should become uniformly wetted, its rate of passage beneath the sprays should be equally uniform. In a rotating tube granulator, forward motion is the result of speed and inclination of the tube, which are constant, and also on the physical rolling characteristics of the material which can vary widely. Thus, should some part of the material beneath the water sprays become wetted more than adjacent parts, it becomes lumpy and less mobile, and forms a temporary weir at that point, which retards forward motion of new material, and in the process becomes wetter still. This position holds until a head of new material builds up behind the temporary obstruction and finally moves it along.

The subsequent rate of flow of dry material is then temporarily increased until normal conditions are resumed. Meanwhile some material has been overwetted, and some insufficiently wetted, with a consequent production of oversize and fines.

Such conditions as described are frequently found in full-scale continuous plants and may arise through non-uniform mixing of raw materials which have a non-uniform rate of progression through the tube.

(d) Uniform Wetting

Another phenomenon, which arises with very fine raw materials, is "slippage" of the mass, which refuses to be "gripped" or lifted by the shell. The result is a see-saw motion of the mass, up and down the shell, with the constituents of the mass relatively static (see Fig. 1).

Uniform wetting of the materials is most likely to take place in a rotating tube only if the materials are smoothly lifted until they cascade over themselves with a "waterfall" effect, thus presenting a constant change of surface for wetting to occur (see Fig. 3). Once the bed becomes static, or ceases to cascade uniformly, some parts must of necessity become wetter than others, and quality of granulation suffers. Insufficient speed can produce a rolling motion without cascading, as shown in Fig. 2. This effect occurs when the speed is below 30% of critical speed - for our fertilizer materials.

Thus items (c) and (d) are interdependent in a continuous tube granulator.

Correct Rolling of Materials

Our small-scale batch unit, because of the small radius of curvature of the tube, ensures adequate lifting of the materials by centrifugal action, without the need to employ lifters. Fertilizer materials, when wetted, tend to stick in the corners of lifters, and after building up for some time can subsequently fall away in large masses, which promote oversize. Sometimes heavy chains are fitted longitudinally inside the tube to ensure adequate lift, and because of their flexible nature, they prevent any substantial build-up taking place, either on the tube or on themselves.

The larger the tube diameter, the less is the centrifugal force and lifting action for a given speed, and it is reasonable to suppose that a small diameter shell, rotating at high speed and with ample inclination, would be better for the wetting process than a large slowly rotating shell which produces a deep bed of material and makes wetting of individual particles less possible.

We have described above the conditions of rolling or cascading desirable to ensure uniform wetting of the raw materials. The conditions necessary for this part of the process are not necessarily the best for obtaining the desired granule size and shape by agglomeration through rolling, and it is desirable to consider this part of the process separately.

It is perhaps in this respect that the plate or "Teller" type granulator has some advantages. Wetting takes place at the upper part of the plate where movement and cascading of material is relatively free. As the granules start to grow, they roll to the lower portion against the rim, where a close shearing action is imparted without so much free movement between granules.

Having discussed the general requirements for good granulation in a rotating tube, it is now proposed to discuss the effect of individual variables as noted on our small batch plant, and relate these effects to those noted on production units.

DESCRIPTION OF SMALL SCALE BATCH GRANULATING UNIT

Figs. 4, 5 and 6 are photographs of the experimental batch granulation unit.

Fig. 4 shows the granulator tube or drum on its mounting. The drum is 1 ft. (25.4 cm) in diameter and 1 ft. long, and is driven by the supporting rollers via a variable speed gear box, giving a range of speeds from 15 r.p.m. to 50 r.p.m.

Large holes in the conical ends permit of sampling during operation, and give access for insertion of the water spray (operated manually). The conical ends were fitted to the drum after the photograph (Fig. 4) was taken.

Figs. 5 and 6 show general views of the drying equipment and drying drum. Fig. 5 shows the air fan and gas mixing valve leading to the combustion chamber. Fig. 6 shows the hot gas duct from the combustion chamber entering the drying drum, and the feed chute.

Accurate means of measuring the water added per batch is essential, and this is accomplished by a wash-bottle filled with a predetermined amount of water from a burette. Pressure is supplied by a small air pump to force the water through a fine fan-type spray nozzle held on the end of a length of tube, which permits the spray to be uniformly distributed along the material as it rolls in the drum.

For normal materials the spraying occupies $\frac{1}{2}$ minute, and the material is then allowed to roll a further 2 minutes before being dried.

A drum speed of 38 r.p.m. is normally employed, which is 50% of the critical speed. Critical speed is defined as the speed at which material could just be carried completely round the drum by centrifugal action, and its value is $\frac{76.5}{\sqrt{d}}$ r.p.m., where d is the drum diameter in feet.

To dry the granules, the contents of the drum are emptied into a similar drum on a separate mounting. This drum rotates at the same speed as the first, but has a supply of hot air injected into one end. The hot air is supplied under slight pressure from a fan via a gas heated combustion chamber, enabling air temperatures up to 500°C., and volumes up to 350 c.f.m. (10m³/min.) to be obtained.

The drying drum is not fitted with lifters, as the centrifugal action ensures sufficient lift, and provision is made on both mountings, by means of variable spaced supporting rollers, to enable any size of drum up to 30" (75 cms) to be employed.

The normal drying time is 10 minutes, and with a hot air temperature of 450°C., and hot air volume of 100 c.f.m., a final moisture content of around 3% is obtained. External knocking of the drum is required during the early stages of drying to prevent materials sticking to the internal surface.

The apparatus enables a test granulation to be carried out in $\frac{1}{2}$ -hour from the preparation of the raw materials, thus permitting quite a number of comparative tests to be done in a day.

Small-Scale Test Procedure

Our normal test procedure is as follows:-

- (1) Carry out sieve grading on all raw materials.
- (2) Carry out moisture determination on the superphosphate (and/or triple superphosphate (T.S.P.) as applicable).
- (3) Determine the free acidity of the superphosphate.
- (4) Mix batch of raw materials to give standard batch weight of 5 lbs. (2.25 Kg). This gives a 10% cross-sectional area loading.
- (5) Carry out preliminary test granulation, using predetermined volume of water.
- (6) Dry the granular material in the separate drum.
- (7) Carry out sieve grading on product.
- (8) Draw grading curves on special grading chart.

An inspection of the grading curve indicates whether the correct amount of water was used, and repeats of items (4) to (8) are then carried out at different water levels to obtain a family of curves giving product size ranges either side of the optimum required.

Inspection of curves provides the following information:

- (1) The water requirement for optimum quality of granulation.
- (2) The sensitivity of the mixture to water requirements.
- (3) Whether the mixture is capable of giving a good granule.

Value of Small-Scale Tests.

It will be appreciated that this test apparatus is of great value for comparative tests, and whilst it has already been stated that good granules from normal fertilizer mixtures can invariably be made on it, it is directly of use for assessing the granulation characteristics of new formulations, and to show what difficulties are likely to be experienced with them on full-scale production on existing plants.

List of Experiments to be Described.

The remaining part of this paper describes some of the comparative tests we have carried out to determine the effect on granulation and granule size of -

- (a) Speed of rotation of drum
- (b) Growth of granules in drier
- (c) Time of retention in the granulator.
- (d) Water requirements of different mixtures.
- (e) Particle size of Sulphate of Ammonia
- (f) Fines addition to raw materials
- (g) Sprays versus jets for water addition.
- (h) Positioning of water sprays.

It is appreciated that Continental manufacturers frequently granulate with superphosphate straight from the den, and that under these conditions it may be unnecessary to add water. Also, owing to the high temperature of the superphosphate, subsequent drying may be unnecessary.

In the United Kingdom, compounds based on superphosphate are sold on their water-soluble P_2O_5 content. It is thus necessary to mature the superphosphate for at least 2 weeks before employing it for granulation, by which time it is cold and less plastic. The maturing ensures attainment of maximum water solubility, and this is not affected by subsequent granulation and drying, unless very high temperatures are employed for the drying.

ESTIMATION OF GRANULE QUALITY IN RESPECT OF SIZE GRADING.

Before describing the various tests, it is first necessary to refer to the special grading chart we employ in order to assess the quality of granular product. (1)

The chart is shown on Fig.7. It will be noted that the abscissa is graduated in log. size dimensions, in units of inches, millimetres and equivalent B.S.S. mesh.

The right-hand ordinate represents cumulative percentage of material retained on a given sieve, to a linear scale, and is the reverse of the left-hand ordinate. Heavy vertical lines are shown drawn through the 1.5 mm and 4.0 mm divisions, and this represents the normal granule size range to which we work.

This chart will for convenience, be referred to as the Fisons grading chart, and Fig. 8 illustrates the curves obtained by plotting the size gradings of good and poor quality granular products on it.

Other methods of plotting size gradings are well known, and a typical log-probability chart is shown in Fig. 9. It will be seen that this accentuates unduly the end sizes at the expense of the mean size, but requires statistical interpretations.

Another method is to plot the percentage of product within a narrow size range against the mean of that size range, producing a curve of the form shown in Fig. 10. This method accentuates well the mean particle size, but is not so useful for comparative work.

The two products illustrated in Figs. 8, 9 and 10 are the same in each case.

Method of Use of Fison Grading Chart

The two curves on Fig. 8 illustrate typical products as made in the drier before screening. All curves shown in this paper represent material ex drier, unless otherwise stated.

Curve 1 represents a good quality product in which 100% (by weight) lies between 1.5 mm and 4.0 mm sizes. This is the size range acceptable in the U.K., and it is also the usual range in which the maximum percentage of product size occurs with our typical raw materials.

Curve 2 illustrates a poor quality product such as may result from the use of a fine particle-size sulphate of ammonia or potash. Here only 4.3% of material ex drier is within the required size range, and such a material would involve recirculation of 37% of fines and 20% of oversize, relative to total drier throughput.

We feel that the main feature of the Fison grading chart shown in Figs. 7 and 8 is the readiness with which closeness of size range is shown by the slope of the centre portion of the curve towards the vertical. Perfect spheres, of uniform size, would be shown by a vertical line. Curves which are roughly parallel, i.e. displaced horizontally, represent geometrically similar products, and this assists in making comparative estimations of products from test runs.

COMPARATIVE EXPERIMENTS ON BATCH UNIT

(a) Speed of Rotation of Drum

Table I has been prepared to show the relative critical speeds of tubes or drums of various diameters from 1 ft. (30.5 cm) to 7 ft. (213.5 cm).

TABLE I

Col. I Diameter	Col. II Critical Speed = $\frac{76.5}{\sqrt{d}}$ r.p.m.	Col. III 50% of Critical Speed. r.p.m.	Col. IV R.P.M. at 120 ft./min. Peripheral Speeds. r.p.m.	Col. V % of Critical Speed when running at 120 ft./min. Peripheral Speed.
1 ft. = 30.5 cms.	76	38	38	50
2 ft. = 61 cms.	54	27	19	35
3 ft. = 91.5 cms.	44	22	12.7	29
4 ft. = 122 cms.	38	19	9.5	25
5 ft. = 152.5 cms.	34	17	7.6	22.3
6 ft. = 183 cms.	31	15.5	6.3	20.3
7 ft. = 213.5 cms.	29	14.5	5.4	18.5

Col. I shows drum diameter; Col. II the critical speed in r.p.m.; Col. III the speeds at 50% of critical speed; Col. IV shows speeds when peripheral speed of drum is 120 ft./min. (36.5 m/min.); Col. V shows equivalent percent of critical speed when running at 120 ft./min. peripheral speed.

Originally, our practice was to employ peripheral speeds of 120 ft./min. on plant granulators of 6'6" diameter, equivalent to a rotational speed of 6 r.p.m. The experimental batch unit was therefore run at the same peripheral speed, i.e. 38 r.p.m., which was found to give excellent cascading of material in the drum.

