THE SIZE ANALYSIS OF PHENOTHIAZINE

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Received May 15, 1959

Results are given for the analysis of particle size distribution of a sample of finely ground phenothiazine by four different methods. Each method is reproducible and is in use for the size analysis of phenothiazine in different laboratories, but the results differ considerably. The advantages and disadvantages of each method are discussed briefly, and a means of comparing the results with those obtained by surface area measurements is shown.

Phenothiazine, or thiodiphenylamine, has been used for many years as an anthelmintic, mainly in sheep and cattle. It is manufactured by the reaction of diphenylamine and sulphur in the liquid state, and crystallises from the melt on cooling, and one or more grinding processes have to be gone through before the material is of a suitable size for use.

Phenothiazine is practically insoluble in water and is now used either as a tablet, a dispersible powder, or a ready-made liquid suspension. The dispersible powder is the most popular in this country, and is included, together with the tablets, in the B.Vet.C. The particle size requirements of the British Veterinary Codex monograph are "not more than 0·1 per cent should be retained on a No. 25 sieve (600 microns) and not more than 5·0 per cent on the No. 100 sieve (150 microns)".

In 1956 Gordon[1] published results of experiments on sheep which showed that the finer the particle size the greater the anthelmintic effect, and since that time the emphasis on "fine particle size" phenothiazine has gradually increased. The particles in "fine particle size" phenothiazine are too small for a sieve analysis to be of value because the finest sieve which is robust enough for practical use is the 200 mesh B.S.S. which passes particles less than 76 microns. Size analysis of particles less than 76 microns and greater than 2 microns can be done by various means but a comparison between the results obtained on a single sample of phenothiazine has not so far been published.

Methods of Size Analysis

The two common methods of sizing in the "fine particle size" range are microscope counting and sedimentation. The results for the two methods are not identical since the parameters are different. With the microscope the projected area of the particle is matched with one of a series of circles on a reference graticule. In sedimentation methods the property measured is the free falling speed, and the particle size is defined as the diameter of a hypothetical sphere whose falling speed is the same as that of the particle under identical conditions. There are several variations of the sedimentation technique, three of which are described. These are the Andreasen pipette[2] (a fixed position pipette method), the Stairmand[3] apparatus (a

liquid column method with sediment extraction), and the Micromerograph[4] (a gas column method with sediment accumulation).

A third method, that of surface area measurement, which may be translated into mean particle size if desired, gives no information on the distribution of particle sizes in the sample. Two well-known methods of measuring surface area of solids are available: one involves measuring the volume of gas, usually nitrogen, adsorbed by a given weight of sample at liquid air temperatures[5], and the other the permeability of a compressed plug of powder, to a fluid, usually air, under ordinary atmospheric conditions[6]. The latter is simpler and gives reproducible results, but the results differ widely from those obtained by the former unless extensive corrections are applied.

Microscope Method

Phenothiazine is best prepared for microscope counting by suspending in arachis oil, and a suitable concentration is obtained by trial and error.

One drop of the suspension is placed on a microscope slide and a cover slip pressed lightly over it. The slide is examined with either a bench microscope, using an 8 mm. objective and a 10x eyepiece, or, preferably, a projection microscope. A reference graticule consisting of a series of circles and rectangles[7] is used. The diameter of the circles against which the particles are matched, are arranged as multiples of $\sqrt{2}$ starting with 76 μ as the highest value, and the rectangles, the lengths of whose sides are simple multiples of the circle diameters, form the fields, inside which the particles are counted. This means that successive circles each have twice the area of the preceding one. The number of fields counted depends on

TABLE I

MICROSCOPE SIZE ANALYSIS OF FINELY GROUND PHENOTHIAZINE

Size range (μ)		Volume factor $\frac{d_1^3 + d_2^3}{2}$	Number of fields observed	Number particles sized	Number of particles per basic area (n)	Volume per unit area nd^3	Weight per cent in grade	Weight per cent less than upper limit of size range
d_1	d_2							
26·5	18·8	12,627	20	14	14	176,778	15·15	100·00
18·8	13·2	4,472	20	48	48	214,656	18·39	84·85
13·2	9·4	1,565	20	124	124	194,060	16·63	66·46
9·4	6·6	559	10	188	376	210,184	18·01	49·83
6·6	4·7	196	5	208	832	163,072	13·97	31·82
4·7	3·3	69·9	5	364	1,456	101,774	8·72	17·85
3·3	2·3	24·1	2	204	3,040	73,264	6·28	9·13
2·3	0	6·1	2	546	5,460	33,306	2·85	2·85
						1,167,094		

