

## Comparison of pellet hardness measurements made with multiple devices

In common with pellet durability, pellet hardness is one of the criterions used to assess pellet cohesion qualities but, in this case, has more to do with zootechnical issues (i.e. grip quality, livestock chewing mechanisms).

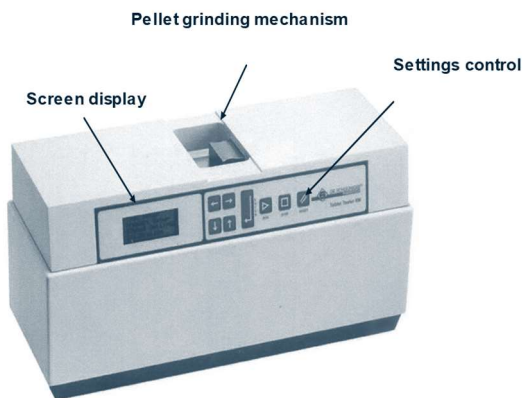
The hardness measurement consists in evaluating longitudinal pellet strength during grinding operations.

The hardness value corresponds to a maximum compressive stress value taken at the exact moment at which the pellet breaks.

### 1. Hardness testers

#### 1.1. Schleuniger 6D hardness tester

This hardness tester (Figure 1) is mainly used for testing tablet hardness in the pharmaceutical sector. The machine displays the compressive stress value measured at the exact time of tablet breakage.



**Figure 1: Schleuniger 6D hardness tester**

This is a semi-automatic table-top hardness tester where the operator just inserts pellets one-by-one according to the cycle programmed between each grinding operation. It includes optional operating software.

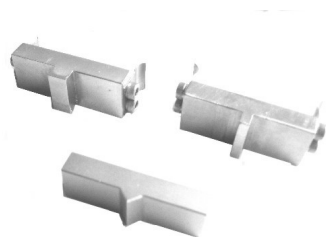
Hardness values are given in Newtons (N), and in other units such as kiloponds (Kp or Kilogram-force), Strong Cobbs (Sc) or Pounds (Lb).

The device has the added advantage of optional on-site calibration using a 5-kg standard weight supplied by the manufacturer.

As the breaking stress depends on the length of

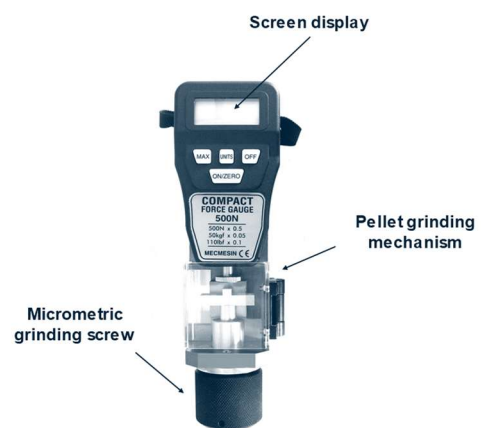
each, individual stressed pellet, this length has to be measured with a vernier calliper prior to grinding. To improve measurement speed and accuracy, Tecaliman, which has been using this device since 1997, has had special jaws made (Figure 2) that are shorter than the pellets (6.4 mm and 3 mm), thus ensuring that the grinding surface is identical on each pellet (see i'Tec\_G2).

This means that the breaking stress is always applied to an identical pellet length, thus avoiding fiddly and often inaccurate measurements with vernier callipers.



**Figure 2: Example of a grinder jaw**

#### Sviac hardness tester (500 Newton)



**Figure 3: Sviac hardness tester**

As with the Schleuniger 6D, this portable device, little known (Figure 3) in the animal feed sector,

measures the hardness of each, individual pellet. In common with the Schleuniger hardness tester, this device only measures the compressive stress value at the time of tablet breakage.

Hardness values are given in Newtons (N), and also in other units such as Kilogram-force (Kgf) or Pound-force (Lbf).

The pellets are ground manually one-by-one against the force sensor using a micrometric control knob that slows the process down and reduces the approach force.

As with the Schleuniger tester, Tecaliman purchased this device and modified the grinding jaw in order to avoid having to measure each individual pellet.

### 1.2. Kahl motorised hardness tester

The Kahl hardness tester (Figure 4) offers a modernised version of the Kahl gripper (see i'Tec\_G2), which has been used in the animal feed sector for many years now.

This version is more recent than the motorised version tested at the time of publishing Technical Datasheet No. 9.



Figure 4: Kahl motorised hardness tester

The pellets are ground vertically one-by-one by a jaw mechanism (truncated cone) designed by the company to display values either as a Kahl hardness or in Newtons (N).

The Kahl device uses a grinding jaw of a specific size that makes it difficult to determine the surface area in contact with the pellet. This means that the measurement cannot be converted into a surface area unit. This prevented its comparison with the other devices.