It was found that mixtures which cascaded well in the small batch unit did not do so on the large plant unit, and reference to Col. V in Table I shows that the equivalent percentage of critical speed in the latter case is about 19.5 r.p.m. Thus it was realized that the centrifugal action, which produces the required cascading effect, was insufficient, as the speed of rotation was only one-third of that required to give the same degree of cascading as given by the batch unit.

Fig. 11 shows results obtained in the batch unit with granulator rotating at speeds of 50% and 20% of critical speeds - the latter being analogous to the speed of our full-scale units. It will be observed that the lower speed gave 11% oversize (> 4 mm), and, because the water used for granulation was the same in each case, there was a consequent increase in proportion of fines. Had more water been used at the lower speed, the curve 2 would have been moved bodily to the right, giving an excessive proportion of oversize.

It is clear that for optimum granulation, the speed of movement of particles must be sufficient to distribute the water uniformly within the mass, and at the same time produce sufficient shearing action to break down oversize particles.

Whilst agglomeration of particles in a rotating tube may appear to be an arbitrary process, there is no doubt it follows definite laws and responds to correct conditions. Hardesty & Ross⁽²⁾ in their early paper of 1938, enumerated the effects of some of the variables in granulation, but used a drum of 32" diameter, rotating at only 10 r.p.m. - equivalent to only 21% of critical speed.

An important point in connection with Fig. 11 is that such a wide difference in product was obtained although the granulation process was followed by 10 minutes of rotation in the drying drum.

An increase in speed of our large plant granulators has effected a considerable improvement in granulation efficiency.

(b) Growth of Granules in Drier

It is a well known fact that particle agglomeration of superphosphate mixtures is promoted by increase in temperature as well as by increase in moisture content.

When granulation of cold raw materials is effected by addition of water, followed by drying in a co-current drier, the product size is finally judged from material issuing from the drier. As practised in the U.K., the art of granulation requires judgement of correct degree of agglomeration ex granulator to produce the required final size ex drier, and operators require long experience to become expert at this. Initially they did not appreciate the true extent of granule growth in the drier, nor of the varying amounts of growth with different compound formulations.

Fig. 12 illustrates the growth of particle size for two compounds having widely different superphosphate contents. Curves 1 and 3 show the grading of materials leaving the granulator, and curves 2 and 4 the product ex drier.

The formulations of the two compounds were as follows:

	Compound 9-9-15 %	Compound 5-12½-10 %
15% Single Superphosphate	21.7	56.8
47% Triple Superphosphate	9.6	2.3
60% Muriate of Potash	25.0	16.6
20% Sulphate of Ammonia	43.6	24.3

It will be seen from Fig. 12 that the growth of the 9-9-15 compound in the drier as shown by curves 1 and 2, is much less than the growth of the 5-12½-10 compound which contained 2½ times more superphosphate, as shown by curves 3 and 4.

An interesting point is shown by the upper ends of curves 1 and 2, where it will be noted that the process of drying produced a reduction in oversize particles present in material ex granulator. This is a feature often noticed in our work, particularly when slightly too much water is added for optimum granulation - as was the case in the tests producing curves 1 and 2.

(c) Time of Retention in Granulator.

As an extension to the work previously described on the subject of granule growth in the drier, we carried out a large number of tests on the effect of varying the rolling time in the granulating drum before emptying the partly formed granules into the drying drum.

It was already obvious that as much growth, if not actually more, occurred in the drying process as during the rolling stage immediately after wetting.

A series of tests were planned to find the effect of various rolling times, starting with the addition of water, varying from half to ten minutes. As the time taken to add the water was in each case half a minute, one test involved removing the granules from the granulator immediately after wetting was completed.

Fig. 12A illustrates the results obtained from the runs with rolling times from half to two and a half minutes. Curve 7 shows the grading of the mixed raw materials, and curves 2, 3, 4 and 5, the grading of the material after rolling times of half, one, one and a half and two and a half minutes respectively.

Thus the difference between curves 1 and 2 illustrates the growth which occurred during the wetting process only, without further rollings. Additional growth with further rolling is shown by curves 3, 4 and 5, and it will be noted that no further growth occurred after one and a half minutes.

A separate series of tests in which rolling times varied between two and ten minutes confirmed that growth had ceased after two minutes.

Curves 6 and 7 show the final product obtained after a further ten minutes drying of the materials from curves 2 and 5 respectively. The curves are hardly distinguishable, and the slight difference is probably the result of experimental error, although the rolling time of two and a half minutes appears to give a slightly better product.

Similar results were experienced in the separate series of tests with rolling times varying between two and ten minutes. Here the final product size was slightly smaller after ten minutes, indicating that continued rolling in the wet state tends to break down granule size rather than build it up.

It should be pointed out that the above tests were carried out with a 9:9:15 compound, incorporating 21.7% of superphosphate, but broadly similar results are obtained on other N P K formulations so long as the rolling conditions during the half minute wetting stage have been such as to ensure uniform distribution of the water.

Summarising the results of the above tests, it appeared that growth of granule size was dependent entirely on the rolling action in the drier, and almost independent of the amount of rolling obtained during the wet stage immediately following the wetting process - with the proviso that correct distribution of water had been attained during the actual wetting. The test already described on page 7 emphasises the importance of this latter point.

Our granulators have been found to give a retention time of two and a half minutes, and this would appear to be adequate without being excessive, in view of the above results on the small scale.

(d) Water Requirements of Different Mixtures

The only control left to the operator to obtain granules of the required size is the addition of water, and investigation of many varied plant conditions showed that the operators tended to over-correct for water addition, without appreciating the critical nature of this control, and how it varies with different compounds.

Fig. 13 shows curves representing tests on a 0-19½-19½ compound with the batch unit. Curves 1, 2 and 3 show the final product obtained after adding 7.15%, 7.6% and 8% respectively of water for granulation, the initial moisture content of the feed being 8.25% in each case.

The respective efficiencies of granulation, i.e. product within the 1.5 mm to 4 mm size range, are 66%, 88% and 39%.

The sensitivity of P-K compounds generally is thus well illustrated by Fig. 13, curve 3 showing particularly the danger of over-wetting the material. The differences in water addition between the curves would represent only 10 g.p.h. (45.3 l./hr.) on a 10 ton per hour plant, out of a total water addition of 170 gall. per hour.

Fig. 14 shows three similar curves for a 9-9-15 compound with similar differences of water addition (i.e. 1 gall. per ton of product), and they serve to illustrate the lower sensitivity of compounds containing a lower proportion of superphosphate. The efficiencies of granulation obtained with the three curves are 86%, 89% and 71%. Thus with this compound, fairly wide variations in water can be made without seriously affecting the percentage of product within the required range.

In almost all the formulations based on superphosphate, sulphate of ammonia and potash, we find when "families" of curves are drawn for various water additions, as in Figs. 13 and 14, that the best percentage in grade, for a size ratio of approximately 1:3, is invariably given with the 1.5 to 4.0 mm range, i.e. the curves most nearly approach the vertical within this range.

(e) Particle Size of Sulphate of Ammonia

We have found that in both small and large-scale work, the effect of sulphate particle size is of paramount importance in granulation with cold water.

The two main sources of sulphate available to users in the United Kingdom are -

- (1) By-product material from gas-works or coke-ovens, normally of fine long needle crystal form.
- (2) Synthetic material, i.e. made from synthetic ammonia, which has a larger and more stubby crystal shape, i.e. greater diameter/length ratio.

Fig. 15 illustrates the effect of a fine crystal by-product sulphate on the grading of the mixed raw materials, and on the resultant granulating efficiency. Curves 2A, 2B and 2C show the fine sulphate, mixed raw material and product, respectively. This material required 17% added water for granulation, yet gave only 74% product in the required size range. This was the best product obtainable, and extra water resulted in formation of excessive oversize.

Curves 1A, 1B and 1C illustrate a coarse synthetic sulphate crystal, the mixed raw material made from it, and the resultant product obtained with only 9.8% added water. The product had 97% within the required size range.

Although it is commonly felt that fine soluble salts in the raw material mixture must inevitably require more water for a given degree of agglomeration, owing to the larger surface area, this is not necessarily the sole reason. We found that the rolling characteristics of the mixture 2B were exceedingly poor, and subsequent work has shown that the uneven moisture distribution, resulting from this poor rolling characteristic, is a contributory cause for the extra water requirement, and for the poor quality of product.

When good rolling characteristics can be induced by other means, the water requirements for fine sulphate mixtures can be reduced, and of course, the chemical uniformity of individual granules is improved when fine sulphate is used.

The need to obtain uniform chemical and physical structure in granules of varying size range involves a compromise in the size of sulphate crystal employed.

Tests carried out on closely graded sulphate crystal sizes show that a mean size of 75 μ makes a good compromise and requires only 2% more water for granulation than a mixture containing a coarse crystal as shown by curves 1A and 1B.

Tests on large particle size potash, and with superphosphate containing particles up to 2 mm size, have confirmed that the presence of a relatively small proportion of such coarse particles modifies substantially the rolling characteristics of the material in the granulator, particularly after the addition of the water. It is at this stage where continuation of the rolling is essential, and raw materials containing no particles over 1 mm size tend to slump at this stage, rather than roll. When this happens, granulation efficiency rapidly deteriorates.

(f) Addition of Recirculated Fines to Raw Materials

The tests described in the previous sections were carried out on raw material mixtures made up to the required formulations.

In full scale practice, it is necessary to recirculate fines, as no known process will give 100% of product ex drier in the required size range.

It is common practice to return the fines to mix with the fresh raw materials, and this naturally alters the size grading of the final mixture.

Fig. 16 shows the grading curve of a typical recycled fines from a 9-9-15 compound manufacture (curve 1), and curves 2 and 3 show the grading of the mixed raw feed with and without the fines.

Curve 4 shows the product obtained after addition of 9.8% water to the raw materials containing 25% fines, whilst curve 5 shows the product after adding 9.4% water to raw materials containing no fines.

The fines concerned were taken from a production unit, and comprised cyclone dust, fine granules screened off the 1.5 mm fines screen, and particles removed by an air classifier for removing dust from the material before entering the plant cooler. The latter item removes occasional particles of product size, which accounts for the particles larger than 1.5 mm size shown on curve 1.

Thus Fig. 16 illustrates the small effect which recycled fines produce on the water requirements for granulation.

This was contrary to our own expectations and to general plant experience. Further investigation showed that fines alone could be granulated to give an excellent product of the required size range with the addition of only 6.7% moisture.

Thus fines have a two-fold effect on granulation -

- (1) They reduce slightly the nett amount of water required for granulation.
- (2) They provide coarser particles to the raw feed which promotes better rolling in the granulator.

Plant difficulties with fines were traced to variations in fines:raw materials ratios. In some of our works, fines were not mixed with the raw materials, and frequently entered the granulator undiluted with raw materials. The change in water requirements made it difficult for the operator to follow with correct control, and instability resulted. By thoroughly mixing the recycled fines with the raw materials, and artificially controlling the rate of addition to the raw materials (so as to maintain a constant ratio), we have eliminated the problems normally associated with recycling of fines.

