# Evaluation Method for Agglomerating Properties of Granular Detergents

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## Abstract

A new method for evaluating the agglomerating property of granular detergents was devised. It is useful for comparing commercial products, screening new products, and for studying anti-caking agents. This method differs from prior methods, as detergent is fluidized by conditioned air in a reverse conical tower, and agglomeration is evaluated from the lowering of level of fluidized particles because of agglomeration. The height of the fluidized particles is inversely proportional to particle size, and the level of fluidized particles becomes lower more rapidly with ease of agglomeration. The coefficient of variation ($\sigma/\bar{x}$) of the method is about 10%, and it is shown that the method coincides with the tendency of detergents to agglomerate in the carton.


## Introduction

THE PHENOMENON OF CAKING is well known in powder handling, but few suitable methods for evaluating it exist. Present methods for evaluating agglomerating properties of powders may be classified as follows: 1) In powders which absorb moisture and agglomerate to some extent but remain fluid, the fluidity is measured by means of so-called "slide angles," such as angle of repose, angle of slide, angle of internal friction, and angle of surface motion (1–8). Also, in particles with low fluidity, those which have low surface-adherence, such as glass beads, are added to increase fluidity of the original powders (9–11). In some cases the time required to flow through a small hole is measured (12). 2) For powders which partially agglomerate because of moisture adsorption, the percentage of powder agglomerated may be measured (13–14). 3) For powders which have caked completely, the compressive strength necessary to break the cake may be measured (15–21). 4) Hygroscopicity, which may be related to agglomeration, is measured, for instance, by measuring the critical relative humidity or water vapor pressure in equilibrium with powder (22–26).

However these methods have several defects. Method 1 evaluates fluidity and therefore does not indicate agglomeration in its true sense. Also, accuracy is poor, and a constant temperature and humidity chamber is necessary to keep the powder in the same condition between preparation and measurement. Method 2 may appear to be more convenient, but there is some danger that the lumps will break down during screening if the powder agglomerates loosely. In Method 3 hardness would be considered as a caking property, and further a storage period of a few days to a few weeks should be required for suitable measurement in many cases. In Method 4 hygroscopicity is measured, and although it is possible to know the extent of moisture absorption under a specific condition, it is difficult to correlate the condition at that time to the agglomerating property. Furthermore, since the agglomerating property of powder as produced is affected by particle size distribution, shape, and condition of surface in addition to composition, this method is not entirely suitable for evaluation. In this study an attempt was made to evaluate agglomeration by measuring the lowering of the particle level in a reverse conical tower, fluidized by introducing moisture-conditioned air.

## Theory

Generally, since a powder has a size distribution range, smaller particles may be scattered outside the tower under conditions in which larger particles are suitably fluidized, and larger particles may not be suspended under conditions in which smaller particles are fluidized adequately. Uniform fluidization of all particles can be accomplished by using a reverse conical tower with a suitable angle and height. All particles absorb moisture and agglomerate uniformly if high-humidity air is introduced into the tower, and agglomeration can be evaluated from the reduction in level of the fluidized particles according to the following theory.

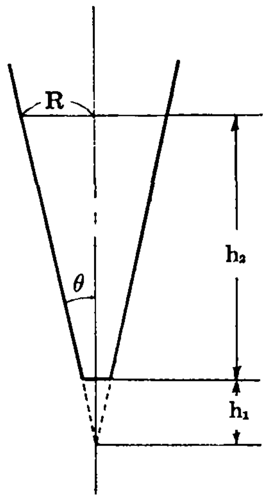


FIG. 1. Shape of tower.

When the conditioned air is introduced from the bottom toward the top at a constant volume, V, into this tower, which has a vertical angle $2\theta$ as shown in Figure 1, the upward force, F, which affects spherical particles having outer radius, $r_1$, and inner radius, $r_2$, and density, d, can be expressed by the following equation on the basis of Stokes' law:

$$F = 6\pi r_1 \eta u \quad [1]$$

where $\eta$: air viscosity at that condition

u: upward air velocity in a section where the radius of tower is $R = (h_1 + h_2) \tan\theta$

