An automated sedimentation balance

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A method of size grading powders using an automated sedimentation balance is described. The results are recorded as percentages of the total powder present in 1 or 5 μm (intervals) of particle size, after they have been multiplied by a simple correction factor. Analyses of calcium carbonate powder using this balance and the Gallenkamp balance have been compared.

The Gallenkamp torsion balance, described by Bostock (1952), is frequently used to size grade powders. In using it a number of unjustifiable assumptions are made. These are:

(1) The powders fall under true Stokesian conditions. That is there is no inter-action between individual particles, no turbulence is produced and wall effects are negligible.

(2) The weights of powders which settle on the pan within the first minute can be accurately recorded.

(3) No powder spills from the pan during the course of the experiment.

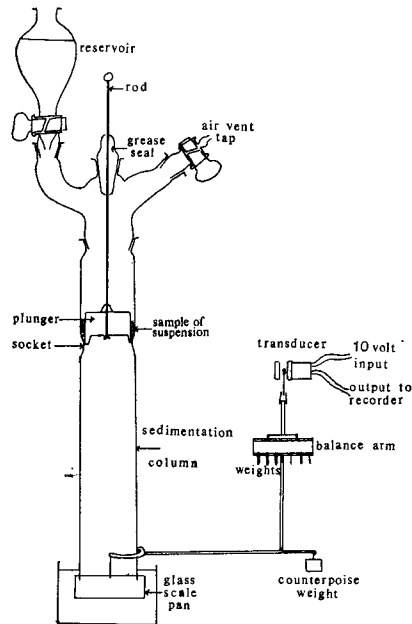Because of these faults, the apparatus illustrated in Fig. 1 was designed.

FIG. 1. Sedimentation balance.

Apparatus

The automated sedimentation balance consists of a glass column containing an annular constriction 25 cm from the lower end. A PTFE plunger fits into this constriction to make an air-tight seal. A rod, attached to the plunger, passes through an air-lock at the top of the assembly. This enables the plunger to be raised and lowered. The top section also incorporates (a) a reservoir containing a solution of the dispersing agent used to fill the lower section of the column and (b) a side tube with a tap that allows the displaced air to escape and also enables the sample to be introduced into the top section of the column.

At the foot of the column is a vessel containing more of the solution of dispersing agent. A glass scale pan is suspended in this solution and is attached by an arm, holding a counterpoise weight, to a single pan balance.

A transducer core is also attached to the arm of the balance, from above, by means of a rod. The core is sited within a transducer, which is connected to a 10 V input supply. The output from the transducer is fed into a variable-speed chart recorder. In this way, movements of the balance arm, which relate to weights of powder falling on the scale pan, are automatically recorded.

Filling of sedimentation column

The column, which is attached to a rack and pinion assembly, is racked down until its lower end is immersed in the solution, and the height between the constriction in the tube (socket) and the scale pan is 26·5 cm. Then the plunger is pressed down into the socket using the rod. The air vent and reservoir taps are opened, so that the solution flows into the upper section of the column. When a suitable volume is present in the upper section, the two taps are closed and the plunger lifted allowing the solution to fall into the lower section. The plunger is pressed down again and the process repeated, until the lower section is filled with solution to just above the constriction (socket). Then the plunger is raised a little and air bled in through the air vent tap, until the level of the solution has fallen to the base of the plunger.

Checks on scale pan

Once the sedimentation column is full, the balance weights are altered until the scale pan swings freely. Small weights are added to the counterpoise until the balance scale has moved to a position near to 100 mg, for example 100·4 mg. Then 100 mg is removed from the balance arm, so that the scale moves towards the 0 mg position.

When the scale indicates 100 mg, the scale pan is deeper in the liquid than it is when the balance scale is at the 0 mg position. Thus, in the example quoted, some of the wire supporting the scale pan will be above the liquid surface when the scale is near to 0 mg, but below the surface when the scale registers 100·4 mg. Consequently, when the 100 mg weight is removed from the balance arm, the scale moves from the 100·4 mg position to the x mg position and not the 0·4 mg position which would be obtained if the pan were suspended in air. This x mg difference in weight is caused by the buoyancy of the wire in the liquid. Therefore, in order to make precise measurements, all scale readings are multiplied by the factor $(100 + x)/100\cdot4$.

When the scale pan is swinging freely the same correction factor will be obtained each time the apparatus is used.

Particle size analyses obtained from this balance were compared with analyses from the Gallenkamp balance.