(g) Effect of Sprays versus Jets for Water Addition

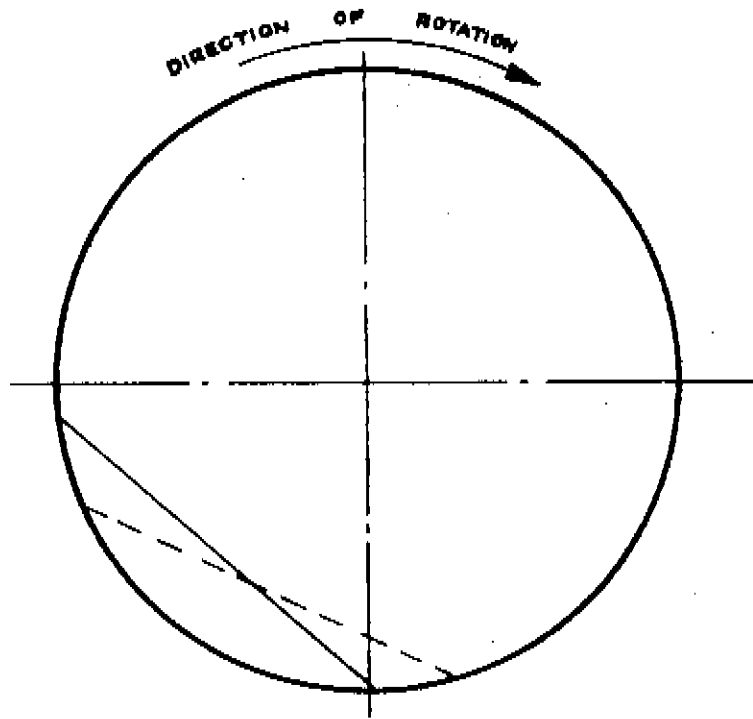
As a result of varied experience on the works, where in some instances an improvement in granulation had resulted from employment of an open water jet in place of sprays, an investigation was carried out on the batch unit to confirm the superiority of one method or the other.

Fig. 17 illustrates the results obtained.

Due to the employment of a rather large particle size feed, the product size was larger than normal with the usual 9.8% addition of water. However, the relative positions of the two curves show that when water was added by jet (curve 3), there was an increase in oversize formation, i.e. of granules above 5 mm, amounting to 12%, and some granules of $\frac{1}{2}$ " (13 mm) were formed. Curve 2 shows the product made by addition of water by spray, and curve 1 shows the grading of raw materials employed for the tests.

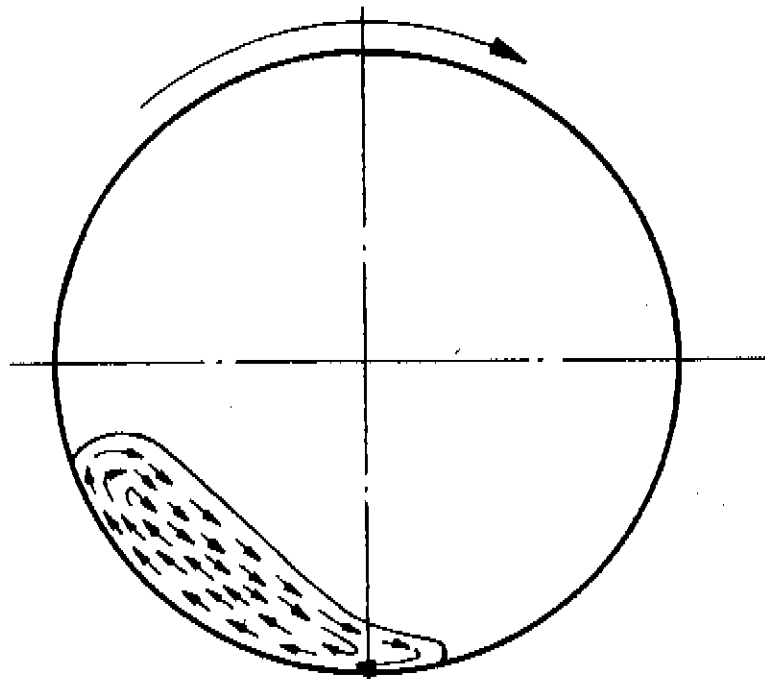
Further investigation on the plants showed that where jets had proved superior to sprays, the latter had been positioned too far from the rolling bed of material, and the spray mist spread to the uncovered surface of the shell. This produced a tendency for the material to stick to the shell surface, from which it eventually broke away in lumps which formed oversize.

A repositioning of sprays on works granulators close to the material prevented undue spread of water mist, and gave improved results over the method of water addition by open jet.



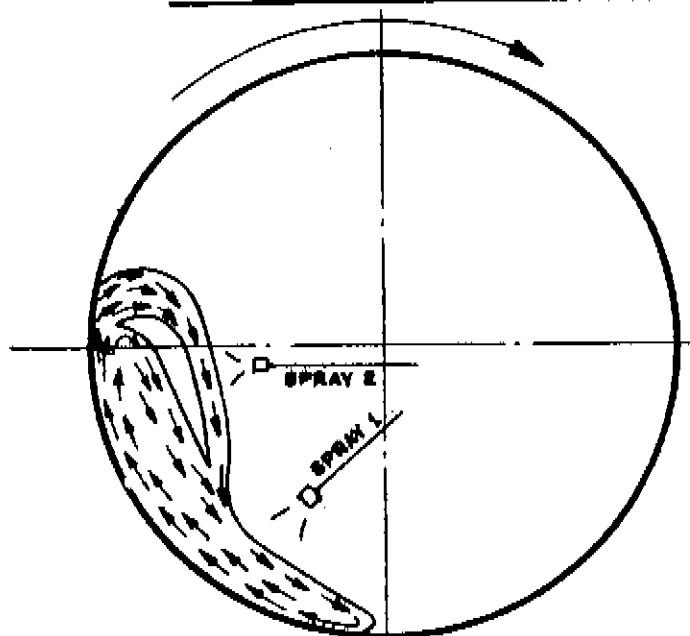
SEE-SAW MOTION WITH MASS RELATIVELY STATIC.

FIGURE 1.



ROLLING MOTION WITHOUT CASCADING
DUE TO LOW SPEED OF ROTATION.

FIGURE 2.



IDEAL CASCADING MOTION
RESULTING FROM CORRECT SPEED OF ROTATION.

FIGURE 3.

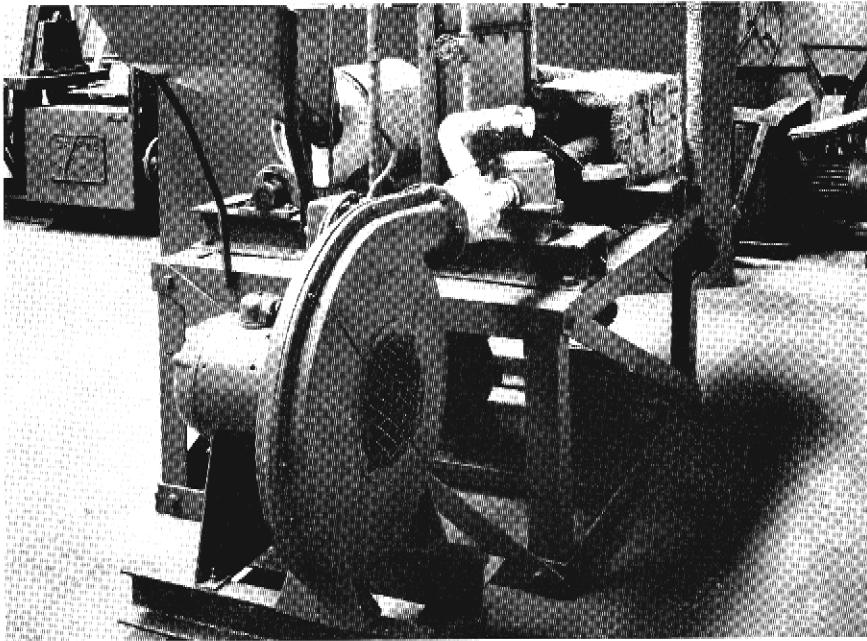


FIG. 5

Drying Apparatus — View of Air Fan, Mixing Valve and Combustion Chamber.

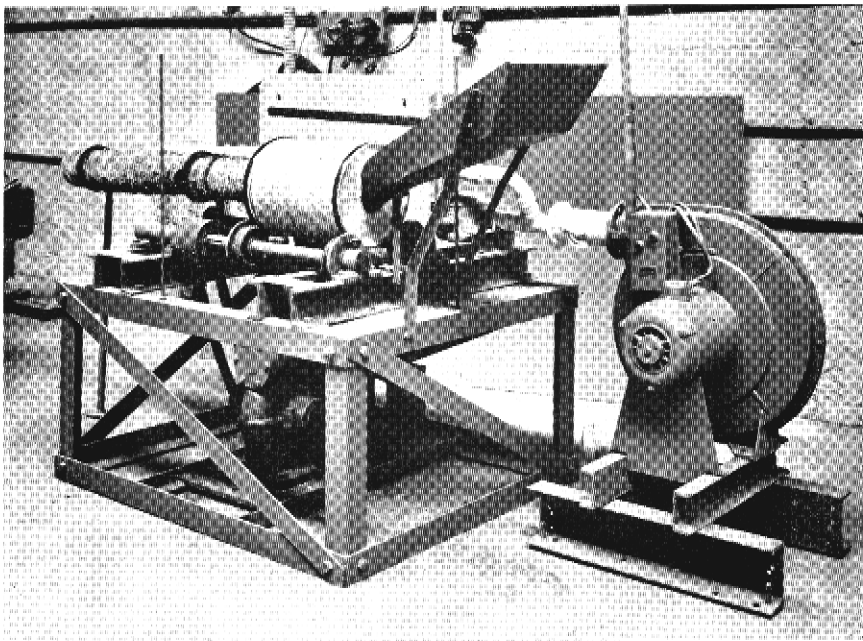


FIG. 6

Drying Apparatus — showing Hot Gas Duct entering Drying Drum, with Feed Chute in Foreground.

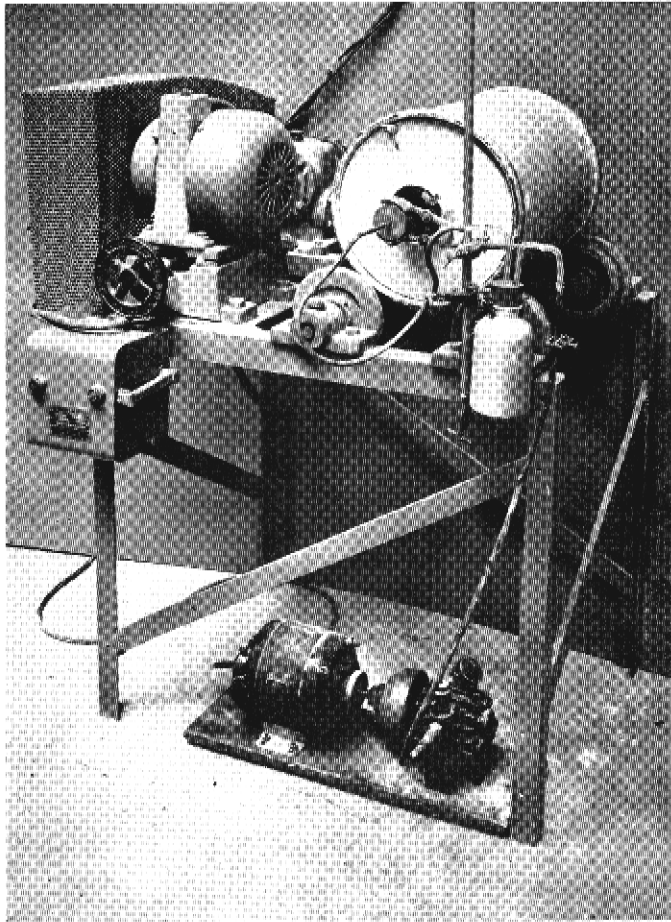


FIG. 4

General View of Granulator Drum on Variable Speed Mounting.

(h) Positioning of Water Sprays

Whilst investigating the effect of granulator speeds, we experimented with various positions of the spray nozzles relative to the bed of rolling and cascading materials.

