

Method for measuring bulk density, tap density and the Hausner ratio for additives and feedstuffs

Following the purchase of a new powder compaction apparatus, Tecaliman decided to modify its measurement procedure for bulk and tap densities, and the resulting Hausner ratio. The objective was to arrive at a procedure similar to the measuring technique used by Carr (1965), as applied to the Hosokawa apparatus. Abdullah and Geldart (1999) demonstrated that this technique did not give the same results as the method based solely on standard ISO 3953-1985, used previously at Tecaliman.

1. Principle

This method is based on both standard ISO 3953-1985 and standard ISO 787/11. The bulk density measurement (B.D.) consists in determining the mass of a known volume of powder after it has flowed freely into a test tube. After increasing both the height of the product column and a compaction phase, induced under a set of specific conditions, the weight of the powder contained in an identical volume is used to determine the tap den-

sity (T.D.). The Hausner ratio (H.R.) is derived from these two measurements.

2. Apparatus

TECALIMAN used the Autotap, a Quantachrome brand compaction machine. A funnel with output diameter 10 mm was placed above a test tube of a known volume (100 ml or 250 ml). The test tubes were made by a glass manufacturer and delivered in 100 ml and 250 ml volume sizes. All the test tubes had a diameter of 4 cm, but a height of 8.0 or 18.7 cm depending on the volume. They were levelled and an olive fitting at their top was used to fit an extension with a volume equal to 50% of the volume of the corresponding test tube. The extension was held on the test tube vertically by a metal clip with a clamping ring (Figure 1). wall effects were reduced by the 40-mm diameter, which was wider than that of the test tube used previously (27 mm). The test tube can be attached to the Autotap compaction apparatus that oscillate vertically when in operation.

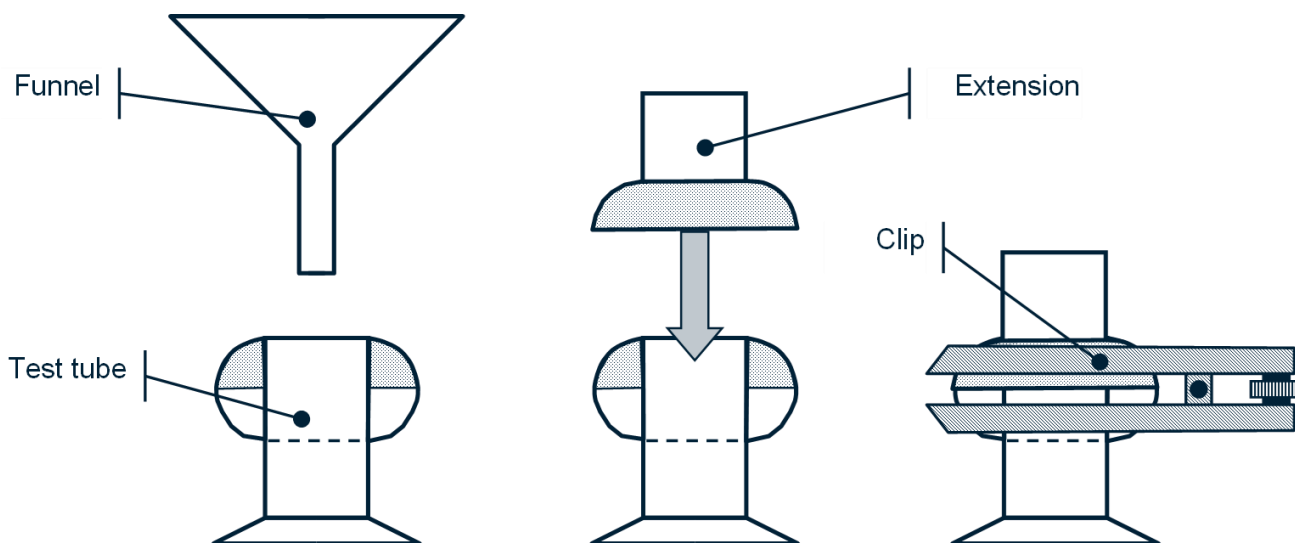


Figure 1: Diagram of a test tube

The number of strokes can be programmed. The frequency and amplitude of this movement were 260 strokes per minute and 3 mm respectively.