METHODOLOGY

Automated sedimentation balance

Introduction of sample and recording of weights. 1 g of calcium carbonate was dispersed in 24·63 cm³ filtered 0·1 % sodium pyrophosphate solution, saturated with calcium carbonate. This produced a 1 in 25 (i.e. 4%) suspension of calcium carbonate. It was prepared in a glass mortar and stirred with a spatula to produce maximal dispersion with minimal grinding of the powder. Some agglomeration of the powder particles took place within 2 min of preparation consequently the sample was taken as soon as dispersion was completed. The sample was drawn into a syringe to which a length of fine-bore silicone rubber tubing was attached.

With the plunger pressed down firmly into the socket, the air vent tap was removed and the tubing fed into the top section alongside the plunger. Then 4 cm³ of the suspension was delivered alongside the plunger, after which the syringe plus tubing was withdrawn. The air vent tap was replaced and closed. The rod was then raised lifting the plunger. At the same time a stop watch was started and the chart recorder switched on at a speed of 1 cm s⁻¹. (Transfer of the suspension and the subsequent raising of the plunger were carried out rapidly to prevent agglomeration of the particles in the suspension.)

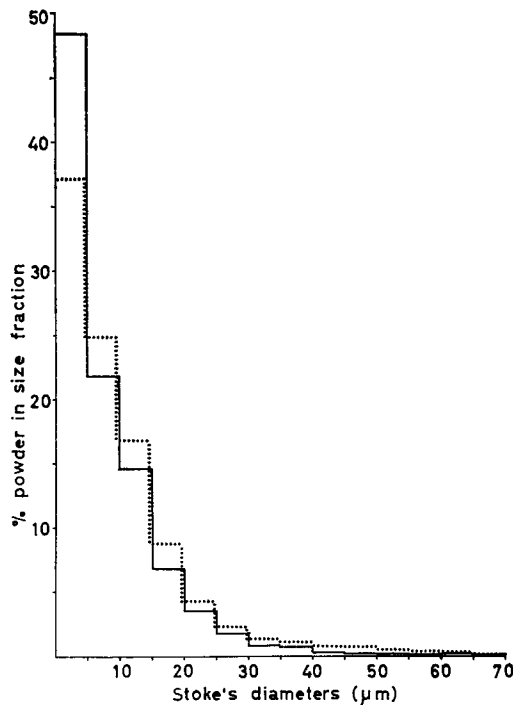When the first particles settled on the scale pan the time was noted. Weights of

FIG. 2. Size distribution of particles in calcium carbonate powder obtained from sedimentation balance readings. —— Gallenkamp balance; · · · · automated sedimentation balance.

powder settling on the pan were then recorded at the times obtained from the print out of a computer program, which was based on Stoke's equation:

$$t = \frac{18\,h\eta}{d^2(\rho_1 - \rho_2)g} \qquad .. \qquad .. \qquad .. \qquad .. \qquad (1)$$

where t is the time (s) at which the weight on the pan should be recorded, h is the height of fall ($= 26\cdot5$ cm), η is the viscosity of the dispersing fluid in poises, d is the diameter of the particles in μm, ρ_1 and ρ_2 are the densities of the solid ($2\cdot7$ g cm^{-3}) and liquid ($1\cdot0$ g cm^{-3}) respectively and g is the gravitational constant. When there there was insufficient time to record the weights at the appropriate times, these were obtained from the print out of the chart recorder.

Gallenkamp balance

The method of introducing a suspension of the powder directly onto the pan of the Gallenkamp balance is fraught with inaccuracies, some of which have been listed. In an attempt to reduce these errors and enable direct comparisons to be made, a two-layer technique was used.

The scale pan attached to the torsion wire was immersed in $0\cdot1\%$ sodium pyro-phosphate solution and the balance adjusted, so that a full scale deflection was obtained when a 500 mg weight rested on the pan. 100 mg weights were then added to the empty pan and the scale reading was noted after each addition. Each 100 mg weight weighed $87\cdot5$ mg in $0\cdot1\%$ sodium pyrophosphate solution and gave a scale deflection of $20\cdot5$ divisions, when it was added to the pan. When 4×100 mg weights were present on the pan, addition of a fifth 100 mg weight gave a smaller deflection. Therefore this section of the calibration curve was plotted precisely by the addition of 10 mg weights to the pan.