On the other hand, gravity which affects downward movement can be expressed by the following equation:

$$F' = m \cdot g = (4/3)\, \pi\, (r_1^3 - r_2^3)\, d \cdot g \quad [2]$$

where m: mass of particle

g: acceleration of gravity

Both forces should be balanced in order to fluidize particles at this position. Air velocity at that condition is expressed by the following equation:

$$u = m \cdot g/6\pi r_1\eta = (4/3)\ \pi\ (r_1^3 - r_2^3)\ d \cdot g/6\pi r_1\eta \quad [3]$$

On the other hand, air velocity is also expressed by the following equation:

$$u = V/\pi R^2 = V/\pi(h_1 + h_2)^2 \tan^2\theta \quad [4]$$

From Equations (3) and (4),

$$(h_1 + h_2)^2 = 9\ r_1\eta V/2\pi(r_1^3 - r_2^3)\ d \cdot g \cdot \tan^2\theta \quad [5]$$

$$9\eta V/2\pi g \tan^2\theta = \text{constant} = k^2$$

$$h_1 + h_2 = k \sqrt{r_1/(r_1^3 - r_2^3)\ d} \quad [6]$$

Generally granular detergents are hollow and have a value larger than zero for $r_2$, but since only the outer particle radius is considered in this case, Equation (6) is simplified in Equation (7) at apparent density d′,

$$h_1 + h_2 = k/\sqrt{r_1^2 d'} = k\ r_1^{-1}\ d'^{-1/2} \quad [7]$$

that is, the height of fluidized particles is a function of $r_1$ and d′. If the humidity of air being sent upward is high, particles are moistened and then agglomerate during fluidization. For simplification, if the shape factor and porosity attributable to agglomeration and variation of density are ignored, the height of fluidized particles lowers with the increase in particle radius because of agglomeration, and the height of fluidized particles lowers faster for a detergent which has higher agglomeration because of moisture. Therefore agglomeration can be evaluated by comparison of the time required for fall under a fixed condition.

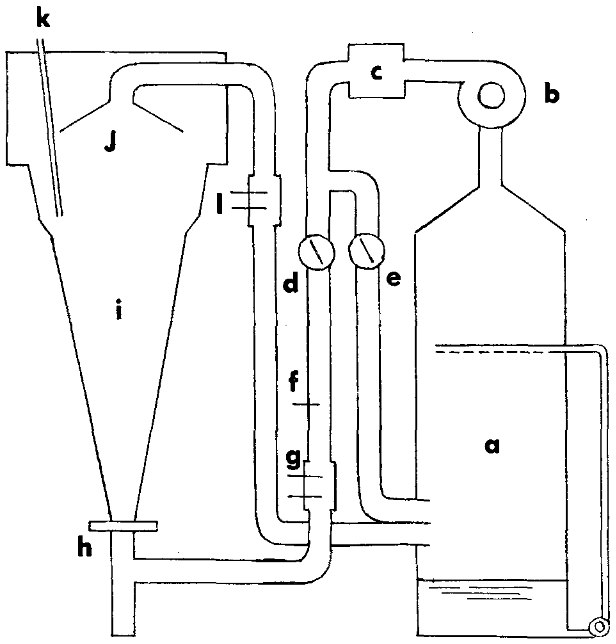


FIG. 2. Outline of equipment; *a,* dew point chamber; *b,* blower; *c,* heater; *d,* damper; *e,* damper; *f,* thermister anemometer; *g,* thermometer and hygrometer; *h,* entrance of air; *i,* tower; *j,* outlet of air; *k,* entrance of sample; *l,* thermometer and hygrometer.