the density of particles on the slide, and since the smallest particles greatly outnumber the largest they need not be counted in all the fields required to obtain a reasonable number of the largest particles. The fields to be counted are selected according to a definite scheme such as that given in Appendix XIII of the British Pharmacopoeia, 1958. Obviously the figures obtained, if used directly, give an analysis relating to the number of particles in given size ranges. It is usual to translate such figures into a weight analysis by assuming all the particles to be spheres of equal density.

The figures then correspond to those obtained by sedimentation methods. The data and derived information is collected in the form of a table (see Table I), and by plotting the figures for "Weight per cent less than upper limit of size range" in the last column against the upper limit of size range in the first column a cumulative curve representing the size distribution is obtained, from which the weight percentage less than any given size may be read.

Sedimentation Methods

These methods depend upon the calculation of free-falling speeds, by means of Stokes' Law, of particles in different size ranges or fractions, and give analyses in terms of weight of powder in a given fraction directly. Stokes' Law may be written,

$$t_1 = (18 \times 10^8 nh)/\{(p - p_1)\, g\, d_1^2\} \quad .. \quad .. \quad .. \quad (1)$$

where t_1 is the settling time for particles, of diameter $d_1\,\mu$ and density p, to fall a height h under the influence of gravity, g, in a fluid of viscosity n and density p_1.

In practice, unless a sedimentation balance, such as the Micromerograph, is used d_1 refers not to a particle of a single definite size, but to a size range, and according to the method, may be either the top size or the mean size of a fraction. As in microscope sizing, a $\sqrt{2}$ progression of diameters is used to define successive fractions and since the time of fall is proportional to the square of the diameter this means that each settling time is double the previous one, so that Stokes' Law has to be applied only once to determine the time, t_1, to extract the initial sample.

Andreasen Pipette

The apparatus consists of a glass parallel-sided sedimentation vessel with a ground glass neck, having a graduated scale 20–0 cm. marked on the side with the zero mark about 3 cm. from the bottom. The pipette is fitted with a two-way tap and side discharge tube and has a capacity of 10 ml. It has a ground glass socket below the bulb which fits into the neck of the sedimentation vessel. The stem from the pipette bulb to the sampling inlet, which coincides with the zero mark on the sedimentation vessel when the apparatus is in use, is made of fine capillary tubing.

A 5 g. sample of phenothiazine is dispersed in a suitable quantity of a solution of a wetting agent and transferred to the sedimentation vessel. It is further diluted to the 20 cm. mark (about 550 ml. in all) with the solution of the wetting agent, *not* water, mixed by inverting the vessel several times, and allowed to settle. The limiting Stokes' diameter, d_1, for the first fraction is taken as 76 μ, that is, all particles greater than 76 μ will have had time to fall 20 cm. and none will be collected, and the time, t, to extract the first sample is obtained from equation 1. Times to extract subsequent samples are all twice the previous time interval, except that a slight correction is made for the decrease in height of the column of liquid in the sedimentation vessel as samples are removed. The 10 ml. fractions are filtered through a No. 4 sintered glass crucible, vacuum dried over P_2O_5, and weighed. Each fraction then contains a mixture of

particles all less than a given diameter in the same proportion of sizes as was originally present in the complete sample, except that the oversize has been removed completely. The calculation required to obtain the weight percentage of particles less than the upper limit of a given size range is

$$\text{Weight per cent} = 100\ w/\mathrm{W}.\ \mathrm{V}/v.$$

where w = weight of fraction in g.
W = weight of initial sample in g.
V = volume of sedimentation vessel
v = volume of pipette

Table II gives the size analysis of the phenothiazine sample obtained with the Andreasen pipette. From this it can be seen that one very practical disadvantage of aqueous sedimentation methods is that in a

