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Using the Heubach[1] method to measure the emission of dust in animal feed additives

During Tecaliman-led experiments on this method, the protocol trialled during the preliminary test phase was that recommended by the equipment manufacturer as part of tests designed to identify the concentration of active agents in dust.
Our objective differed slightly in that the focus here was to develop a simple dust measurement procedure that could be applied to all animal feed additives. This alternative objective meant that the manufacturer's recommended protocol was no longer appropriate; a series of tests led to the design of the protocol described below.

1. Principle

This involves measuring a product's dust concentration, i.e. the quantity of dust generated by a given quantity of stirred additives. This dust was measured by aspirating it into an air flow and capturing it on a filter.

2. Equipment and apparatus

The measurement was taken using a Heubach-brand [1] model II "Dustmeter".
This device consists in (Figure 1):

- one motor unit, one vacuum pump and their respective controls.
- one 2.2-litre dust-generation drum in alloy steel.

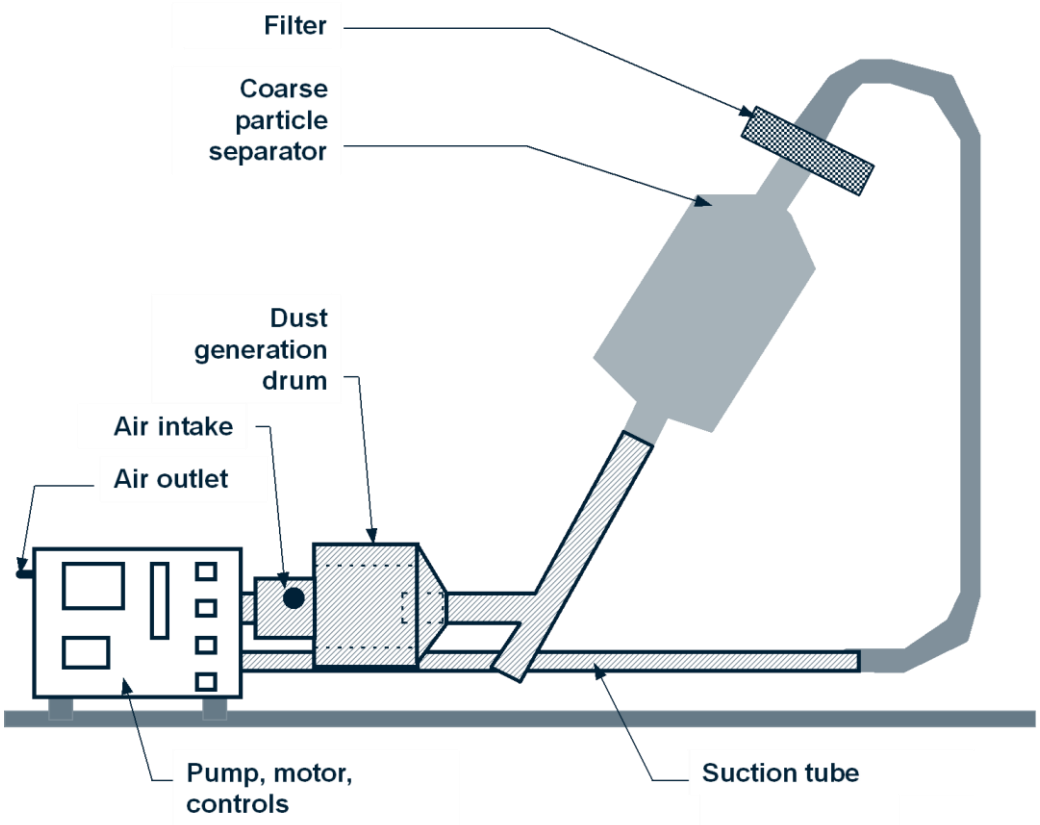

Figure 1: Diagram of the apparatus ([1] Heubach Engineering Gmbh, Heubachstasse 7, D-3394 Langelsheim**)**

The drum contains three baffles that lie at an angle of 45° to the wall, and a motor that rotates it around its axis. This axis provides the air intake through four bores with a diameter of 5.2 mm at their outer edges, on the motor side. The drum can be opened fully on the opposite side via a "hatch" attached with two snap fasteners. This hatch is made leaktight with an O-ring seal.