## Apparatus

Figure 2 shows an outline of the equipment. The main parts are composed of a reverse conical tower for fluidizing particles and anapparatus for preparing conditioned air, which is saturated by water vapor in the dew-point chamber (a), warmed by the heater (c) to the designated temperature, and is introduced into the tower from the lower end (h), and returns to (a) through outlet (j). The volume of air which is introduced into the tower is controlled by the blower speed (b) and dampers in the main line (d) and the by-pass (e). The temperature and humidity of this air are controlled by the heater and the water-flash temperature in the dew-point chamber. The tower is made of plastic resin, which has sufficient transparency to permit internal observation, and is provided with a jacket by which the interior is maintained at a designated temperature. The sample is put into the tower from the hole (k) in the upper part of the tower and is fluidized by circulation of conditioned air. At the conditioned-air entrance in the lower end of the tower (h) an apparatus, used for removing agglomerated particles, is composed of a thin metal plate with a hole covered with wire netting at one side and an uncovered hole at the other as shown in Figure 3. This plate is set with the wire netting hole in place during measurement and, after that, the uncovered hole is set in position in order to remove the agglomerated particles. The temperature of the circulating water for maintaining the temperature inside the tower is automatically controlled. The temperature and humidity of air are measured at the tower entrance and outlet with resistance thermometers, and the water temperature in the dew-point chamber and air temperature are adjusted to the designated condition by connection with a controlling system. A thermistor anemometer is installed at the position (f) in the main circuit, and the air velocity at the entrance of tower is thus measured. The rotational speed of the blower can be varied by means of a stepless reduction system. In order to protect the equipment from corrosion by contact with high-humidity air, all parts of this equipment, with the exception of the tower, are of noncorrosive metal, and insulation is applied to prevent heat loss.

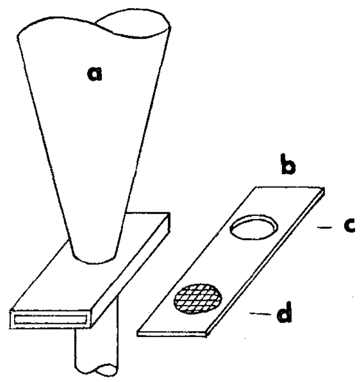


FIG. 3. Outline of lower end of tower; *a,* tower; *b,* insert plate; *c,* uncovered hole; *d,* wire melting hole.

Figure 4 shows the tower part of a practical design and the condition of fluidization. The tower has a 16° vertical angle, height of 150 cm, and entrance diameter of 7 cm.

## Experimental Section and Discussion

Agglomerating properties of various detergents were measured with this equipment, and the agglomerating properties in the carton were observed. Comparison of both results is shown below.

1. Comparison of agglomerating properties of heavy-duty detergents at conditions of 35C and 95% R.H., using 50-g samples, empty-tower air velocity 5.3 m/sec. Twelve representative commercial Japanese

TABLE I

Properties of Heavy-Duty Detergents[1]

| | | A | B | C | D | E | F | G | H | I | J | K | L |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Particle size distribution (mesh) | On 12 | 6 | 7 | 5 | 0 | 2 | 5 | 1 | 1 | 3 | 5 | 1 | 2 |
| | 12– 20 | 14 | 19 | 14 | 1 | 10 | 11 | 3 | 13 | 11 | 13 | 11 | 23 |
| | 20– 32 | 27 | 55 | 37 | 11 | 33 | 27 | 23 | 35 | 42 | 30 | 19 | 31 |
| | 32– 42 | 21 | 19 | 21 | 30 | 21 | 23 | 32 | 24 | 23 | 22 | 18 | 21 |
| | 42– 55 | 20 | 11 | 12 | 33 | 18 | 18 | 19 | 16 | 14 | 19 | 25 | 12 |
| | 55–100 | 9 | 6 | 8 | 12 | 12 | 11 | 15 | 8 | 6 | 9 | 19 | 7 |
| | Through 100 | 3 | 3 | 3 | 13 | 4 | 5 | 7 | 3 | 1 | 2 | 7 | 4 |
| Mean particle | size (mm) | 0.47 | 0.59 | 0.55 | 0.34 | 0.46 | 0.45 | 0.39 | 0.49 | 0.54 | 0.48 | 0.34 | 0.55 |
| Bulk density | | 0.31 | 0.36 | 0.33 | 0.30 | 0.30 | 0.35 | 0.33 | 0.29 | 0.30 | 0.32 | 0.28 | 0.28 |
| Shape of particle | Degree of spherical shape | Middle –Bad | Middle | Middle | Good–Middle | Middle | Middle –Bad | Good–Middle | Middle | Good | Middle | Very good | Bad |
| | Degree of hole on surface | Middle | Middle | Middle | Small | Small | Middle | Small | Middle | Middle | Middle | No | Middle |
| | Degree of crushed particle | Middle | Middle | Middle | No | Small | Large | Small | Small | Small | Middle | No | Large |
| Analytical composition | Moisture | 12.5 | 11.0 | 12.9 | 5.1 | 9.5 | 10.4 | 9.5 | 12.8 | 7.9 | 4.5 | 9.7 | 9.7 |
| | ABS-Na | 26.4 | 27.2 | 21.0 | 0 | 24.6 | 22.0 | 18.3 | 24.5 | 22.6 | 22.3 | 19.0 | 23.7 |
| | Soap | 1.0 | 0 | 0 | 0 | 0 | 1.4 | 2.0 | 2.0 | 1.1 | 1.2 | 3.0 | 2.0 |
| | Nonionic | 0 | 0 | 0 | 8.8 | 3.0 | 0.7 | 3.8 | 0 | 1.1 | 1.2 | 0 | 0 |
| | $Na_2SO_4$ | 28.6 | 25.1 | 23.6 | 43.9 | 35.1 | 38.1 | 36.6 | 28.7 | 39.1 | 47.8 | 43.8 | 33.5 |
| | STPP | 26.3 | 25.9 | 30.9 | 30.2 | 19.7 | 19.2 | 18.2 | 22.8 | 18.8 | 15.7 | 11.6 | 23.1 |
| | $Na_2CO_3$ | 3.5 | 3.0 | 4.8 | 7.7 | 1.5 | 4.0 | 6.0 | 2.0 | 1.8 | 1.8 | 6.5 | 2.1 |
| | Silicate | 3.4 | 2.2 | 5.0 | 2.6 | 2.0 | 3.1 | 4.2 | 5 6 | 4.5 | 5.4 | 5.6 | 2.9 |
| | CMC | + | + | + | + | + | + | + | + | + | + | + | + |
| | Perborate | – | – | – | – | – | – | – | – | 1.5 | – | – | – |
| | Anticaking agent | + | + | + | – | + | – | – | + | – | + | + | + |