The sedimentation column was then filled with $0\cdot1\%$ sodium pyrophosphate solution (saturated with calcium carbonate) to $26\cdot0$ cm above the scale pan. Calcium carbonate $0\cdot8$ g, was dispersed in 25 cm^3 of the same solution and immediately introduced into the top of the column through the upper vessel. At the same time a stop watch was started. The suspension raised the liquid level to $26\cdot5$ cm above the pan. Scale readings were recorded at the times indicated in Table 2.

RESULTS

Automated sedimentation balance

A reading of $100\cdot4$ mg read at the top end of the scale was equivalent to $106\cdot4$ mg at the other end. Similarly a reading of $98\cdot4$ mg at the top end of the scale was equivalent to a reading of $104\cdot3$ mg at the bottom end of the scale. In either case the correction factor for the buoyancy of the wires supporting the scale pan was

$$\frac{106\cdot4}{100\cdot4} = \frac{104\cdot3}{98\cdot4} = 1\cdot06.$$

The weight of powder present in 4 cm^3 of a 4% suspension of calcium carbonate is 160 mg. This weighs 100 mg in $0\cdot1\%$ sodium pyrophosphate solution. Hence, when all the weights had been multiplied by the correction factor ($1\cdot06$), they were recorded as percentages. The weights of 5 μm ranges of particle diameters were obtained by subtraction.

The average percentage weights of 11 replicates for each size range are given in Table 1 with their standard deviations and coefficients of variation.

Gallenkamp balance

Calcium carbonate, 800 mg, weighs 500 mg in 0·1% sodium pyrophosphate solution. When the scale readings had been converted to weights, they were divided by 5 to give percentages.

Table 1. *Automated sedimentation balance analysis of 160 mg calcium carbonate powder suspended in 0·1% sodium pyrophosphate solution, saturated with calcium carbonate. (11 replicate determinations).*

Times from introduction of sample (s)	Stokes' diameters (μm)	Balance scale weights (mg)	Weights corrected for buoyancy i.e. × 1·06 (mg)	Percentage (weight) differences W (%)	Standard deviation s (%)	Coefficient of variation $\frac{s}{W} \times 100$ (%)
58·4	70	2·97	3·16	—	—	—
67·7	65	3·13	3·32	0·16	0·09	56·2
79·4	60	3·48	3·69	0·37	0·11	29·7
94·5	55	3·9	4·13	0·44	0·16	36·4
114·4	50	4·39	4·65	0·52	0·23	44·2
141·2	45	5·07	5·37	0·72	0·22	30·5
178·8	40	5·84	6·19	0·82	0·31	37·8
233·5	35	6·87	7·28	1·09	0·23	21·1
317·8	30	8·19	8·68	1·4	0·39	27·8
457·6	25	10·47	11·1	2·4	0·58	24·2
715·0	20	14·5	15·4	4·3	0·37	8·6
1271	15	22·7	24·1	8·7	1·69	19·4
2860	10	38·6	40·9	16·8	2·69	16·0
11 440	5	62·1	65·8	24·9	2·39	9·6
∞	0	—	103·2	37·4	5·39	14·0

Table 2. *Gallenkamp (Bostock) balance analysis of 800 mg calcium carbonate powder suspended in 0·1% sodium pyrophosphate, saturated with calcium carbonate. (11 replicate determinations).*

Times from introduction of sample (s)	Stokes' diameters (μm)	Percentage (weight)* differences (%)	Standard deviation (%)	Coefficient of variation (% standard deviation) (%)
39·6	85	0·19	0·092	48·4
44·7	80	0·12	0·081	67·7
50·8	75	0·16	0·064	40·0
58·4	70	0·15	0·089	59·3
67·7	65	0·17	0·110	64·7
79·4	60	0·24	0·102	42·5
94·5	55	0·22	0·11	50·0
114·4	50	0·24	0·13	54·2
141·2	45	0·32	0·08	25·0
178·8	40	0·40	0·17	42·5
233·5	35	0·64	0·12	18·7
317·8	30	0·86	0·22	25·6
457·6	25	1·70	1·03	60·0
715·0	20	3·04	0·76	24·6
1271	15	6·87	2·4	34·8
2860	10	14·6	3·3	23·3
11 440	5	21·8	7·5	34·4
∞	0	47·0	16·5	35·1

* Obtained by converting scale readings to mg weights then dividing by 5.