- one spring-loaded, flush-fit lipped bushing forming a seal between the fixed and mobile sections.
- one elbow section angled at 110°. The tube penetrates the dust-generation drum to a distance of 3 centimetres.
- a one-litre glass separator for coarse particles. Coarse particles fall in the separator due both to the air speed decreasing with increasing diameter and, in the case of this model, to the rising air flow.
- filters in a filter holder.
- suction tube used to close the air circuit.

Inside the motor/pump unit, the air flowing through the suction tube was filtered before entering the vacuum pump. The air flow was measured at the level of the pump.

High-precision scales (0.0001 g) were also required to make the pre- and post-test weight measurements.

There were two filters:

- one Sartorius brand cellulose nitrate filter with a diameter of 50 mm.
- one glass fibre filter with a diameter of 5 cm. (Labomoderne: LMR/PVF/50 GF92).

3. Operating procedure

The test parameters were as follows:

- drum rotation speed: 30 rpm
- air flow: 4 litres/min.
- measurement time: 5 minutes
- two filters were placed in the filter holder. The dust was carried along in a direction that took it first against the cellulose filter, and then against the glass filter. The glass filter was mainly included to protect the apparatus.

The measurement procedure involved:

- cleaning the apparatus
- weighing out the desired quantity of additive: m_a(g)
- introducing the additive into the dust-generation drum.
- closing the drum
- placing the filters in the filter holder
- weighing the filter holder: m_1 (g)
- switching on the pump
- switching on the motor that rotates the dust generating drum
- stopping the assembly at the end of the measurement time
- weighing the filter holder: m_2 (g)

Weighing the whole filter assembly limits the dust loss that would occur by removing the cellulose filter from the filter holder.
The cellulose filter was changed after each measurement. The glass fibre filter was changed after every third measurement, or less if it showed clear signs of soiling.

4. Interpretation of the results

The three weighings (those of the product and the filter) should be made as accurately as possible. The difference between the two filter holder weighings $m_2 - m_1$ gave the mass of the collected dust.
The concentration was expressed in milligrams of dust per gram of additive using the equation:

$$C = \frac{(m_2 - m_1) \times 1000}{m_a}$$

According to Heubach, if the mass found on the filter was below one gram, the measurement could be considered valid. Otherwise, the measurement would have to be repeated with only half the product quantity. Note, however, that this comment referred to a subsequent measurement of active agent concentration in the collected dust.
The result of the measurement of the mass of dust collected on the filter can be interpreted as follows: the greater the mass of collected dust, the more the product is likely to generate dust during manual or mechanical handling in the circuits.

5. Mass of the test portion

Using five representative additives, preliminary tests were carried out to determine the optimum quantity of additive, between 10 and 50 g, that would provide a meaningful result.
This method is limited by the presence of additives with highly variable dust concentrations. For additives with low dust concentrations (Figure 2-products 2 and 4), increasing the test portion had very little effect on the quantity of dust collected in the filter holder. For these additives, the protocol would focus on increasing the size of the test portion in order to limit weighing errors.