[1] Commercial products are designated as A, B, C, ——L.

products were purchased and tested on the "as received" basis, and their properties were determined (Table I).

*Measurement of Agglomerating Properties by this Method.* Initial height of fluidized particles of these detergents was about 80 cm, and the time required for agglomeration to lower the height to 20 cm was measured. These values are shown in Table II. To measure reproducibility, these 12 samples were tested on three separate days, and the data were subjected to analysis of variance (Table II). Significant differences between samples existed (d = 0.97+) at the 0.05 significance level.

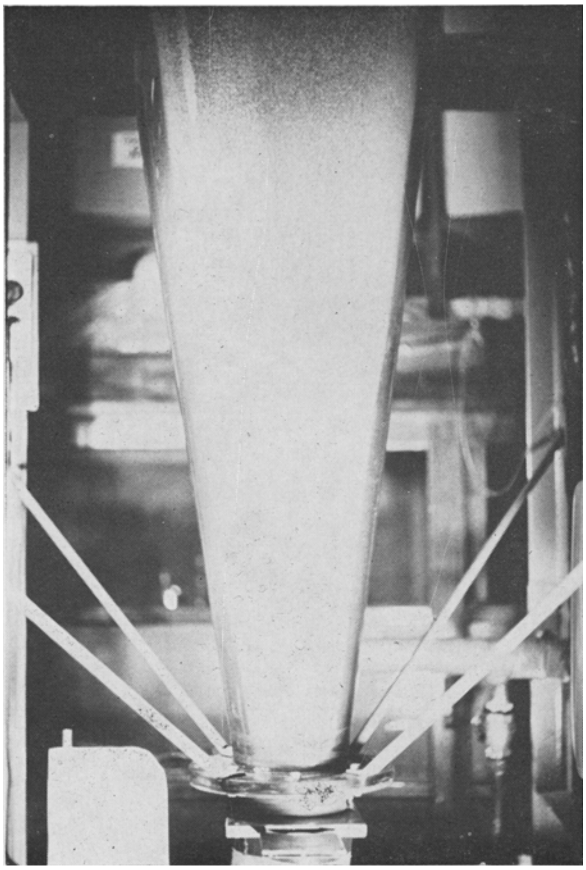

FIG. 4. Practical equipment.