TABLE II

ANDREASEN PIPETTE SIZE ANALYSIS OF FINELY GROUND PHENOTHIAZINE

Size range (μ)	Time			Weight of sample (g.)	Weight per cent less than upper limit of size range
	Hr.	Min.	Sec.		
76–53	—	4	11	0·0940	100·6
53–37·5	—	8	10	0·0918	98·3
37·5–26·5	—	15	51	0·0914	97·8
26·5–18·8	—	31	0	0·0899	96·2
18·8–13·2	1	0	20	0·0874	93·5
13·2–9·4	1	57	20	0·0805	86·1
9·4–6·6	3	44	20	0·0742	79·4
< 6·6	7	13	—	0·0610	65·3

normal working day particle sizes down to 6·6 μ only can be collected. Since according to the results quoted, 65 per cent by weight of the powder under test is smaller than 6·6 μ the analysis can hardly be said to be completed, although sufficient information has been obtained for most purposes.

For the sedimentation vessel used $\mathrm{V} = 535$ ml., $v = 10$ ml., and $\mathrm{W} = 5{\cdot}0$ g., $\therefore$ Weight per cent $= 100\ w/5{\cdot}0 \times 535/10 = 1070\ w$.

Stairmand Method

This differs from the Andreasen pipette method in that the whole of the sedimenting solid is collected in the form of fractions removed at predetermined times, by allowing the sediment to flow out at the bottom of the glass sedimentation tube, which in our case is 32 cm. long. The volume of liquid and sediment removed is replaced by fresh solution from the reservoir. The limiting Stokes' diameter, d_1, is taken to be the geometric mean of the size limits and not the upper size limit, since the whole of this fraction together with varying proportions of the other fractions is collected after the first time interval t_1. Because of this the calculation is different to that used in the Andreasen pipette method, but is fully described in Stairmand's original paper[3]. In effect the weight obtained as fraction 1 is doubled and the weight of fraction 2 subtracted from it to obtain the weight of particles in the first size grade. This procedure is followed for each grade until the last fraction is reached when the weight of this is doubled and the weight of solids still remaining in suspension (obtained

by difference from the total weight originally put in to the apparatus) added to it. The results given in Table III show how this works out in practice, and the figures for "weight per cent less than mean size" in the last column can be compared with the figures obtained by the two previous methods.

Micromerograph

The particles of dry powder are sedimented in still air down a seven foot long vertical tube and collected on the pan of an automatic servo-controlled torsion balance at the bottom of the tube. The powder is dispersed at the beginning of the experiment by blowing it into the sedimentation column through an annular slit with a known volume of dry nitrogen. Because the adhesive force between particles, and their fragility,

TABLE III

STAIRMAND SEDIMENTATION SIZE ANALYSIS OF FINELY GROUND PHENOTHIAZINE

Size range (μ)	Mean sizes	Time			Weight of sample (g.)	Weight in grade	Weight per cent in grade	Weight per cent less than mean size
		Hr.	Min.	Sec.				
104–76	89	—	3	24	0·0223	0·0102	2·04	100·00
76–53	63	—	6	48	0·0121	0·0091	1·82	97·96
53–37·5	44·5	—	13	36	0·0151	0·0106	2·12	96·14
37·5–26·5 ..	31·5	—	27	12	0·0196	0·0119	2·38	94·02
26·5–18·8 ..	22·2	—	54	24	0·0273	0·0065	1·30	91·64
18·8–13·2 ..	15·7	1	48	48	0·0481	0·0294	5·88	90·34
13·2–9·4 ..	11·1	3	37	36	0·0668	0·0708	14·16	84·46
9·4–6·6	7·9	7	15	12	0·0628	0·0733	14·66	70·30
6·6–4·7	5·6	14	30	24	0·0523	0·0707	14·14	55·64
< 4·7	3·9	29	0	48	0·0341	0·2075	41·50	41·50

varies for different powders, a wide range of shear forces can be applied by varying the nitrogen pressure and the slit width, so that the optimum dispersion conditions can be found by trial and error methods for each material.

Since the powder accumulates steadily on the balance pan the calculation is not based on the time required to extract the first sample and the equation for Stokes' Law is rearranged so that particle diameter is given as a function of settling time: $d_1^2 = (18 \times 10^8 nh)/(p - p_1)\, g\, t_1$.