Reference to Fig. 3 shows two alternative positions of water spray, (1) and (2).

Position 1 was that normally employed on our Works, but position 2, tried on the batch granulating unit with material cascading correctly as shown in Fig. 3, produced an immediate improvement by reduction in oversize formation. For a given amount of water, granules over 4 mm size were reduced by as much as 5%. This made a noticeable difference to the appearance of the product, although it does not show up appreciably on grading curves.

When the material merely rolls without cascading as in Fig. 2 the position of the spray does not appear so important.

Undoubtedly, the improvement with the higher spray position, when the material cascades, is due to the better penetration and distribution of water through the falling curtain.

CONCLUSIONS

The preceding tests illustrate the effect on granulation of some of the more important variables which occur in actual practice on the Works.

Many other factors such as time of retention in the drier, depth of bed in the granulator, effect of build-up within the granulator shell on formation of oversize, positioning of hammers to remove build-up, moisture content of raw materials, effect of different formulations and proportion of soluble salts, and effect of temperature of raw materials, have been studied.

The author believes that of the 700 individual tests we have carried out on the small batch unit, those experiments which have been described in this paper have resulted in the most noticeable improvements in granulation by the rotating drum method.

ACKNOWLEDGEMENTS

The author wishes to express his indebtedness to his colleagues, and in particular to Mr. R. W. Palmer, who supervised the experimental work described, and assisted in the preparation of the paper - and to the Directors of Fisons Limited for permission to publish the work.

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Note: Millimetre scale can be used to measure size ratios

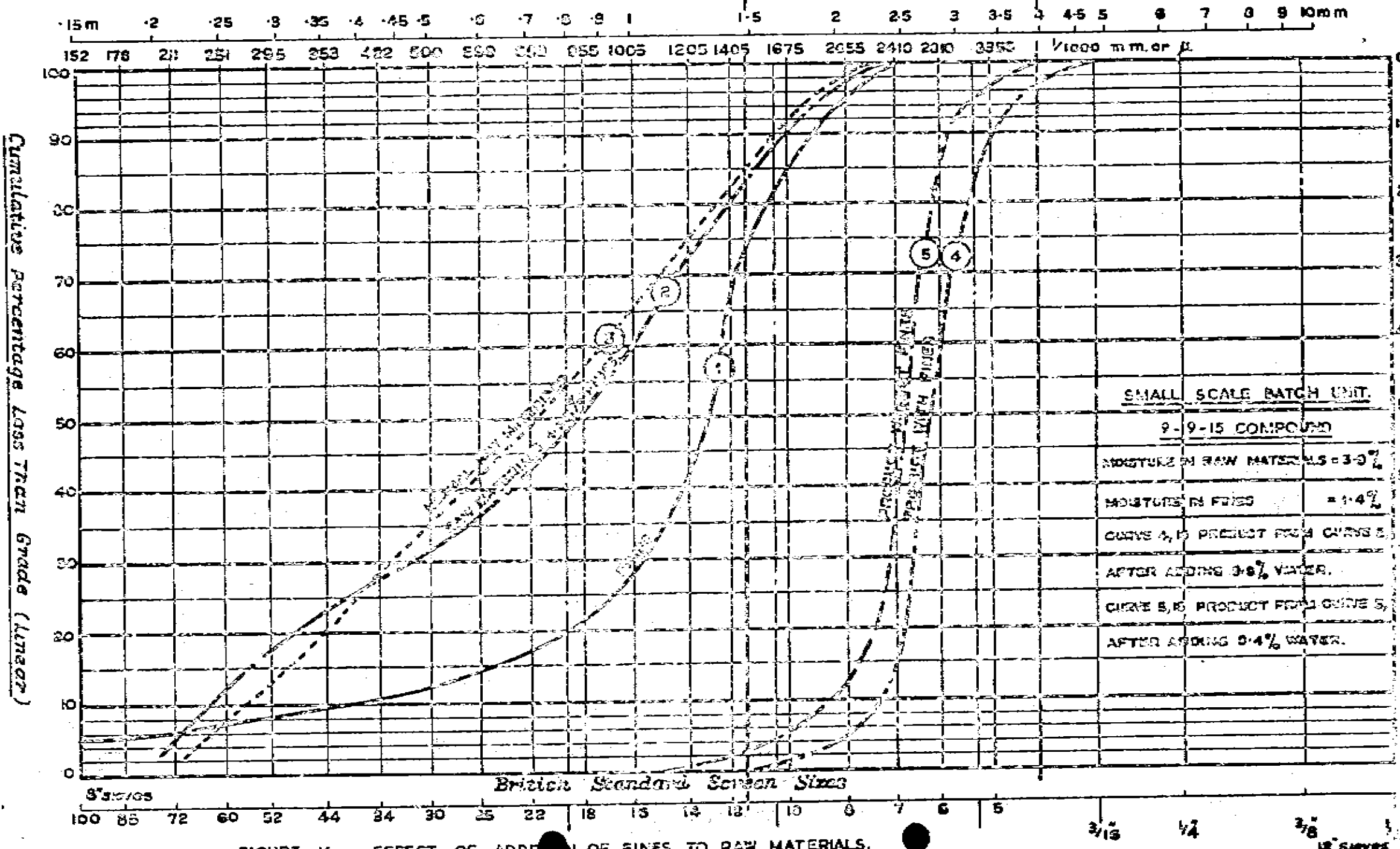


FIGURE 16. EFFECT OF ADDITION OF FINES TO RAW MATERIALS.

3/8 1/2 5/8 3/4 1 1 1/4 1 1/2 1 3/4 2 2 1/4 2 1/2 3 3 1/4 3 1/2 4 4 1/4 4 1/2 5 5 1/4 5 1/2 6 6 1/4 6 1/2 7 7 1/4 7 1/2 8 8 1/4 8 1/2 9 9 1/4 9 1/2 10

Note: Millimetre scale can be used to measure size ratios

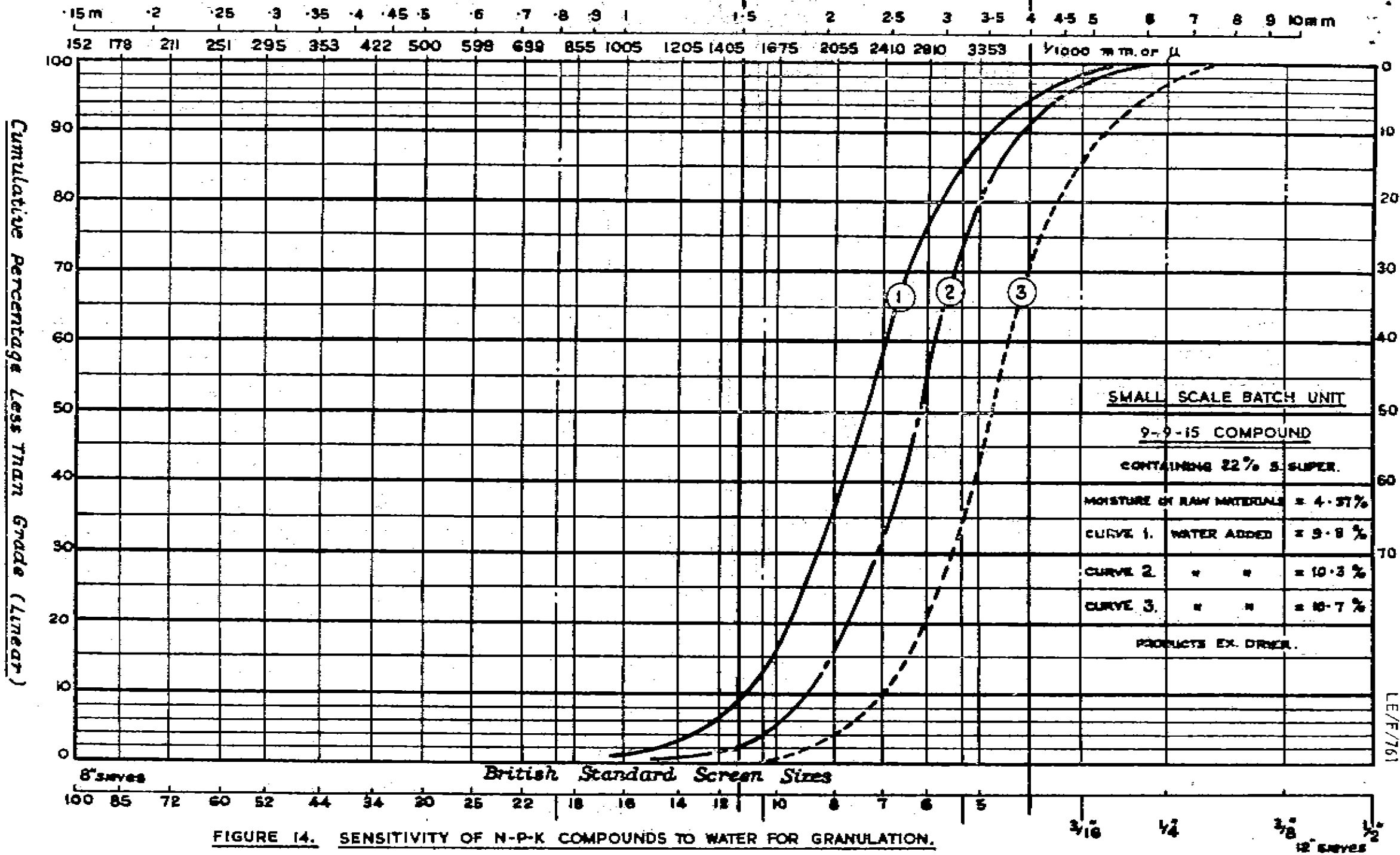
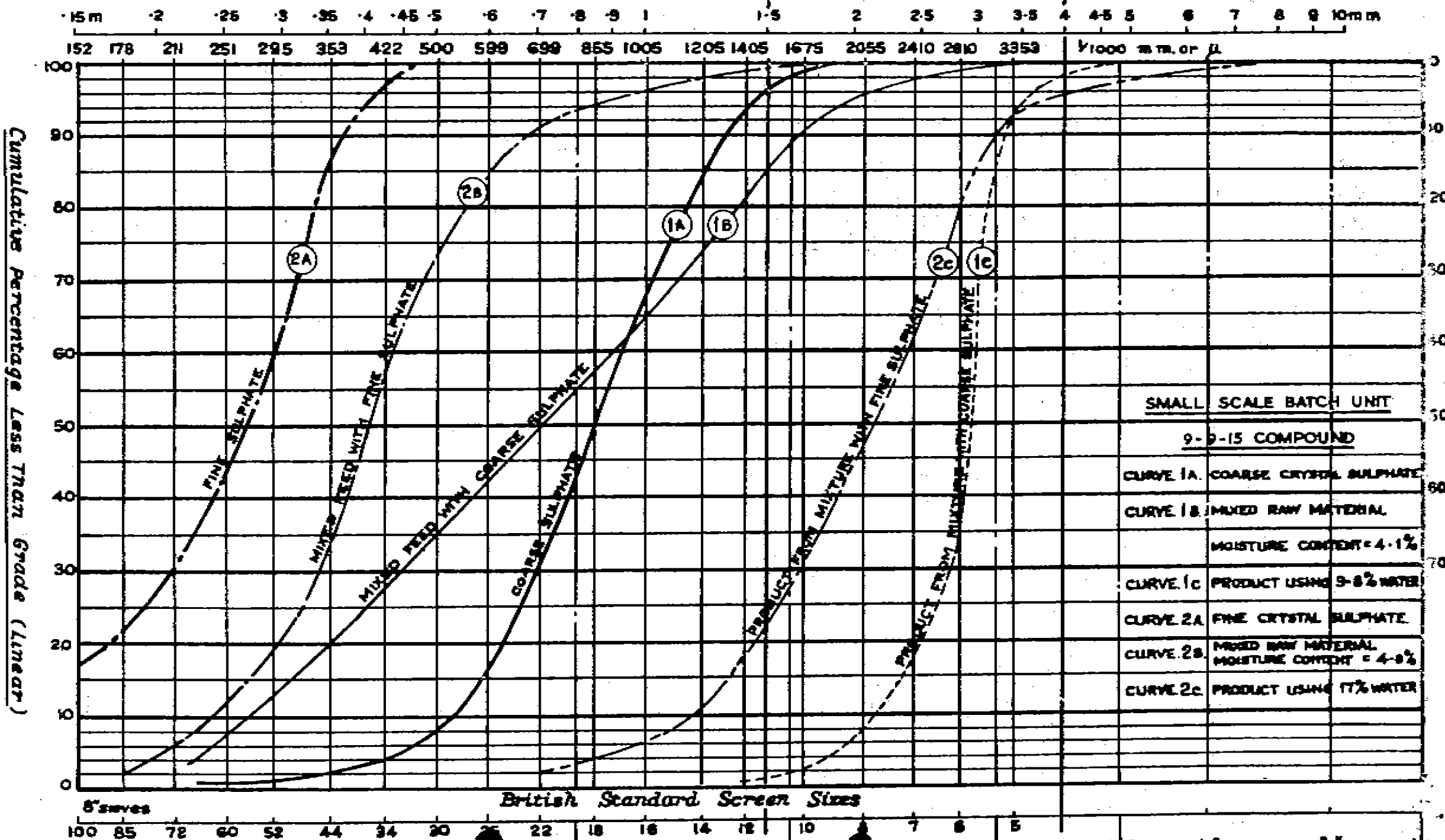