*Measurement of Agglomerating Properties of Detergents in Cartons.* Cartons with bottom dimensions of 3 cm × 8 cm and height of 10 cm were made with cardboard having a water vapor permeability of about 800 g/m$^2$/day. Several cartons containing each of these samples were stored at 35C, 95% R.H. One was taken out each day, the condition of agglomeration was observed, and the following points were evaluated by the apparatus or appearance: sliding angle —a carton with the upper part opened was set in the measuring apparatus (Figure 5) and inclined gradually at a constant rate, and the angle at which the powder starts to slide was obtained; free-flowing property—the condition when powder flows out of the carton was observed and scored with the initial condition as 10 points; degree of agglomeration—the powder was removed from the carton, and the agglomerated portion was examined visually and measured for percentage. Results from the initial through the fourth day were shown in Table III.

*Comparison of Sensory and Agglomerating Procedures.* According to experience, free flow of the product is the most important property for indicating agglomeration of commercial detergents. The relation between the time required to attain the standard "fall" in the present procedure and the average sensory value of the free-flowing properties in the cartons is shown in Figure 6. The correlation coefficient, $r_s$, was calculated (0.849) and indicated 99% reliability. Thus it may be said that the results of the procedure coincide with sensory value.

Similarly the relation between agglomerating property by the present procedure and free-flowing property by sensory measurement was obtained at 35C, 85% R.H. for heavy-duty detergents, and at 35C, 95% R.H. and 35C, 85% R.H. for light-duty detergents. The data and correlation by both procedures are shown below.

TABLE II

Evaluation of Agglomerating Property by the Fluidized Bed Method
Heavy-Duty Detergents 35C; 95% R.H.; 50-g Sample; Air Velocity 5.3 m/sec

| Replication | Sample A | B | C | D | E | F | G | H | I | J | K | L |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| First day | 4.9 | 5.0 | 3.9 | 9.0 | 5.7 | 3.3 | 6.5 | 4.7 | 1.7 | 7.9 | 3.0 | 4.4 |
| Second day | 5.4 | 5.6 | 5.4 | 8.3 | 6.4 | 3.0 | 5.9 | 5.2 | 1.3 | 8.4 | 3.0 | 3.4 |
| Third day | 5.7 | 5.5 | 5.7 | 7.6 | 5.3 | 3.1 | 4.9 | 5.2 | 1.5 | 7.0 | 3.4 | 3.0 |
| Mean | 5.3 | 5.4 | 5.0 | 8.3 | 5.8 | 3.1 | 5.8 | 5.0 | 1.5 | 7.8 | 3.1 | 3.6 |

unit = min.

Analysis of Variance

| Variable | Sum of squares | Degree of free-dom | Mean square | Fo | F |
|---|---|---|---|---|---|
| Detergent | 123.58 | 11 | 11.23 | 37.1 | $F_{22}^{11}$ (0.01) = 2.26 |
| Day | 0.50 | 2 | 0.25 | 0.76 | $F_{22}^{2}$ (0.05) = 3.44 |
| Error | 7.25 | 22 | 0.33 | | |
| Total | 131.33 | 35 | | | |

d(0.05) = 0.973 $\sigma = 0.469$

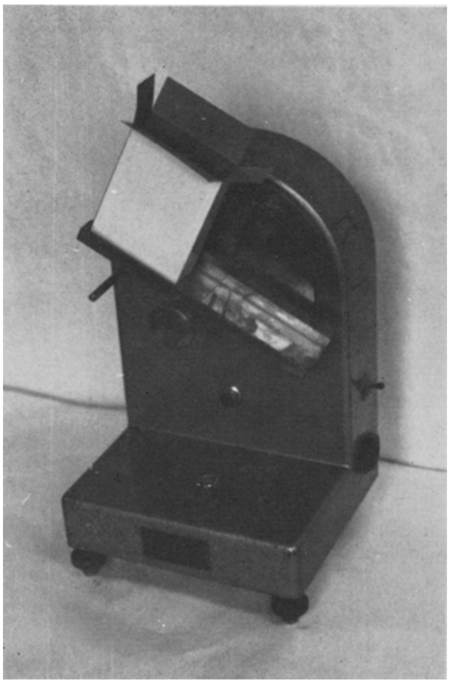

FIG. 5. Apparatus for measuring sliding angle.