Electronic scales, accurate to 0.01 g, were used to weigh the powder.

3. Production

The 100-ml test tube was used for additives, and the 250-ml test tube for animal meals. A separate sample had to be used for each measurement. Compacted samples should never be used as their internal structure is likely to have been modified.

The distance between the funnel and the upper end of the test tube was 20 mm. The measurement protocol was as follows:

- The calibrated test tube was weighed to identify its tare weight: **T**
- The product was fluidised using a metal whisk
- The test tube was centred under the funnel
- The test portion was poured carefully into the funnel until the test tube ran over
- The stand holding the funnel was removed carefully
- The base of the test tube was held firmly making sure there were no vibrations, then the powder at the top of the test tube was levelled using a flat scraper or card
- The test tube was tapped two or three times so as to prevent any powder being lost during travel
- The outside of the test tube was cleaned with a brush
- The full test tube was weighed; i.e. **Ma**
- The appropriate extension was fitted onto the test tube (same number) and filled to the top by pouring the powder in directly, without using the funnel, up to a height of approx. 2 cm
- The whole assembly was placed on the compaction machine and the clamping mechanism fitted
- The machine was programmed for 1250 strokes, and switched on
- The test tube was removed from the machine
- The extension was removed and the powder at the top of the test tube levelled using a flat scraper or card.
- The outside of the test tube was cleaned with a brush
- The full test tube was weighed; i.e. **Mt**

The bulk (BD) and tap (TD) densities were calculated:

$$MVA = \frac{Ma - T}{V} \text{ in g/cm}^3$$

$$MVT = \frac{Mt - T}{V} \text{ in g/cm}^3$$

The Hausner ratio (HR) was calculated using the equation:

$$IH = \frac{MVT}{MVA}$$

The Hausner ratio would provide a flowability index for the tested powders. The higher the ratio, the greater a powder's tendency to self-compact.

4. Intrinsic qualities

This assessment was based on measurements that were taken in series of 3, on 4 separate days, on 5 additives selected as being representative of the additives found in animal feeds, and on 4 feedstuffs (P: piglet, DC: dairy cow, Ch: chicken, T: turkey). This calculation method demonstrated highly effective qualities (Table 1 to Table 3), which are superior to those of the previous method, particularly in terms of sensitivity and accuracy (concerning additives). In many cases, the result was exact as from the first measurement (Table 3). It was nevertheless recommended to take two measurements in order to refine the results and provide for later statistical analyses.

Products	A	B	C	D	E
BD (g/cm³)	1.38	0.84	1.47	0.42	0.13
<i>Group</i>	<i>b</i>	<i>c</i>	<i>a</i>	<i>d</i>	<i>e</i>
TD (g/cm³)	1.79	1.04	2.14	0.52	0.26
<i>Group</i>	<i>b</i>	<i>c</i>	<i>a</i>	<i>d</i>	<i>e</i>
Hausner ratio	1.30	1.24	1.46	1.25	1.99
<i>Group</i>	<i>c</i>	<i>d</i>	<i>b</i>	<i>d</i>	<i>a</i>

Table 1: Results of the test on the method's intrinsic qualities for additives

Products	P	DC	Ch	T
BD (g/cm³)	0.69	0.65	0.58	0.58
<i>Group</i>	<i>a</i>	<i>b</i>	<i>c</i>	<i>d</i>
TD (g/cm³)	0.80	0.70	0.76	0.72
<i>Group</i>	<i>a</i>	<i>d</i>	<i>b</i>	<i>c</i>
Hausner ratio	1.15	1.09	1.30	1.25
<i>Group</i>	<i>c</i>	<i>d</i>	<i>a</i>	<i>b</i>