FIGURE 14. SENSITIVITY OF N-P-K COMPOUNDS TO WATER FOR GRANULATION.

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 Note: Millimetre scale can be used to measure size ratios



SMALL SCALE BATCH UNIT

9-9-15 COMPOUND

CURVE 1A	COARSE CRYSTAL SULPHATE
CURVE 1B	MIXED RAW MATERIAL
	MOISTURE CONTENT = 4.1%
CURVE 1C	PRODUCT USING 9.8% WATER
CURVE 2A	FINE CRYSTAL SULPHATE
CURVE 2B	MIXED RAW MATERIAL
	MOISTURE CONTENT = 4.8%
CURVE 2C	PRODUCT USING 17% WATER

FIGURE 15. EFFECT OF FINE SULPHATE / AMMONIA ON MIXED FEED & PRODUCT.

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Note: Millimetre scale can be used to measure size ratios

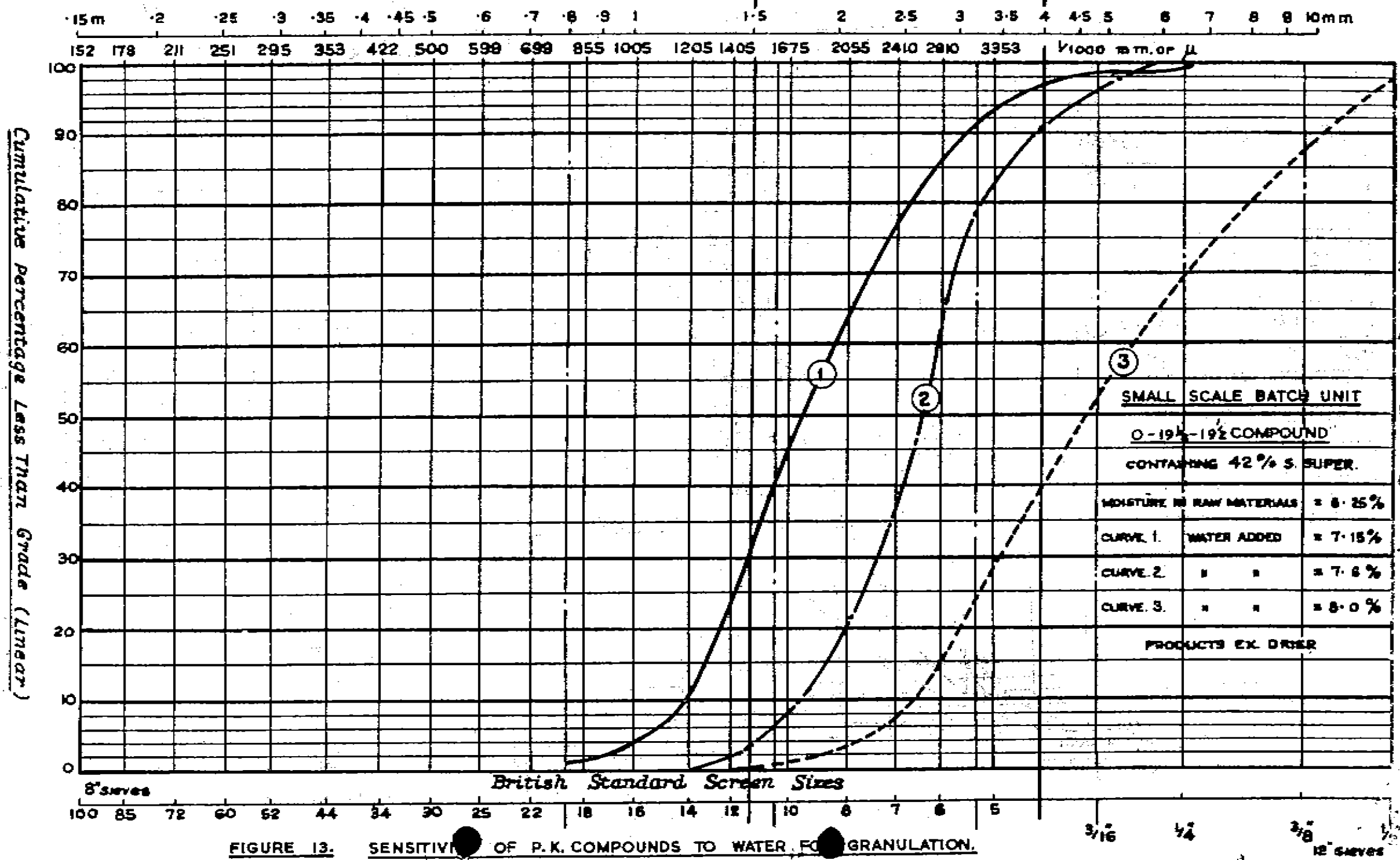


FIGURE 13. SENSITIVITY OF P.K. COMPOUNDS TO WATER FOR GRANULATION.

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Note: Millimetre scale can be used to measure size ratios

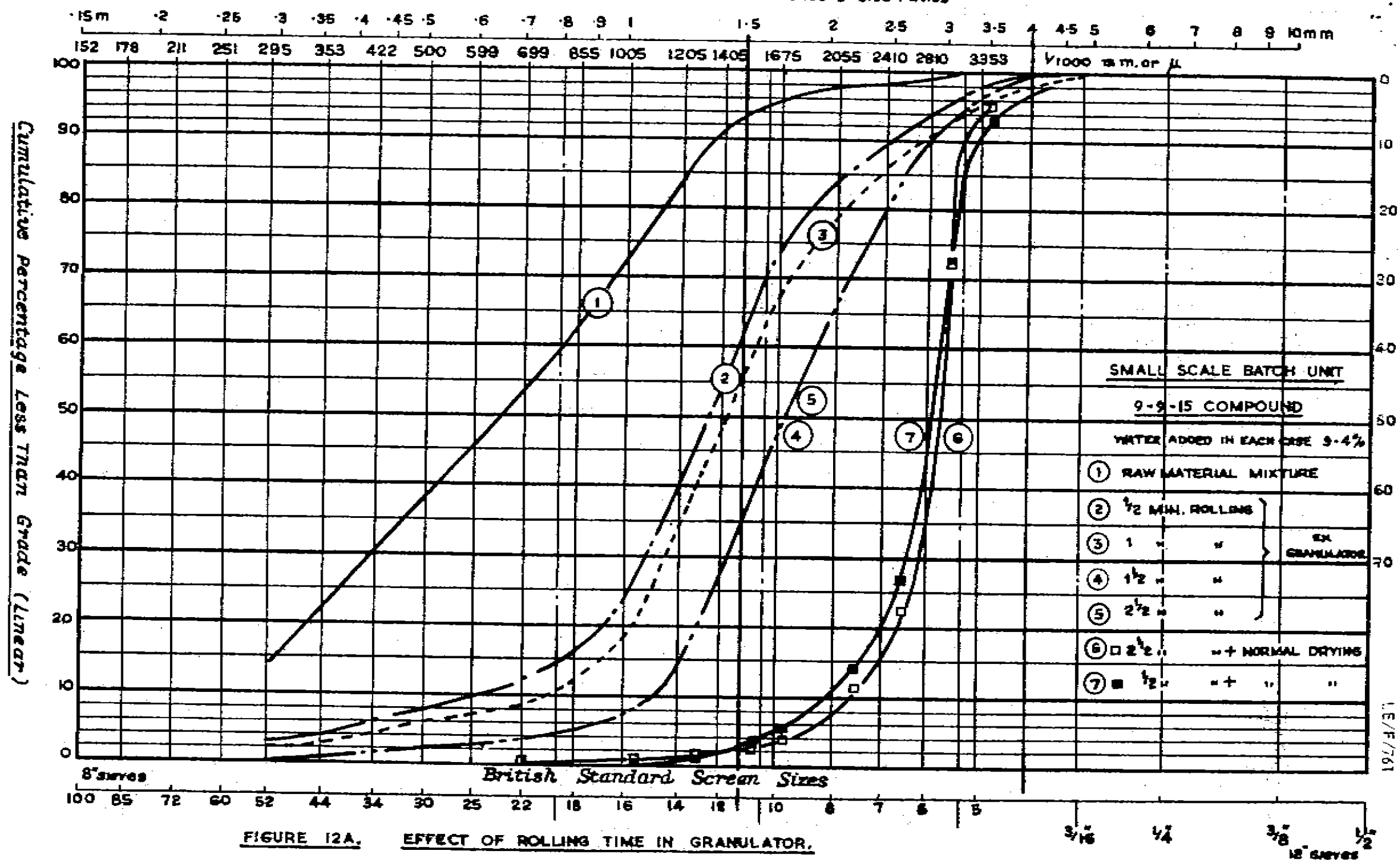


FIGURE 12A. EFFECT OF ROLLING TIME IN GRANULATOR.