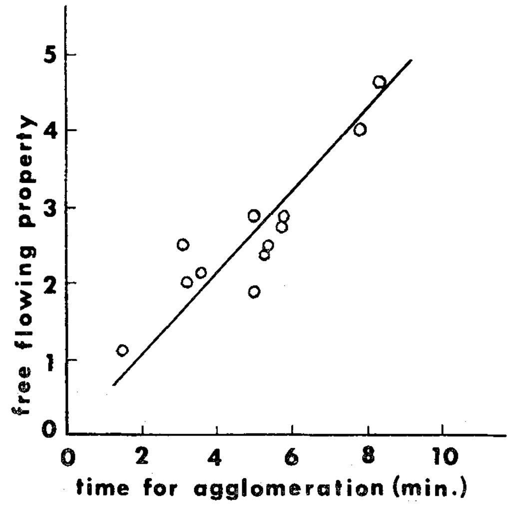


FIG. 6. Relation between measured value and sensory value.

TABLE III

Evaluation of Agglomerating Property of Detergents in Carton

| | | Sample A | B | C | D | E | F | G | H | I | J | K | L |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Initial | Moisture content % | 12.5 | 11.0 | 12.9 | 5.1 | 9.5 | 10.5 | 9.5 | 12.8 | 7.9 | 4.5 | 9.7 | 9.7 |
| First day | Moisture content % | 18.6 | 17.8 | 20.6 | 15.8 | 15.3 | 17.1 | 17.4 | 17.5 | 16.5 | 14.4 | 18.0 | 17.1 |
| | Sliding angle | 90 | 90 | 51 | 75 | 90 | 90 | 71 | 67 | 90 | 62 | 90 | 90 |
| | Free-flowing tendency[a] | 5.5 | 5.5 | 5.5 | 7.5 | 5.5 | 5.5 | 6 | 4.5 | 2.5 | 7 | 5 | 5.5 |
| | Degree of agglom-eration % | 25 | 15 | 15 | 10 | 15 | 20 | 15 | 25 | 50 | 5 | 10 | 25 |
| Second day | Moisture content % | 21.3 | 23.3 | 26.1 | 24.0 | 21.7 | 23.9 | 20.5 | 19.6 | 22.4 | 19.6 | 23.5 | 21.7 |
| | Sliding angle | 90 | 90 | 90 | 90 | 90 | 90 | 90 | 90 | 90 | 90 | 90 | 90 |
| | Free-flowing tendency[a] | 2.5 | 3 | 2.5 | 7 | 4 | 1.5 | 2.5 | 2 | 2 | 3 | 2 | 2 |
| | Degree of agglom-eration % | 80 | 50 | 65 | 15 | 75 | 80 | 50 | 80 | 70 | 70 | 70 | 80 |
| Third day | Moisture content % | 27.4 | 27.8 | 28.8 | 28.0 | 28.7 | 29.1 | 26.8 | 26.2 | 24.9 | 24.6 | 28.4 | 27.4 |
| | Sliding angle | 90 | 90 | 90 | 90 | 90 | 90 | 90 | 90 | 90 | 90 | 90 | 90 |
| | Free-flowing tendency[a] | 1.5 | 1.5 | 2 | 2.5 | 2 | 1 | 2.5 | 1 | 0 | 1.5 | 2 | 1 |
| | Degree of agglom-eration % | 85 | 100 | 80 | 20 | 90 | 100 | 80 | 95 | 100 | 80 | 85 | 90 |
| Fourth day | Moisture content % | 31.5 | 36.2 | 34.8 | 32.4 | 28.6 | 33.5 | 34.0 | 32.4 | 28.3 | 28.2 | 28.7 | 32.7 |
| | Sliding angle | 90 | 90 | 90 | 90 | 90 | 90 | 90 | 90 | 90 | 90 | 90 | 90 |
| | Free-flowing tendency[a] | 0 | 0 | 1.5 | 1.5 | 0 | 0 | 0 | 0 | 0 | 0.5 | 1 | 0 |
| | Degree of agglom-eration % | 100 | 100 | 90 | 100 | 100 | 100 | 100 | 100 | 100 | 100 | 100 | 100 |

[a] Basis of 10 as completely free-flowing.