### 1.3. Instron 5543 hardness tester (1000 Newton)

This sophisticated measurement system (Figure 5) records the compressive load according to the distance travelled by the compression plate.

Controlled by an appropriate operating software system, this device enables dynamic investigation into the grind-strength of whole pellets (Figure 6), or of a group of pellets and, possibly, their breaking strength.

Whole pellets are ground vertically along their entire length on a plate. As the grinding force is applied over the whole plate, comparison of the results requires that each pellet, or its whole length, has to be measured with a vernier calliper before each grinding operation.

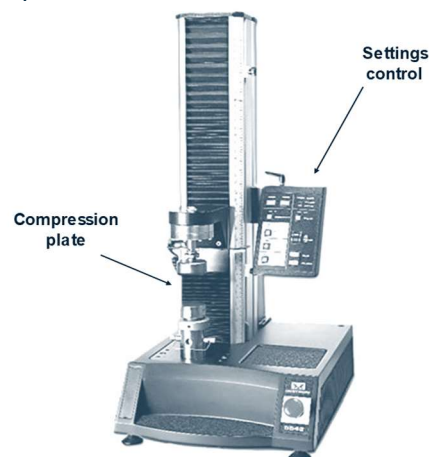


Figure 5: Instron 5543 hardness tester

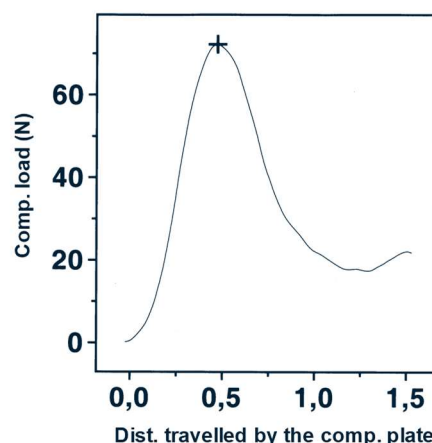


Figure 6: Example of a curve

## 2. Expressing the hardness value

Measuring devices generally display the breaking force or breaking stress in Newtons (N) or in kilogram-force (Kgf).

To compare pellets of identical diameter, it is necessary to know the pellet grinding length (force application).

This comparison can be made with many measuring devices, provided that the jaws are shorter than the pellet.

Hardness is therefore expressed in N/cm:

$$\text{Dureté (N/cm)} = \frac{F}{L}$$

Hardness expressed in N/cm  
F = Pellet breaking force in N

L = Length in cm of force application on the pellet  
 To compare pellets of differing diameters, it is necessary to take account of the length of the pellet and its diameter.

In this case, the reference unit is the megapascal.

$$\text{Dureté (MPa)} = \frac{F \times 2}{\pi \times d \times L}$$

Hardness expressed in MPa

F = Pellet breaking force in N

d = pellet diameter in mm

L = Length in mm of force application on the pellet

### 3. Method

A set of 5 feed pellet samples, of varying hardnesses, based on a single poultry feed, was produced by pelleting the feed at 5 different treatment temperatures at the conditioner output (40, 45, 55, 70 and 99°C); all other pelleting line settings were identical.

### 4. Comparison of hardness testers

The results were based on 30 values for the Sviac, Schleuniger and Kahl hardness testers, and 100 values for the Instron hardness tester.

First, a Box & Whiskers plot test was run to remove outliers.

Out of the set of 5 samples, the Sviac and Kahl hardness testers only distinguished between 3 groups, while the Schleuniger and Instron hardness testers were able to distinguish between 4 groups (Table 1).

The Schleuniger and Sviac hardness testers gave virtually identical mean hardness values (Figure 7).

Hardness (Mpa) modality	per Schleuniger	Sviac	Kahl (*)	Instron (**)
Modality 99°C	1.4 (a)	1.7 (a)	44.2 (a)	0.9 (a)
Modality 70°C	1.0 (b)	1.1 (b)	42.9 (a)	0.8 (b)
Modality 55°C	0.8 (c)	0.8 (c)	35.5 (b)	0.6 (c)
Modality 45°C	0.7 (c,d)	0.8 (c)	29.7 (c)	0.5 (d)
Modality 40°C	0.6 (d)	0.8 (c)	28.4 (c)	0.5 (d)
Significantly different samples	4	3	3	4

(\*): Kahl hardness is expressed in N, rather Mpa.

(\*\*): Instron mean values were calculated using 100 measurements per sample.