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Note: Millimetre scale can be used to measure size ratios

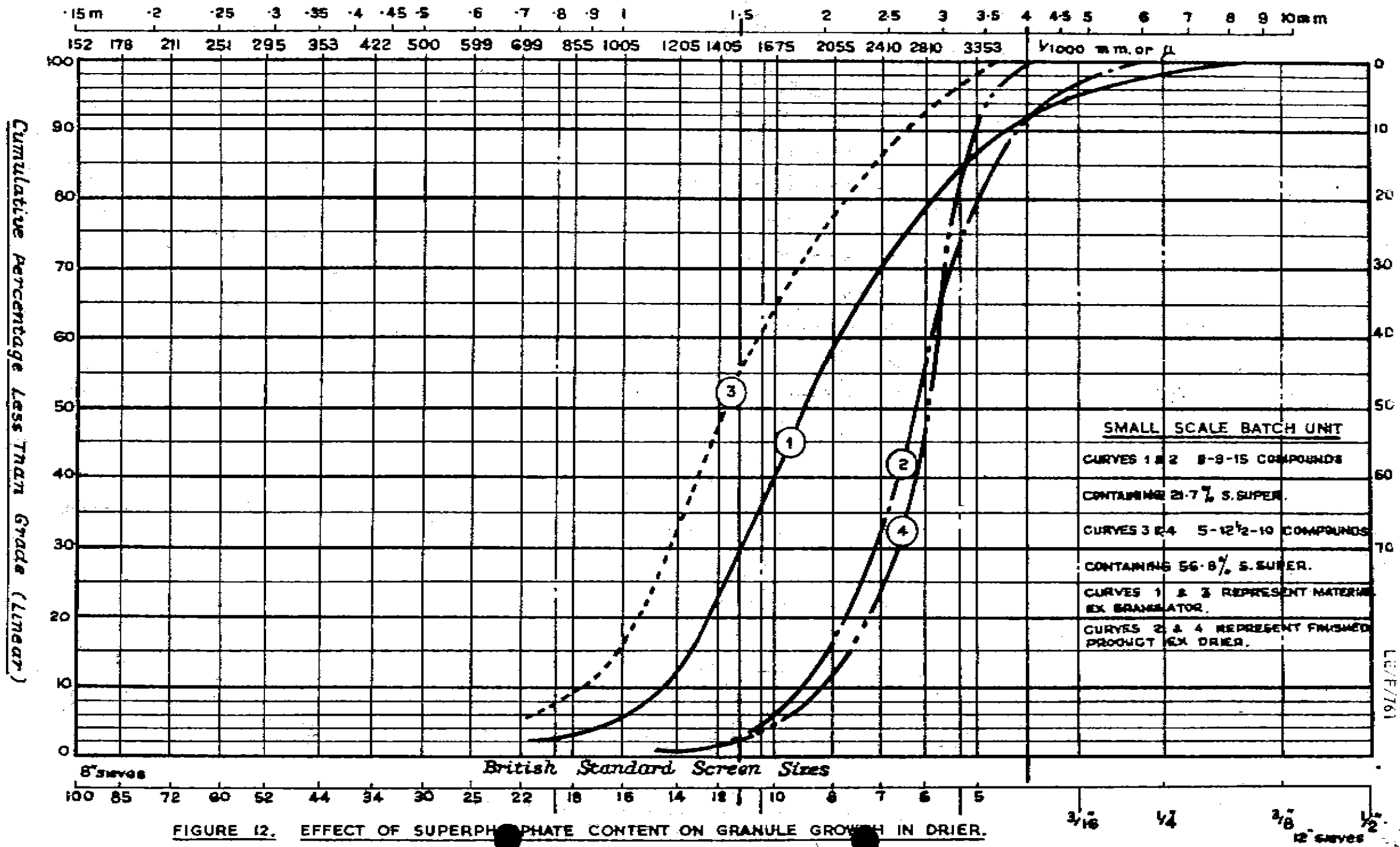
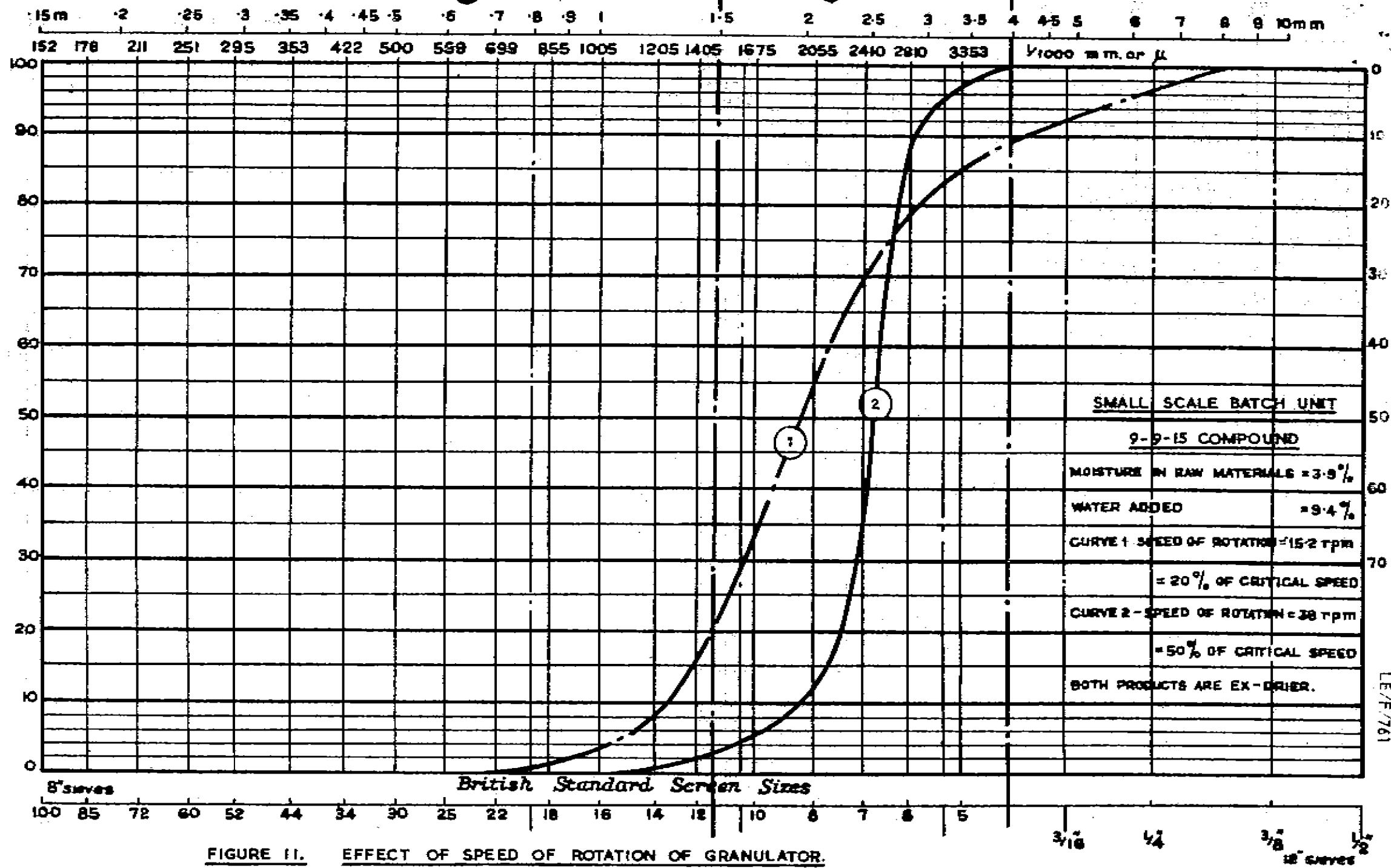


FIGURE 12. EFFECT OF SUPERPHOSPHATE CONTENT ON GRANULE GROWTH IN DRIER.

Cumulative Percentage Less Than Grade (Linear)



SMALL SCALE BATCH UNIT
9-9-15 COMPOUND
 MOISTURE IN RAW MATERIALS = 3.9%
 WATER ADDED = 9.4%
 CURVE 1 - SPEED OF ROTATION = 15.2 rpm
 = 20% OF CRITICAL SPEED
 CURVE 2 - SPEED OF ROTATION = 38 rpm
 = 50% OF CRITICAL SPEED
 BOTH PRODUCTS ARE EX-DRIER.

FIGURE 11. EFFECT OF SPEED OF ROTATION OF GRANULATOR.

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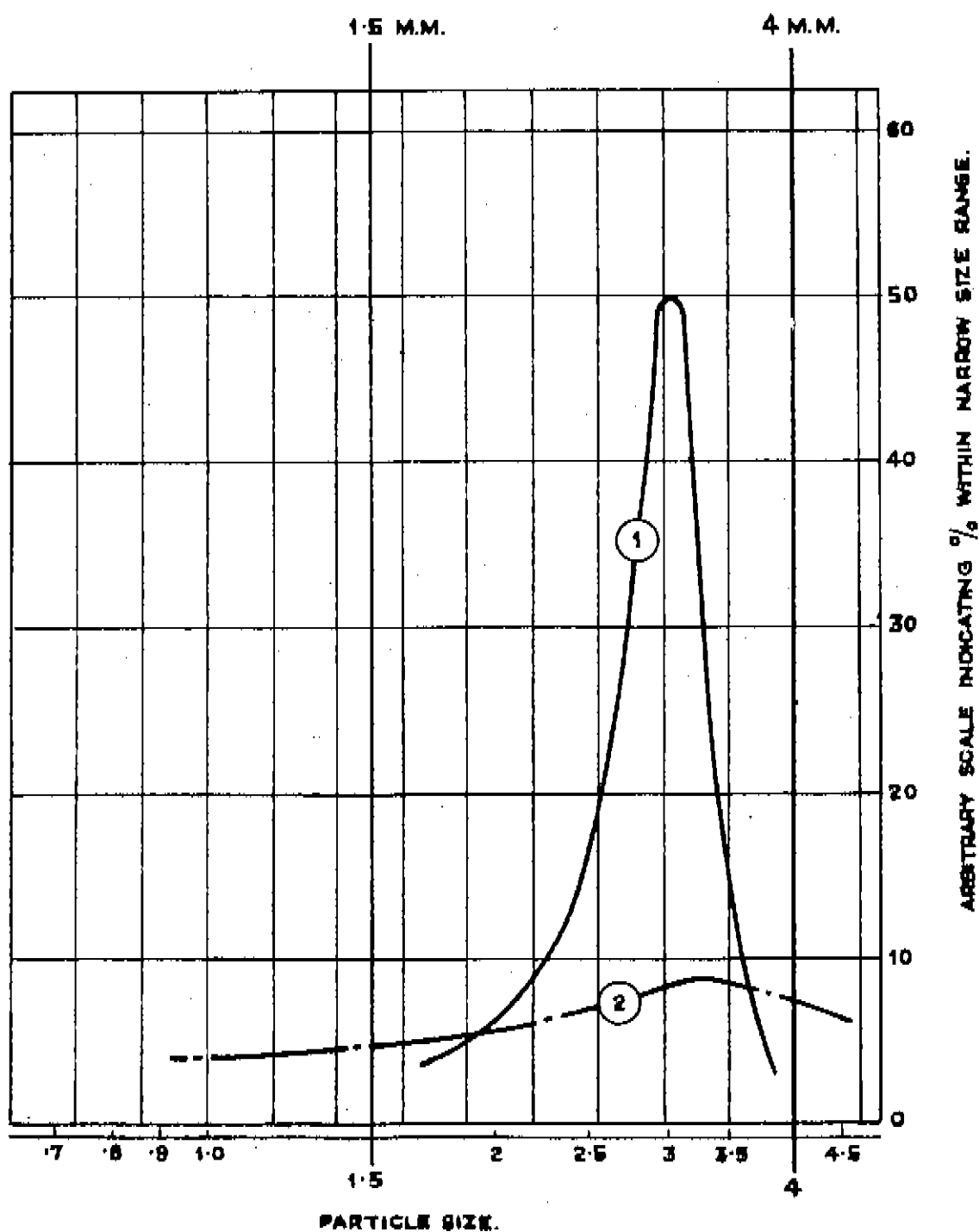


FIGURE 10.

SLOPES OF CURVES ① & ② FROM FIG. 8.

SCALED TO EMPHASIZE THE DIFFERENCE IN SIZE DISTRIBUTION.