TABLE IV

Evaluation of Agglomerating Property by the Fluidized Bed Method
Heavy-Duty Detergents 35C; 85% R.H.; 50-g Sample; Air Velocity 5.3 m/sec

| Replication | Sample A | B | C | D | E | F | G | H | I | J | K | L |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| First | 13 | 15 | 86 | 52 | 18 | 6 | 122 | 17 | 3 | 46 | 10 | 16 |
| Second | 15 | 16 | 83 | 64 | 21 | 6 | 105 | 19 | 4 | 41 | 8 | 12 |
| Third | 16 | 14 | 91 | 61 | 19 | 8 | 98 | 18 | 3 | 37 | 11 | 15 |
| Mean | 15 | 15 | 87 | 59 | 19 | 7 | 108 | 18 | 3 | 41 | 30 | 14 |

unit = min.

Analysis of Variance

| Variable | Sum of squares | Degree of freedom | Mean square | Fo | F |
|---|---|---|---|---|---|
| Detergent | 38517 | 11 | 3501.54 | 172.9 | $F_{24}^{11}(0.01) = 2.21$ |
| Error | 486 | 24 | 20.25 | | |
| Total | 39003 | 35 | | | |

d(0.05) = 7.69 $\sigma = 4.50$

2. Comparison of the agglomerating properties of heavy-duty detergents at 35 C, 85% R.H., using 50-g samples, was made with an empty-tower air velocity 5.3 m/sec. These data are shown in Table IV, indicating the longer agglomeration period for the lower relative humidity condition and the continued differences between samples at the 0.05 significance level.

Table V shows the result of measurement of agglomerating properties of detergents in cartons. The correlation coefficient, $r_s$, between the time required to attain the standard "fall" in the present procedure and the average sensory value of the free-flowing properties in the cartons was calculated (0.797), indicating a reliability level of 99%.

3. Comparison of agglomerating properties of light-duty detergents at conditions of 35C, 85% R.H. and 35C, 95% R.H. using 30-g samples, empty-tower air velocity 4.0 m/sec. Six representative commercial Japanese products were purchased and tested as received. Their properties are shown in Table VI. The

TABLE V

Evaluation of Free-Flowing Tendency of Detergents in Carton

| Free flowing tendency | Sample A | B | C | D | E | F | G | H | I | J | K | L |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| First day | 9.5 | 9.5 | 9.5 | 9.5 | 9.5 | 8.0 | 9.5 | 9.0 | 7.0 | 9.0 | 9.5 | 9.5 |
| Second day | 7.5 | 7.5 | 9.0 | 7.0 | 7.5 | 7.0 | 9.0 | 8.0 | 6.0 | 7.0 | 7.5 | 7.0 |
| Third day | 7.0 | 6.5 | 8.5 | 7.0 | 6.5 | 5.5 | 8.5 | 6.0 | 5.0 | 6.0 | 7.5 | 7.0 |
| Fourth day | 5.5 | 5.5 | 8.0 | 7.0 | 5.0 | 5.0 | 7.5 | 5.5 | 3.0 | 5.5 | 6.0 | 5.5 |
| Fifth day | 4.5 | 4.5 | 6.5 | 7.0 | 4.5 | 2.0 | 7.0 | 4.5 | 2.5 | 3.0 | 4.0 | 4.0 |
| Mean | 6.8 | 6.7 | 8.2 | 7.5 | 6.6 | 5.5 | 8.2 | 6.6 | 4.7 | 6.1 | 6.9 | 6.6 |

TABLE VI

Properties of Light-Duty Detergents[1]