Table 2: Results of the test on the method's intrinsic qualities for feedstuffs

	BD	TD	HR
Sensitivity	Excellent		
Accuracy	Excellent		
Min. nb of additive measurements	0.99	1.70	0.37
Min. nb of feedstuff measurements	0.11	0.07	0.15
Reproducibility	Yes	Yes	Yes

Table 3: Intrinsic qualities of the methods

5. Range of additives

A range of 29 additives selected in 1995, stored in a cold room, was reused. As before, the characterisation of these 29 representative animal feed additives revealed significant variations for bulk or tap densities as the spread (deviation between the minimum and maximum values) represented 9 to 7 times the minimum value (Table 4). The spread of the Hausner ratio variation increased significantly, rising from 0.27 to 0.875 with this new method.

Criteria	BD (g/cm ³)	TD (g/cm ³)	HR
Mean	0.739	0.971	1.308
Standard deviation	0.063	0.093	0.037
Minimum	0.141	0.278	1.093
Maximum	1.466	2.154	1.968
Spread	1.325	1.876	0.875
Median	0.611	0.758	1.253

Table 4: Statistical report on the range of 29 representative products

The frequency histograms for these two parameters (Figure 2 and Figure 3) suggested a dispersed population with most products being around 0.55 g/cm³ for BD and 0.65 g/cm³ for TD.

The range of variation for the Hausner ratio (Figure 4) was greater than that recorded with the previous method: this approached 2, where the maximum was 1.3 with the previous method. Most products lay between 1 and 1.6. According to Castel (1995), products with a Hausner ratio greater than 1.4 would be cohesive. This new method classified eight products in this category (compared to zero with the previous method): a former growth factor, a vitamin, a coccidiostatic and, above all, 5 out of 6 oligo-elements in the range.



Figure 2: Frequency histogram for the bulk density of the additive population

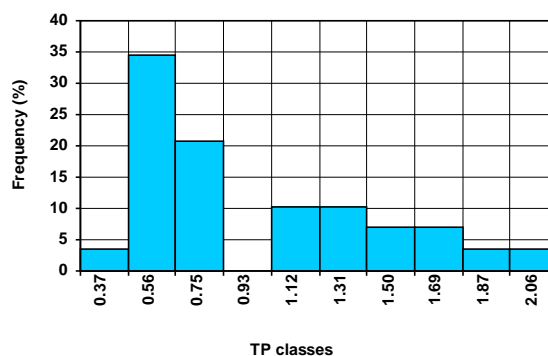


Figure 3: Frequency histogram for the tap density of the additive population

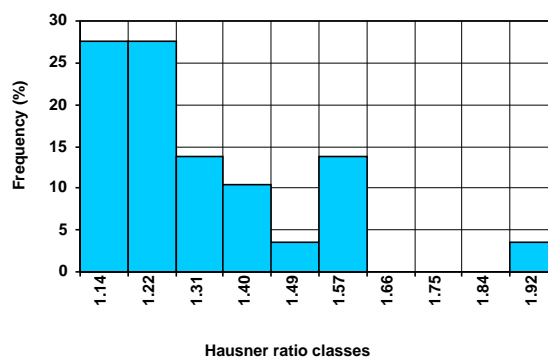


Figure 4: Frequency histogram for the Hausner ratio of the additive population

The discriminating power distinguishes a given product from another, similar, product, despite the variation in supplies.

Based on the measurements taken on three samples of 29 representative products, it was demonstrated that the discriminating power of this method exceeded that of the previous method for all 3 criteria, as the number of significantly different groups was always greater (Table 5).

	Previous method	New method
BD	11 groups	17 groups
TD	16 groups	21 groups
HR	15 groups	17 groups

Table 5: Number of significantly different groups distinguished by the methods within the population of 29 additives

Admittedly, as can be seen in Figure 5, both density measurements correlated very well in terms of redundancy. The slope and deviations from the correlation line reveal the variation in the Hausner ratio.