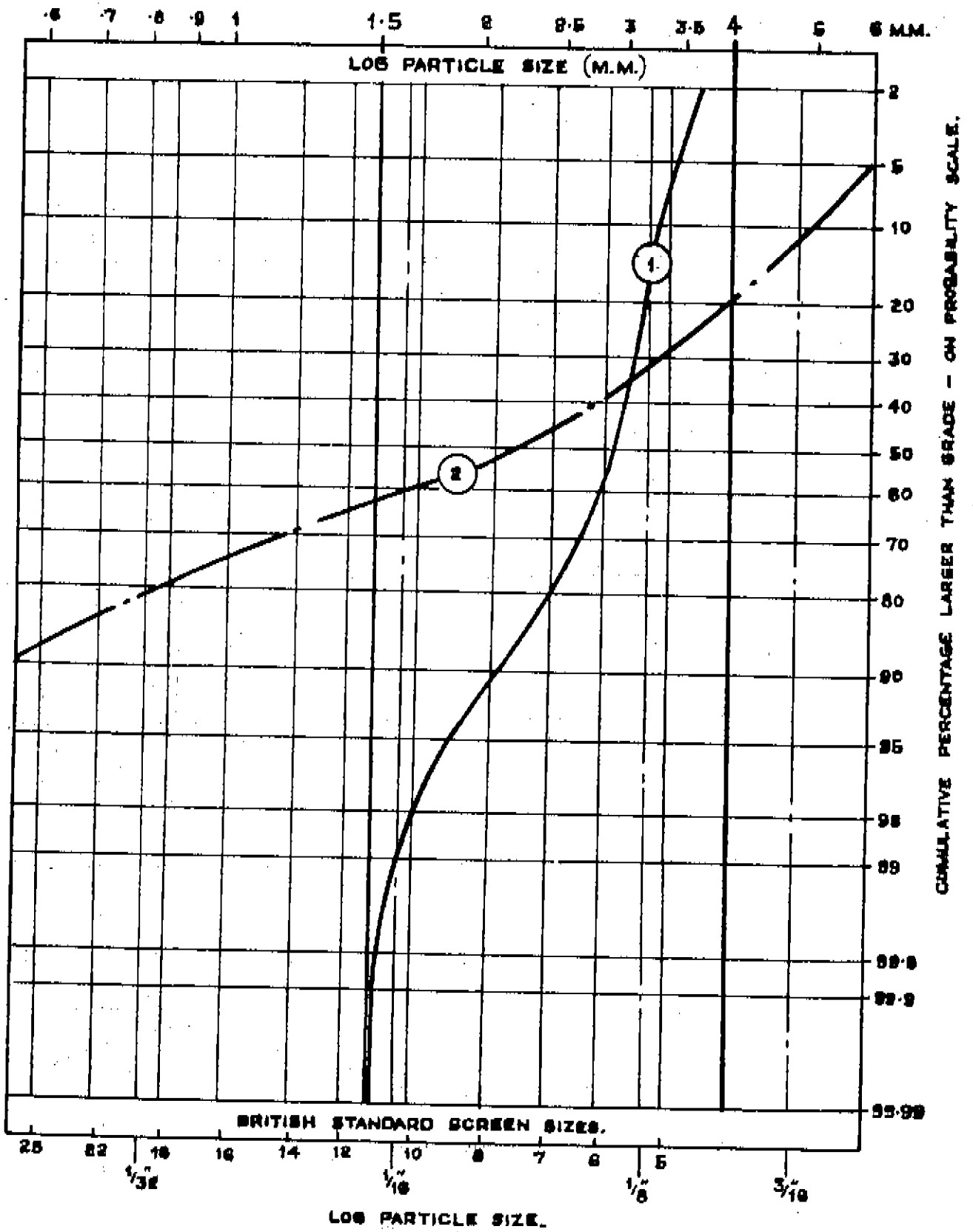


FIGURE 2-

CURVES FOR GOOD QUALITY PRODUCT ① & POOR QUALITY PRODUCT ②
 PLOTTED ON LOG SIZE V PROBABILITY CHART.

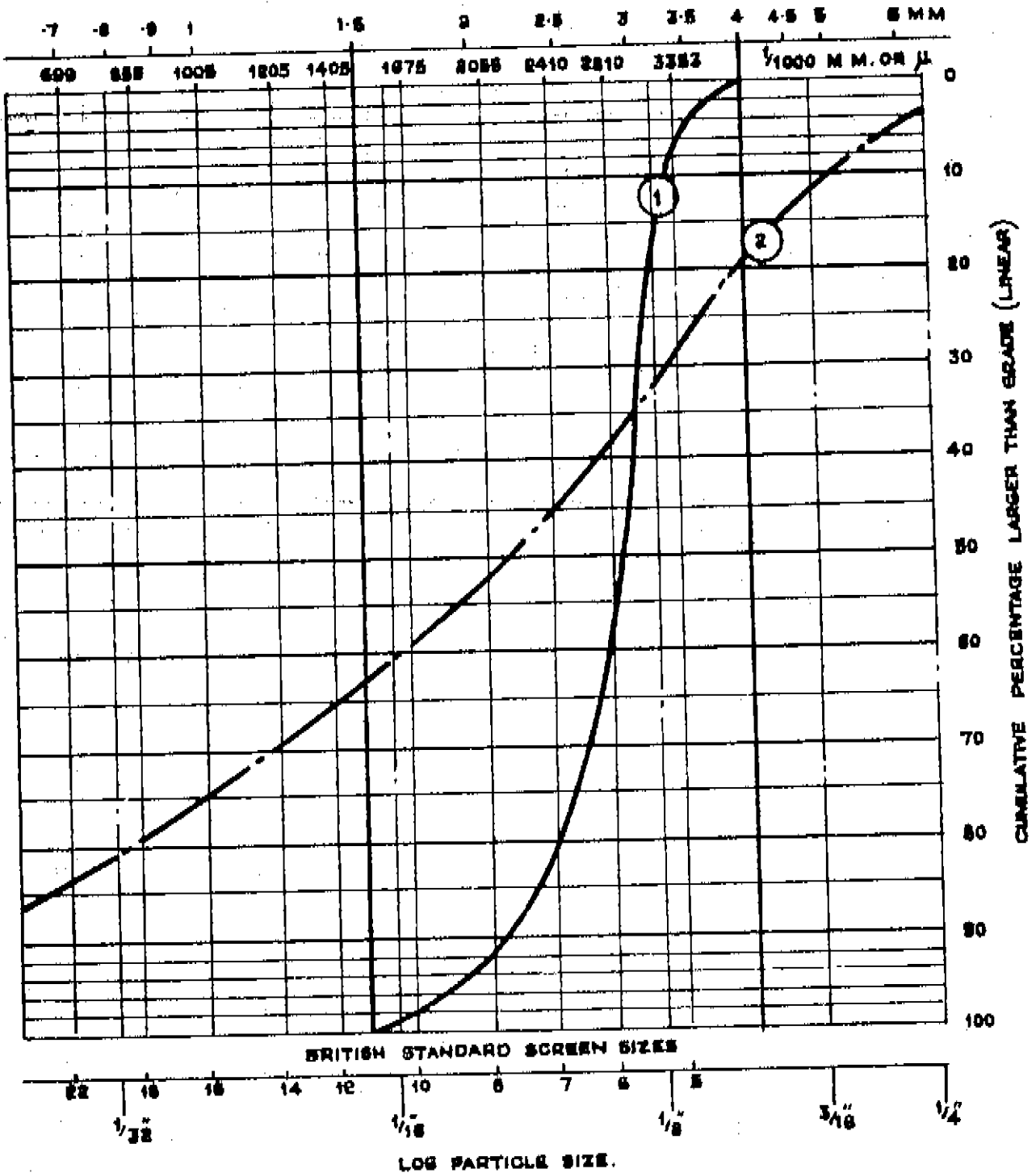
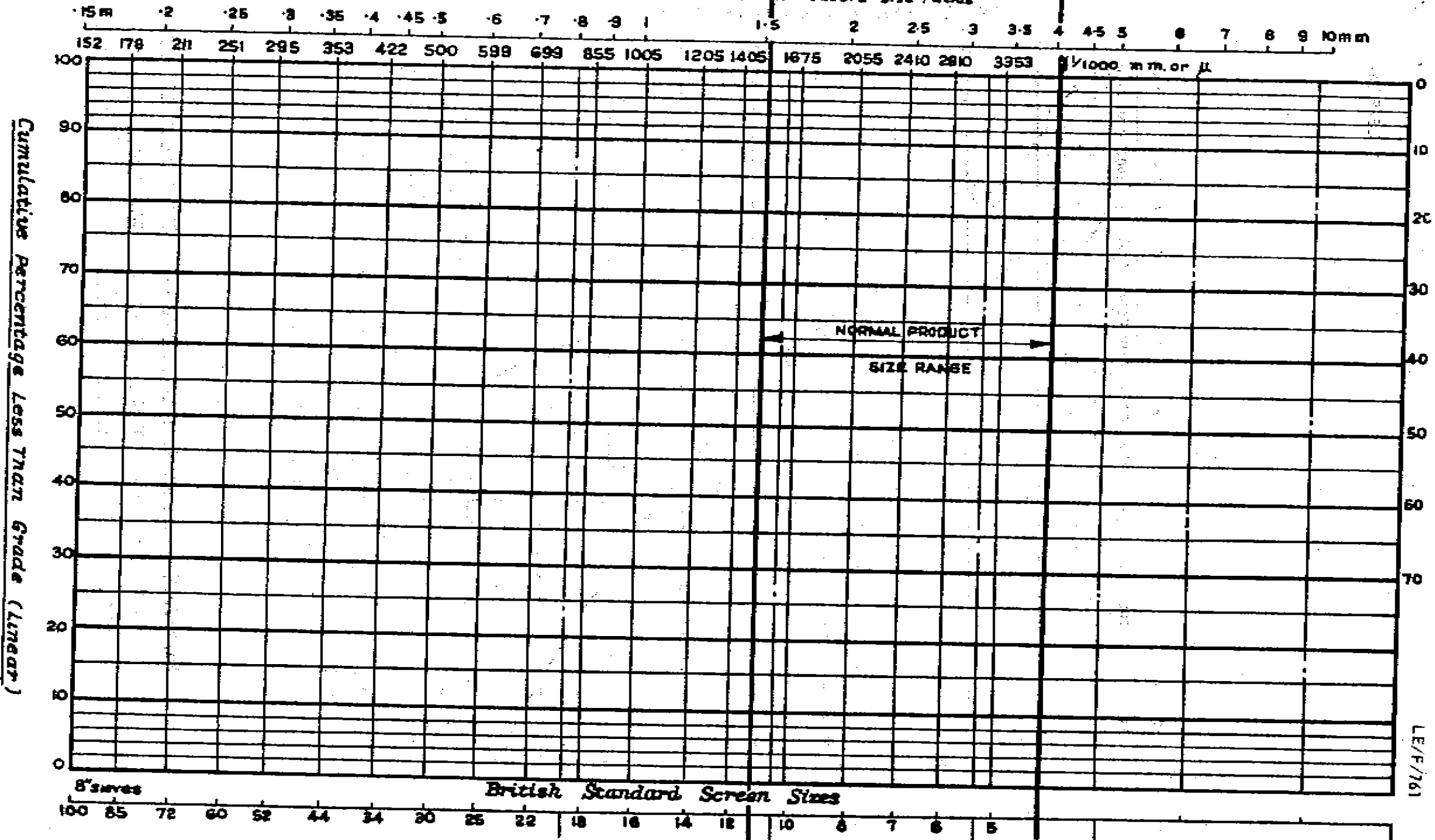


FIGURE 8.

SHOWING GOOD & POOR QUALITY PRODUCTS PLOTTED ON FISON'S CHART.
 CURVE 1 - GOOD PRODUCT WITH 100% IN SIZE RANGE 1.5-4.0 M.M.
 CURVE 2 - POOR " " 43% " " " 1.5-4.0 M.M.

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Note: Millimetre scale can be used to measure size ratios



Cumulative Percentage Less Than Grade (Linear)

LE/F/161

FIGURE 7. 'FISON' GRADING CHART.