The average percentage weights of 11 replicates for each size range, obtained from the Gallenkamp balance readings, are given in Table 2, together with their standard deviations and coefficients of variation. Plots were made of logarithms of the diameters (μm) against probabilities of the cumulative percentage oversize for each of the 22 analyses. The geometric mean diameters (M) in μm were the diameters above which 50% of the particles were present. The geometric standard deviations (σ) were obtained from the equation given by Ames, Irani & Callas (1959):

$$\sigma = \frac{\text{diameter at } 15 \cdot 87\% \text{ cumulative oversize}}{M}$$

$$= \frac{M}{\text{diameter at } 84 \cdot 13\% \text{ cumulative oversize}} \qquad .. \qquad .. \qquad (2)$$

For the Gallenkamp balance M = 5·9 μm, σ = 2·53 (average of 11 values) range 4·4–6·7 μm.

For the automated balance M = 7·18 μm, σ = 2·50 (average of 11 values) range 6·5–7·7 μm.

DISCUSSION

The geometric mean diameters, obtained from the automated balance results, show less variation about their mean of 7·18 μm, than the diameters, obtained from the Gallenkamp balance results, show about their mean of 5·9 μm. Furthermore, more of the coefficients of variation for each size range are lower for the automated balance results than for the Gallenkamp balance results. This indicates that of the two balances the former gives the more accurate results.

The percentage weights in the 0–5 μm range for each balance were obtained by subtracting the cumulative weights up to 5 μm particle diameter from 100%. If any losses occur, these will result in a low value for the cumulative percentage weights up to 5 μm particle diameter and a corresponding high value for the 0–5 μm particle diameter and a corresponding high value for the 0–5 μm range. This will lower the value of the geometric mean diameter.

I have found that some 3·9% of the total powder was *not* delivered to the upper surface of the liquid in the sedimentation column of the Gallenkamp balance. This agrees with Leschonski & Alex (1970), who found that 1·5–4·0% of the suspension remained in the upper vessel. They also found that 6·5% error was caused by losses from the balance pan. Allen (1974) suggests that pan losses are about 8%.

A value of 47% was obtained for the 0–5 μm range from the Gallenkamp balance results, compared with a value of 36·5% for the same range from the automated balance results. The difference of 10·5% agrees with the total losses which Leschonski & Alex stated could take place. This indicates that the automated balance values are probably nearer to the true values.

Further support for this comes from Ames & others (1959), who found that the geometric mean diameters for various calcium phosphates obtained from analyses with an automated Gallenkamp balance were, with one exception, 2–11% lower than those obtained from a flying spot particle resolver. The fact that no particles larger than 70 μm were obtained with the automated balance indicates that slightly less agglomeration of particles took place than in the Gallenkamp balance, which recorded the presence of 85 μm particles. In either case suspensions were transferred to the balances as soon as they were prepared, to minimize agglomeration.

After one of the experiments, the complete sedimentation column of the automated balance, including the plunger, was washed with hydrochloric acid. This was boiled and back titrated against potassium hydroxide solution. It was calculated that only 0·4 mg calcium carbonate had been left in the column. Thus, there was less than 0·4% error caused by non-release of the suspension into the column of the automated balance, compared with a 3·9% error in the Gallenkamp balance.

The pan used in the automated balance is made of glass and 3·0 cm deep, this ensures that powder is not lost from it during an experiment, while the "pumping action" which occurs when a suspension is introduced directly onto the pan of the Gallenkamp balance results in the losses already mentioned. Glass was used in preference to metal because it does not corrode and has a smoother surface. Therefore there is less chance of small bubbles attaching themselves to the irregular surface to produce another source of error. The balance pan used in the Leschonski (1962) balance is shallow and slight losses of powder occur. The pan must also be connected to a self-compensating balance which makes the complete instrument expensive.

CONCLUSION

Thus the automated sedimentation balance enables reasonably accurate size grading of a powder to be achieved.

The advantages of the apparatus are:

(1) Size grading of particles up to 100 μm can be achieved in a single experiment.
(2) Interparticulate impedence of fall is minimal, because the largest particles fall through a clear liquid onto the balance pan, once the sample has been introduced into the top of the column of liquid.
(3) Powder is NOT lost from the scale pan during the experiment.
(4) The balance records *weights* of powder settling on it, therefore no mathematical treatments or assays are required to derive weights.
(5) The chart recorder enables accurate measurements of the weights of powder, falling on the scale pan at short time intervals, to be obtained.
(6) The times at which readings are taken are arranged so that the weights of either 1 or 5 μm size fractions of the powder are obtained. These times are obtained from the print out of a simple computer program.
(7) The instrument can be left recording overnight, if weights at inconvenient times are required.
(8) No sticking of the pan occurs.

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