The density of the fluid p_1 is so small for air that it can be neglected; the viscocity of air, n, the height of fall, h, and the acceleration due to gravity, g, are all constant so that the equation may be written, $d_1 p^{\frac{1}{2}} = \mathrm{K} t_1^{\frac{1}{2}}$.

The method requires that the total duration of the run shall be predetermined. To calculate this it is assumed that a value of $d_1 p^{\frac{1}{2}} = 2$ represents the lowest size which will have any appreciable effect on the analysis, and this enables the entire run to be completed in about $3\frac{1}{2}$ hours. After the preliminary calculations have been made the Micromerograph produces automatically a chart representing the total weight of powder collected on the balance pan as a continuous function of time and from this the analysis is derived directly by means of a template supplied by the makers. The experimental values are given in Table IV.

Surface Area Methods

The surface area of a powder can be measured by various means but the one in common use, owing to its simplicity, is air permeability, and the apparatus available can be divided into two classes.

The first one depends on a direct measurement of the pressure drop across a compressed plug of powder when air at a fixed inlet pressure is passed through it. This was developed by Carman[6] and is now sold commercially as the "Fisher Sub-Sieve Sizer". The apparatus includes a complicated chart from which a mean particle size is read directly, and

TABLE IV

MICROMEROGRAPH SIZE ANALYSIS OF FINELY GROUND PHENOTHIAZINE

Pressure 150 psi. Slit width 150 μ

$dp^{\frac{1}{2}}$	$d(\mu)$	Recorder chart reading	Weight per cent less than d
66	56·5	0	100
60	51·5	0·2	99·3
54	45·3	0·2	99·3
48	41·2	0·2	99·3
42	36·0	0·3	99·0
36	30·9	0·3	99·0
30	25·8	0·3	99·0
27	23·2	0·4	98·7
24	20·6	0·6	98·0
21	18·0	1·5	95·1
20	17·2	1·9	93·8
19	16·3	2·3	92·5
18	15·5	3·0	90·2
17	14·6	3·9	87·2
16	13·7	5·0	83·6
15·1	13·0	6·0	80·3
14	12·0	7·8	74·4
12	10·3	11·9	61·0
10	8·6	16·2	46·9
8	6·9	20·7	32·2
6	5·2	25·7	17·0
5	4·3	27·3	10·7
4	3·4	29·0	5·0
3	2·6	30·0	1·8
2·5	2·1	30·3	0·8
2	1·7	30·6	0

results are, therefore, obtained in terms of a mean particle size derived from what is actually a surface area measurement. The implications of this are discussed later.

The second variation depends on the time taken for oil in a manometer tube to approach equilibrium by flowing under gravity between two fixed levels, the air so displaced being forced through a compressed plug of the powder. This was developed by Rigden[8].

The basic equation for the calculation of specific surface which applies to both variations, was evolved by Kozeny from Poiseuille's Law, and may be written, $S^2 = \frac{A}{knLp^2} \cdot \frac{e^3}{(1-e)^2} \cdot \frac{\Delta P}{V}$.

where S = specific surface area in cm.^{2}/g. A = area of cross section of the plug of powder in cm.^{2} L = length of plug of powder in cm. n = viscosity of air = $1{\cdot}81 \times 10^{-4}$ poises at room temperature. p = density of powder = 1·36 for phenothiazine. k = Kozeny's constant = 5·0. e = Porosity factor or void volume = volume of air in 1 g. of compressed

powder. ΔP = pressure drop across the plug, and V = velocity of air through the plug in cm./sec.

The porosity factor is defined as $e = (AL - W/P)/AL$ where W is the weight of sample in g. The calculation may be simplified if the length of the plug, L, is kept constant and the weight of sample is numerically equal to the density of the sample. This is the recommended method of using the Fisher Sub-Sieve Sizer, since the only variables are then ΔP and V which are readily measured by means of a water flowmeter. Rigden prefers the plug of powder to be packed using as far as possible the same pressure, so that L and e vary with each sample. This makes the calculation slightly more lengthy but is more accurate. The calculation of $\Delta P/V$ is mathematically more difficult with the Rigden apparatus since the air pressure drops steadily throughout the experiment, but this is largely an instrumental factor and once it has been determined the only experimental data required is the time taken for oil to flow between two fixed points in a manometer.