Table 1: Report on mean hardness values

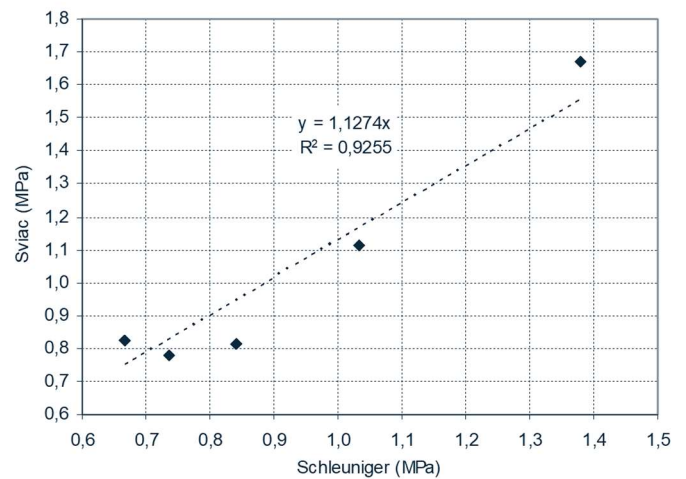


Figure 7: Sviac tester hardness values against Schleuniger tester hardness values

A study of an identical sample of pellets, measured by 3 different operators, revealed that the manually operated Sviac hardness tester is operator-sensitive. For the Instron hardness tester, the mean values plotted below those of the Schleuniger and Sviac machines.

The first point could be due to the need to measure pellet length manually prior to each grinding operation. As the ends of the pellets are cut obliquely, the vernier calliper over-estimates the length, thus mathematically reducing the hardness result.

An alternative hypothesis would be that the grinding surface area is greater for the Instron hardness tester (whole pellet) than for the other hardness testers that use jaws with a surface area inferior to that of the pellet. This would minimise the impact of formula particle size (pieces of wheat, corn, etc.) on hardness values.

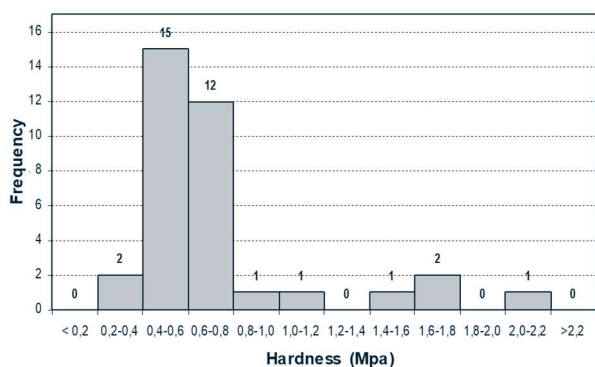
## 5. Conclusion

The Sviac portable hardness tester is very simple to use and enables the most accurate possible checks on pellet production; however, the operator needs to have received the appropriate training and must perform all the measurements in order to be able to make the comparisons.

Schleuniger and Kahl table-top hardness testers are designed to be used for laboratory checks and facilitate measurement repetition.

The Schleuniger hardness tester has the added advantage of easy-adjustment of a range of compression jaws. The pointed jaw (triangular) appears to apply greater stress on the pellets' particulate bonds, while the square jaw (rectangular), which acts over a greater surface area, appears to apply greater stress on the steam-generated solid and liquid bonds. One possible interpretation could be that the triangular jaw corresponds more closely to the action of incisors, while the rectangular jaws mimic action by molars. Given its cost-in-use, the Instron hardness tester is designed more specifically for research and development labs.

Generally speaking, and as illustrated in Figure 8, the hardness values for a given pellet sample vary widely. Often, a few very hard pellets can be found in a pellet population with a lower hardness value, thus shifting the mean upwards and disrupting the sample assessment. The lower the number of measurements taken, the greater the impact of these outlier pellets.



**Figure 8: Hardness frequencies obtained with the Schleuniger hardness tester in the 45°C test**

In order to optimise pellet sample comparisons, it is advisable to increase the number of measurements ( $n > 30$ ) and express hardness values in megapascals wherever possible.

## 6. List of hardness tester suppliers

Schleuniger de Pharmatron: Distributed in France by Farpi France Z.I. "Le Chanay" - B.P. 3 - 69720 Saint Bonnet de Mure.

Sviac: Sviac: 2, rue André-Charles-Boulle 92160 Antony.

Kahl Amandus Kahl: 510 Robert Estienne BP 40090 60403 Noyon

Instron: Instron France S.A.S. Bâtiment C Rond-Point de l'Epine des Champs C.S. 40532 - 78996 ELANCOURT

## 7. Bibliography

Melcion J.P., 1981. Expression de la dureté.

i'Tec\_G2. Mesure de la dureté des granulés à l'aide de l'appareil « Schleuniger », Février 1997.

Tecaliman Report No. 458, 2007. Etude de nouvelles méthodes caractérisant la texture des granulés et miettes dans le secteur de l'alimentation animale.