Abb. 1

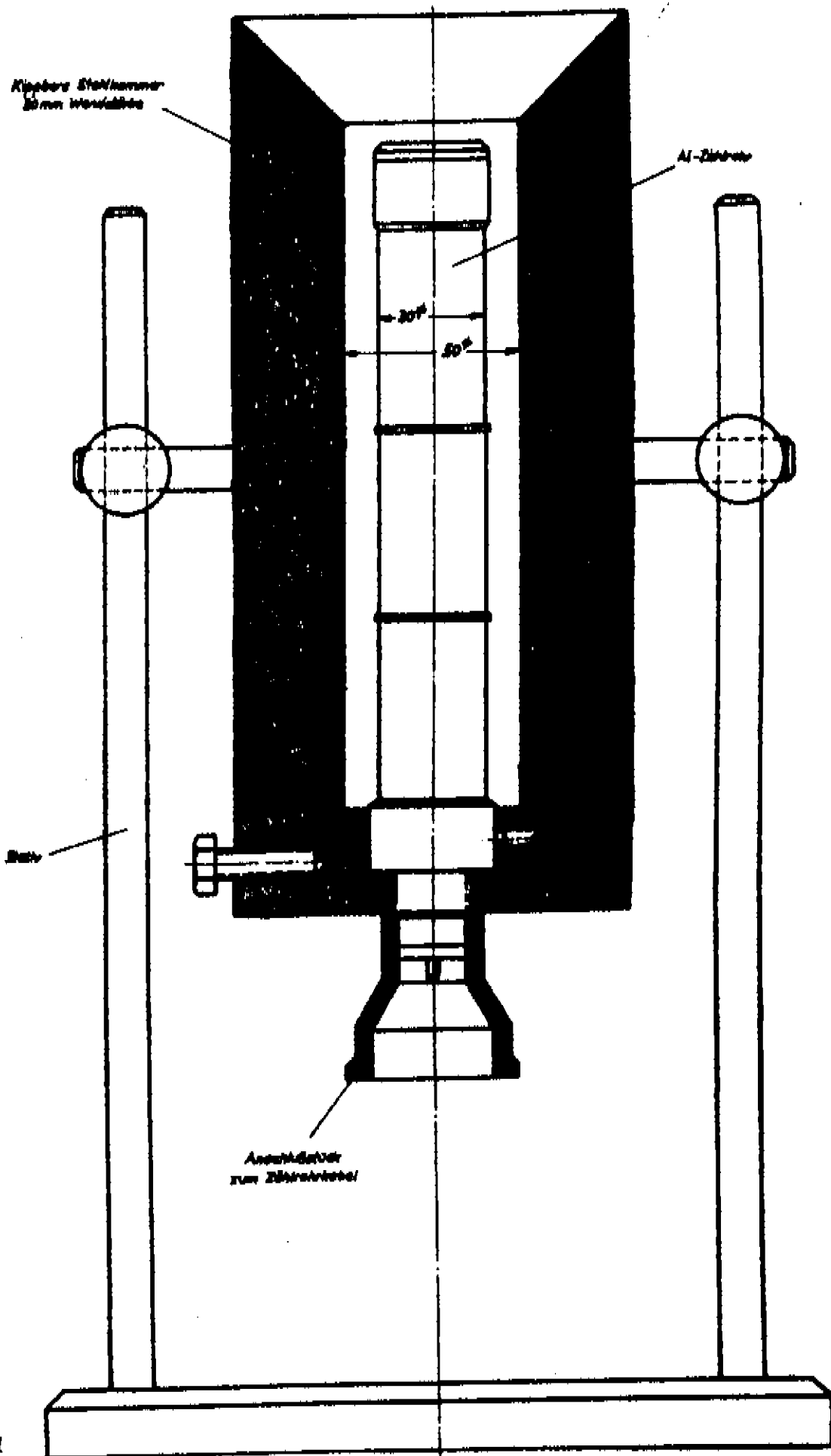


Abb. 1

Abb. 2

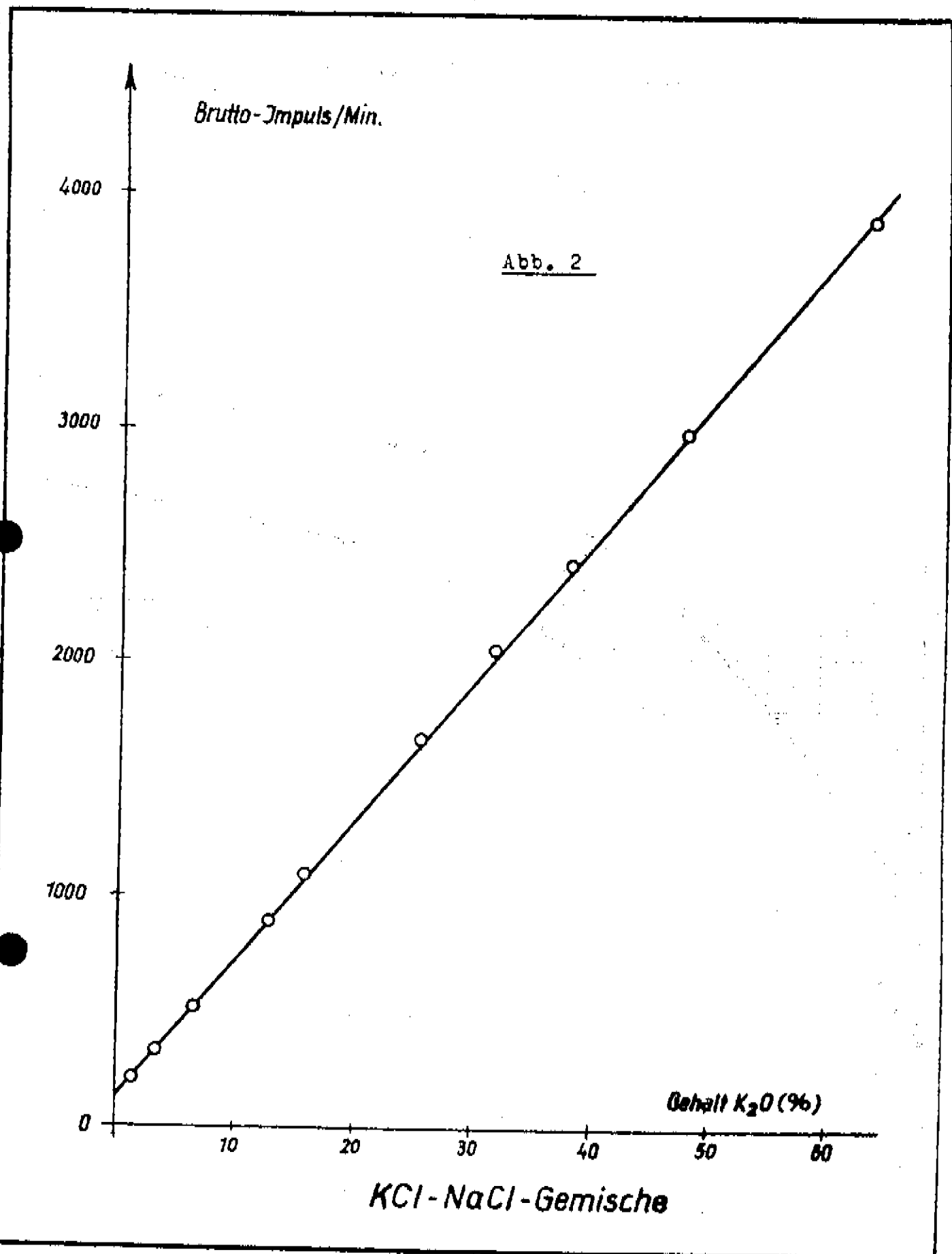


Abb. 2

KCl-NaCl-Gemische

Abb 3

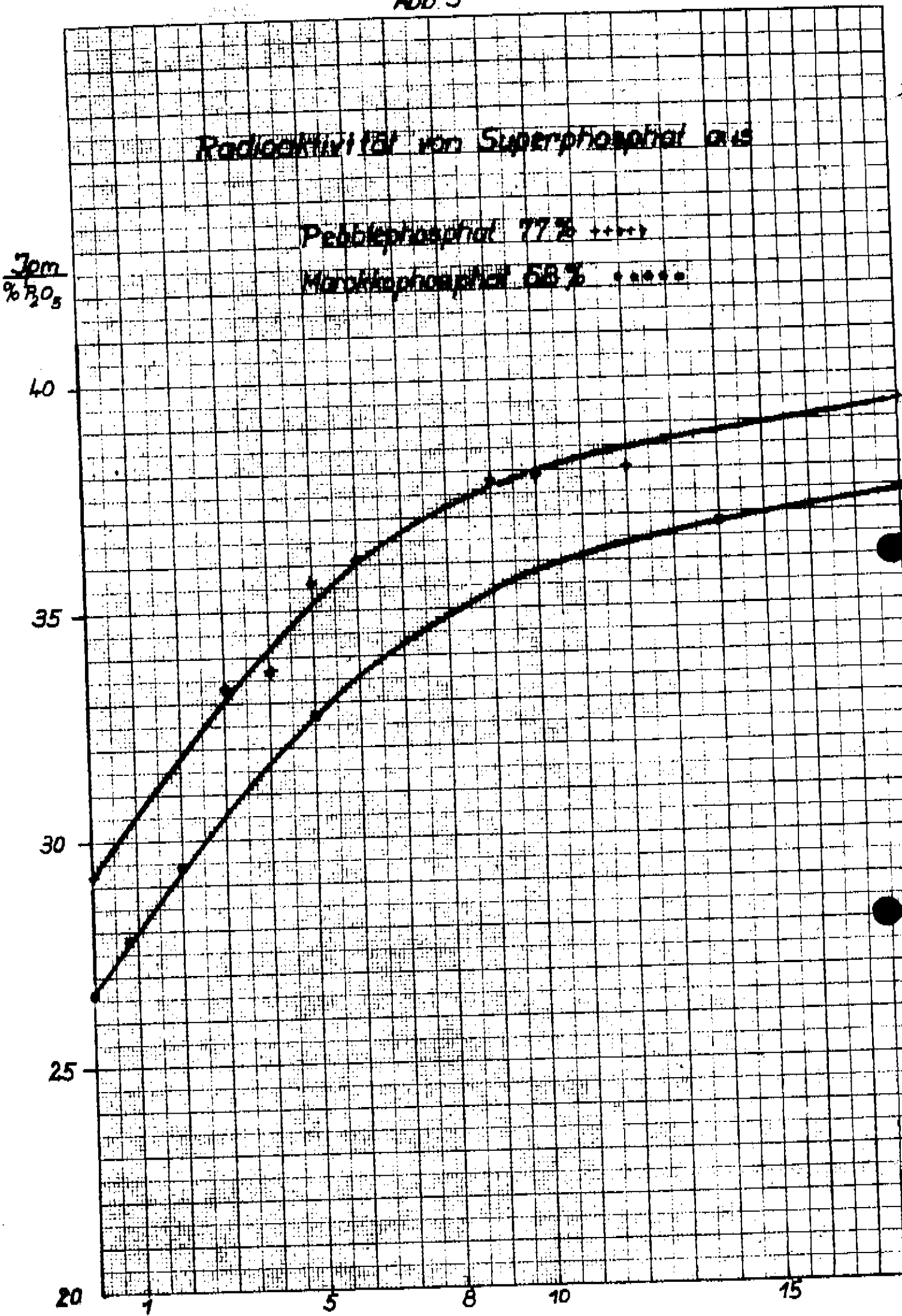


Abb. 4

Eichung des β -Zählrohrs mit
 $KCl - (NH_4)_2 SO_4$ -Gemisch