The results on the sample of phenothiazine are as follows:

Fisher Sub-Sieve Sizer:	Mean size 2·4 μ
	Equivalent to a surface area of 18,400 cm.^{2}/g.
Rigden Apparatus:	Surface area 14,400 cm.^{2}/g.

There is a fixed difference between these two pieces of apparatus, the Fisher giving results about 25 per cent higher than the Rigden, which is probably caused by inaccuracies in determining the instrumental constants. Since it is not known which is the more correct no attempt has been made to alter the constants of either apparatus.

Discussion

The methods described are all in use throughout the world for the size analysis of phenothiazine, but where results are quoted in the manufacturers' literature insufficient attention is paid to the differences which arise from the use of different methods. The magnitude of these differences can be seen if figures for the percentages of particles less than 30 μ and less than 10 μ, obtained by the different methods on the same sample of phenothiazine are taken from Figure 1, as follows.

	$<30\,\mu$	$<10\,\mu$
Microscope	98·4	55 per cent
Andreasen pipette	97·4	80 per cent
Stairmand	97·4	78 per cent
Micromerograph	98·8	60 per cent

Each of these methods give reproducible results, but it is practically impossible to determine which, if any, of them is correct. Obviously, if every manufacturer of phenothiazine used the same method of analysis this would not be necessary, but since, so far, they do not, it is desirable to have some idea of the advantages and disadvantages of each method.

The two wet sedimentation methods require very little special apparatus beyond that normally present in a chemical laboratory and are, therefore, popular on the grounds of economy. They are, however, the most time-consuming and probably the least accurate since flocculation of the finest particles occurs if the settling time is prolonged, and Brownian movement of particles less than 2 μ completely upsets the Stokes' Law relationship. The turbulence which exists immediately after the contents of the tube have been mixed makes the zero time difficult to interpret and can cause errors in the estimation of the first few samples. Inspection of Figure 1 shows that the results obtained in the highest fractions do not

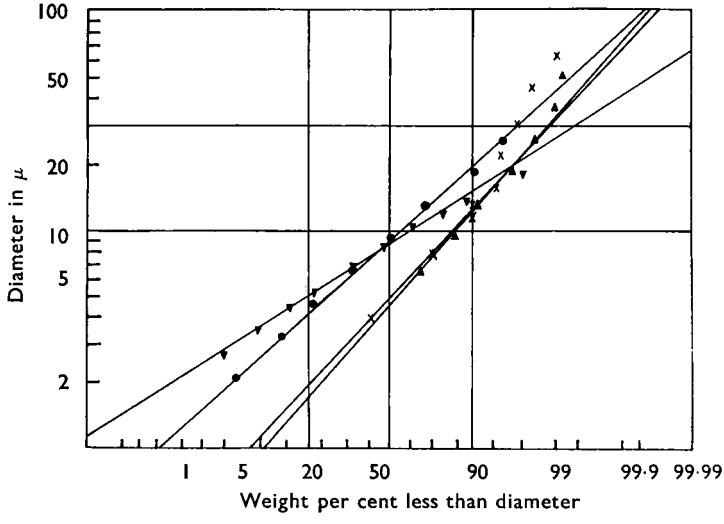

FIG. 1. Logarithmic probability graph of the particle size distributions of a phenothiazine sample by four different methods.

▼—▼ Micromerograph. ×—× Stairmand.
●—● Microscope. ▲—▲ Andreasen.

fit the lines drawn to represent the distributions. Apart from such inaccuracies the time factor is important because a complete experiment cannot be fitted into a normal working day if sizes less than 6 to 7 μ require estimation. The results obtained by these two methods are similar, as would be expected, the chief difference being that since the Stairmand apparatus requires more dilute suspensions the particles should obey Stokes' Law more closely and therefore a more accurate result should be obtained. On the other hand, the Andreasen pipette is the only method of those considered where only one sample needs to be taken at a pre-determined time to produce a percentage figure less than any required size, without the necessity for determining a complete analysis. This simplified treatment makes the method much more attractive since it eliminates the inaccuracies at both ends of the distribution and yields all the information required for routine control, if two samples, corresponding to sizes less than, say, 30 μ and 10 μ are taken.