This Hausner ratio is supposed to be a flowability index. However, comparison with the angle of repose (flow) showed that these two parameters were not inter-correlated (Figure 6). The relationship breaks down into two areas:

- Below a Hausner ratio of 1.4, the angle of repose varies over a wide range of 35 to 70°

- Above 1.4, the ratio increases more rapidly, while the angle of repose appears to stagnate, showing an increase of only a few degrees

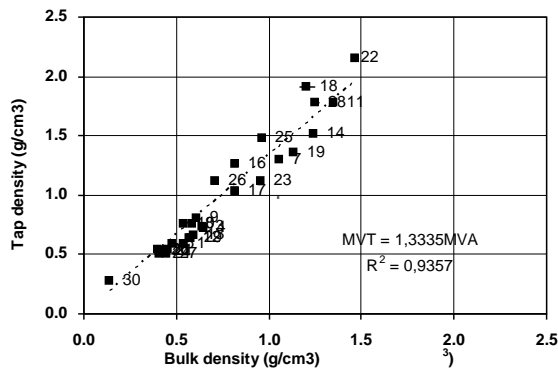


Figure 5: Correlation between the two density measurements

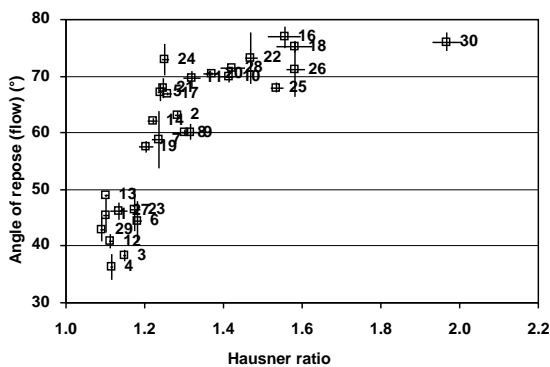


Figure 6: Relationship between the Hausner ratio and the angle of repose (flow)

6. Range of feedstuffs

The four feedstuffs used for the accuracy tests (coded 1) were supplemented by four feedstuffs of the same formula (coded 2). They were sourced at the same plant, but manufactured at a later date.

	P1	DC2	DC1	T1	P2	Ch1	Ch2	T2
BD	0.69	0.65	0.65	0.64	0.64	0.60	0.59	0.56
g/cm3	a	b	bc	bc	c	d	d	e
TD	0.80	0.77	0.76	0.74	0.73	0.72	0.70	0.70
g/cm3	a	b	b	c	c	d	e	e
HR	1.28	1.25	1.24	1.20	1.16	1.15	1.09	1.08
	a	b	c	d	e	e	f	f

Table 6: Classification of eight feedstuffs according to three criteria

By classifying the feedstuffs in their descending order of bulk density, we showed that the method could be used to discern differences between feedstuffs that are theoretically similar (Table 6). Based on the three measurements taken on each feedstuff, the variance analyse and the Fischer test were used to classify the eight samples into 5 groups for density and 6 very different groups for the Hausner ratio.

7. Conclusion

This new technique for determining bulk density requires apparatus that is slightly more complex than that used previously, as it involves the use of a special calibrated test tube to measure tap density. However, it is just as quick and very simple to implement with approx. 5 minutes handling time.

The identification of the two densities and the Hausner ratio takes a little time, but less than before, as it only takes 5 minutes to apply the 1250 strokes. A measurement therefore takes approx. 15 minutes. The cost is comparable though due to amortisation of the "Autotap" apparatus.

This method demonstrates an all-round increase in quality: sensitivity, accuracy, repeatability, measurement range. These tests also made it possible to apply and check this quality with the animal feed. This improved quality is certainly explained by:

- The larger diameter of the test tubes, which limits the wall effect
- The regularity of the apparatus' powder compaction strokes
- The 2 successive weight measurements, which provide more accurate results than the combination of weight and volume measurements.

8. Bibliography

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