| | | M | N | O | P | Q | R |
|---|---|---|---|---|---|---|---|
| Particle size distribution (mesh) | On 20 | 9 | 3 | 5 | 1 | 4 | 1 |
| | 20– 32 | 28 | 20 | 26 | 7 | 26 | 25 |
| | 32 42 | 19 | 29 | 25 | 39 | 20 | 26 |
| | 42 55 | 13 | 16 | 15 | 32 | 19 | 17 |
| | 55-100 | 17 | 14 | 15 | 17 | 13 | 20 |
| | Through 100 | 14 | 18 | 14 | 4 | 18 | 11 |
| Mean particle size (mm) | | 0.38 | 0.36 | 0.37 | 0.34 | 0.35 | 0.36 |
| Bulk density | | 0.17 | 0.17 | 0.17 | 0.16 | 0.22 | 0.18 |
| Shape of particle | Degree of spherical shape | Middle-Bad | Middle | Middle | Good | Good | Good |
| | Degree of hole on surface | Large | Large | Large | Middle | Large | Large |
| | Degree of crushed particle | Large | Very large | Large–Middle | Small | Large | Middle–small |
| Analytical composition | Moisture | 2.0 | 3.0 | 1.8 | 3.2 | 2.8 | 1.8 |
| | ABS-Na | 19.3 | 9.4 | 31.2 | 19.7 | 18.9 | 19.8 |
| | Alcohol sulfate-Na | 0 | 25.7 | 9.8 | 11.0 | 11.6 | 9.1 |
| | Soap | 0 | 0 | 0 | 0 | 0 | 1.1 |
| | Nonionic | 7 | 0 | 0 | 0 | 0 | 5.1 |
| | $Na_2SO_4$ | 60.9 | 60.2 | 53.0 | 52.3 | 64.1 | 61.9 |
| | STPP | 0 | 0 | 0 | 9.5 | 0 | 0 |
| | $Na_2CO_3$ | 1.3 | 0 | 0.6 | 0 | 1.2 | 1.0 |
| | CMC | + | – | – | + | – | + |
| | Silicate | 2.6 | – | – | – | – | – |

[1] Commercial products are designated as M, N, ——R.

TABLE VII

Evaluation of Agglomerating Properties of Light-Duty Detergents
35C; 85% R.H.; 30-g Samples; Air Velocity 4.0 m/sec

| Measuring condition | Method | Sample M | N | O | P | Q | R | |
|---|---|---|---|---|---|---|---|---|
| 35C, 85% R.H. | Fluidized bed method | 14.2 | 9.8 | 7.8 | 4.3 | 3.8 | 1.2 | unit = minute d(0.05) = 1.72 $\sigma = 0.70$ Mean of duplication |
| | Observation of free-flowing tendency | 6 | 5 2 | 4.8 | 1 | 0.8 | 0.7 | Mean of several 7, 15, 24 hrs |
| 35C, 95% R.H. | Fluidized bed method | 255 | 170 | 160 | 106 | 61 | 38 | unit = sec. d(0.05) = 23.6 $\sigma = 9.65$ Mean of duplication |
| | Observation of free-flowing tendency | 7.2 | 5 2 | 5.2 | 2 | 0.8 | 0.7 | Mean of several 4, 17, 15 hrs |

data for agglomerating properties by the present procedure and the data for free flowing properties in cartons are summarized and shown in Table VII. Light-duty detergents show an earlier agglomerating property than heavy-duty detergents in either condition. Correlation coefficients between sensory and agglomeration procedures, $r_s$, were 0.935 in 35C, 85% R.H. and 0.920 in 35C, 95% R.H. with 99% reliabilities of estimate.

Because discussion with respect to the effect of composition on the agglomerating properties of detergents is not the main object of this report, it has been omitted. However it is clearly indicated that the agglomerating property of detergents is influenced by particle shape. The precision of this procedure is about 10% in coefficient of variation ($\sigma/\overline{X}$), as summarized in Table VIII. Precision and correlation with actual tendency were not clear in former methods, but the present method is sufficient for practical use and indicates good agreement with actual conditions. Measurement in a very short time is possible.

TABLE VIII
Summary of Precision

| | Measuring condition | Measured Value (Minute) | | | Variance $\sigma$ | Variation coefficient $\sigma/\overline{X}$ |
|---|---|---|---|---|---|---|
| | | Max. | Min. | Mean | | |
| Heavy-duty detergent | 35C, 95% R.H. | 9.0 | 1.3 | 5.0 | 0.47 | 9.4 |
| | 35C, 85% R.H. | 122 | 3 | 33 | 4.5 | 13.6 |
| Light-duty detergent | 35C, 95% R.H. | 4.3 | 0.5 | 2.2 | 0.16 | 7.3 |
| | 35C, 85% R.H. | 15.3 | 1.1 | 6.8 | 0.70 | 10.3 |
| Mean | | | | | | 10.2 |

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