The microscope counting method is perhaps the most well known of all methods of sizing particles, but is often avoided as being very tedious for routine measurement and liable to produce eye-strain in the operator. Using a projection microscope and an experienced operator, however, complete analyses can be produced with a high degree of reproducibility within two hours. Where results are required in a hurry this is an obvious advantage over wet sedimentation methods. The statistical necessity of obtaining a reasonable number of the largest particles when counting places a restriction on the top limit of particles sized and it may be that

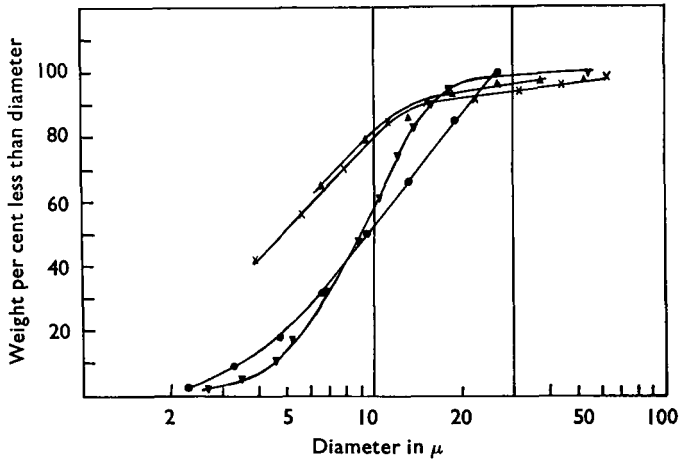

FIG. 2. Cumulative plot of the particle size distributions of a phenothiazine sample by four different methods.

▼—▼ Micromerograph. ×—× Stairmand.
●—● Microscope. ▲—▲ Andreasen.

one or two of the particles included in the top grade are bigger than the upper limit. For this reason the size corresponding to 100 per cent less than the upper limit of size range is always smaller than that obtained by sedimentation methods (see Fig. 2). In producing a weight analysis from a count of the number of particles it is usually assumed that all particles are spherical. The larger particles of phenothiazine may, however, deviate considerably from sphericity since the crystals are flaky, which means that the large particle end of a distribution becomes over-emphasised so that in this region a microscope analysis gives higher results than other methods. Stokes' Law as applied to sedimentation methods also assumes that the particles are spherical but small deviations from sphericity have a negligible effect on free-falling speed.

The Micromerograph is a very expensive piece of apparatus and one not likely to be bought by anyone whose sizing problems are with phenothiazine only, unless his manufacture is on a very large scale. Once installed, however, it is a useful instrument which gives reproducible results with a minimum of attention and within a few hours. It is the only one of the four instruments considered, whose use is restricted to dry

powders, so that ready-made aqueous suspensions or drenches cannot be analysed. There is some doubt whether complete dispersion without grinding is achieved; certainly results on phenothiazine always show a closer-sized distribution than any other method which could mean that some grinding of the large particles occurs when the material is blown into the sedimentation chamber whilst the smallest particles are not completely dispersed and fall as small aggregates. The agreement between the microscope and Micromerograph analysis in the middle ranges of the distribution is reasonably good.

Surface area measurements can be compared only with particle size distributions if some assumptions are made about the type of distribution usually encountered. It is generally accepted[9] that size analyses of ground powders, where the material is the product of a single grinding operation and has not been obtained by mixing products ground to different degrees, obey at least approximately a logarithmic probability law. For this reason one of the most popular ways of plotting the results of a size analysis is as a cumulative graph of weight per cent less than a given diameter on an arithmetic co-ordinate, against the given diameter on a logarithmic co-ordinate. This gives a symmetrical S-shaped curve, and Figure 2 shows the results quoted in Tables I to IV plotted in this way. Particle size requirements for phenothiazine are usually requested as a certain percentage less than a given size and from this graph such information can be read.

The statistical equation for a logarithmic normal distribution may be written, $df/\mathrm{N} = 1/\sqrt{2\pi} ln\,\alpha \exp.\,[-(ln\mathrm{D}-ln\mathrm{M})^2/2ln^2\,\alpha]\,\alpha ln\mathrm{D}$, when f is the frequency with which a particle of diameter D occurs in a number of particles, N. M is the geometric mean diameter and α the geometric standard deviation of the distribution. The equation relates the fractional numbers of particles in each size grade (f/N) to the logarithm of the diameter of the size grade (lnD) and represents a non-cumulative distribution completely defined in terms of two parameters M and α. The standard deviation, α, and the weight mean diameter, d_w, are obtainable by the application of the above equation to a plot of the results of particle size analyses on logarithmic probability paper (see Fig. 1) and it only remains to relate d_w with the surface mean diameter d_s to calculate the surface area figure.

Referring to Figure 1 the weight mean diameter is the diameter corresponding to the 50 per cent figure on the probability co-ordinate, and the standard deviation is the diameter corresponding to the 84·13 per cent figure divided by the 50 per cent figure. These two parameters can thus be determined directly from the graph for all four size distributions. To use surface area measurements a surface mean diameter, d_s, must be defined. This can be done by means of two of the Hatch-Choate[10] equations, $\log d_s = \log \mathrm{M} + 5{\cdot}757 \log^2 \alpha$, and $\log d_w = \log \mathrm{M} + 8{\cdot}059 \log^2 \alpha$; log M, whose value is unknown, can be eliminated from these two equations, and log d_s related directly to low d_w, as $\log d_s = 2 \log d_w - 5{\cdot}302 \log^2 \alpha$. If it is assumed that the particles are spheres then the relationship between the surface mean diameter, in microns, and the

specific surface area in cm.²/g. is given by, $S_w = 6 \times 10^4/p.d_s$ where p is the density of phenothiazine (1·36 g./cm.³).

The derived values for the standard deviation, the weight mean diameter, the surface mean diameter, and the surface area of the four size distributions are given in Table V, together with the air permeability values, obtained from the Sub-Sieve Sizer and the Rigden apparatus, for surface area and surface mean diameter.

It must be realised that the data for the Stairmand and Andreasen sedimentation methods is incomplete and the extrapolation required to produce the straight line on the logarithmic probability graph may have introduced serious errors.

This argument shows how a size analysis, provided it follows the log probability law may be equated to a surface area value but it must be realised that the converse does not hold. A surface area measurement

TABLE V

SURFACE AREA VALUES FOR FINELY GROUND PHENOTHIAZINE

Method	$d_w(\mu)$	α	$d_s(\mu)$	S_w (cm²/g.)
Micromerograph	8·75	1·72	6·52	6,800
Microscope	9·2	2·17	5·05	8,750
Stairmand	4·9	2·45	2·20	20,100
Andreasen	4·6	2·62	1·82	24,200
Sub-Sieve Sizer	—	—	2·4	18,400
Rigden	—	—	3·3	13,400

can relate to any number of distributions and therefore such a measurement cannot be used to define a distribution completely. Moreover it can be applied only to dry powders and the method, like the Micromerograph method, cannot be applied to suspensions in water. Another disadvantage is that the effect of wetting agents which are present in dispersible powders, even in quantities as low as 0·5 per cent, materially affect the surface area figures obtained and comparison of samples of phenothiazine dispersible powder whose origin and formulation is unknown, cannot be attempted. The only worth-while application of surface area measurements is, therefore, as a routine check of the grinding efficiency of a mill, for which purpose they are ideally suited.

Some attempts have been made, both in this country and in South Africa and New Zealand, to define a method for the standardisation of particle size analysis of phenothiazine. The generally preferred method, mainly on the grounds of cheapness and availability of apparatus than on inherent accuracy, is the Andreasen pipette.

Acknowledgement. I am indebted to Mr. M. W. Vincent of Sharples Super Centrifuges Limited, for the Micromerograph analysis.

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After Mr. Thornton presented the paper there was a DISCUSSION. The following points were made.

All the methods referred to by the author were reliable. The Andreasen pipette was probably the most widely used. The author preferred the microscopical method, but the use of a haemocytometer was not satisfactory, as the counting chamber was too deep. Self-attrition, a problem encountered with a centrifugal classifier, was also a failing of the micromerograph. Anomalous results with blended samples of phenothiazine had not been encountered by the author. Flocculation sometimes caused difficulties in wet sedimentation methods, and the choice of wetting agent was important.