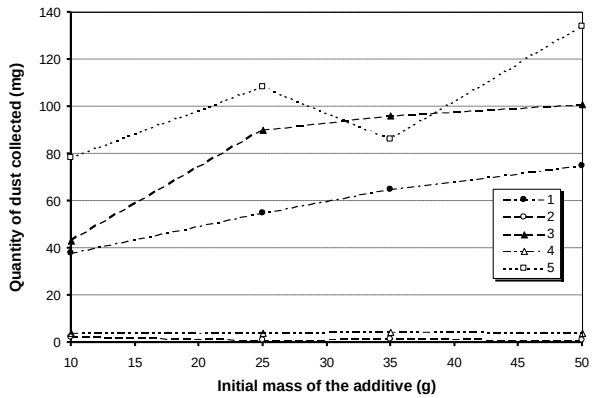

Figure 2: Change in the quantity of collected dust in relation to the initial quantity of the test portion for five additives

For additives with a high dust concentration (products 1, 3 and 5), the rate at which a quantity of collected dust increases declines on reaching the limit value of the test portion. It would appear that above a certain quantity of dust, the filter becomes partially clogged and is no longer able to capture all the dust carried in the air flow. This dust is then deposited in the bottle, giving a default measurement. The fraud prevention agency considers that the type of filter used can capture up to 300 mg of product. This value was not exceeded in these tests, despite the filter limit appearing to have been reached for products 1, 3 and 5. In addition, it would appear that not all the additives, which were of identical initial mass, reached this filter clogging limit value, further complicating the task of identifying a benchmark mass for a single protocol.

However, with the exception of one plot point (the 35-g test portion in product 5), which was perhaps due to a measurement error, the additives' dust rate ranking applied regardless of the mass of the test portion.

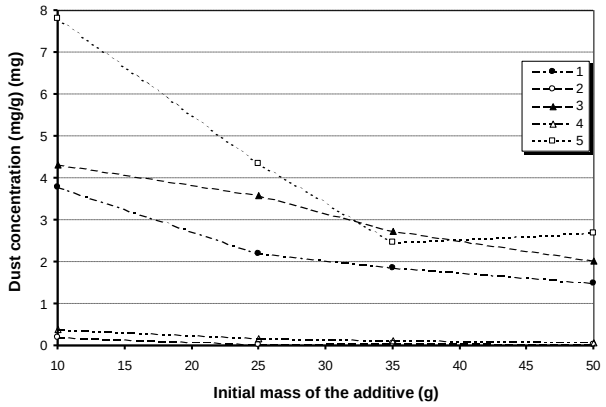

Figure 3: Change in dust concentration in relation to the initial quantity of the test portion for five additives

A mass of 25 g could offer a compromise between measurements of additives with high or low dust concentrations. At this mass (Figure 3), additives with high dust concentrations were found on separate levels, and none of them reached their limit value. Conversely, the two additives with low dust concentrations were not sufficiently distinct, and the quantities of collected dust, always very low, were subject to significant handling errors.

6. Intrinsic properties

The method's intrinsic properties were assessed based on the results expressed as a concentration of dust. The method's sensitivity, accuracy and reproducibility were assessed based on an experiment plan that proposed measuring five additives that were representative of the additives currently used in the animal feed industry. These measurements were taken over four separate and non-consecutive days, and repeated three times each day.

Despite the changes made to the manufacturer's protocol, the results were decidedly average: good sensitivity, but poor accuracy (14%) allied with doubtful reproducibility. This poor reproducibility would be mainly due to overlaps between additives 2 and 4, and to the variation in the quantity of dust generated by product 5 from one day to the next. This has a significant impact on the overall mean.

Additives	1	2	3	4	5
Concentration (mg/g)	2.18	0.03	3.59	1.61	4.42
Group	*c*	*d*	*b*	*d*	*a*

Table 1: Results of the test on the method's intrinsic properties

There are several possible reasons for these poor results. The focus of our study, which sought to identify a method with a single protocol that would be valid for all animal feed additives, implied the acceptance of errors in either the upper or lower part of the range.

The variance between additives 1, 3 and 5, on the one hand, and additives 2 and 4, on the other hand, made it possible to verify the assumed non-homogeneity of variances over the whole domain. This observation casts doubt over whether the results of the variance analysis had been correctly interpreted.

The results were therefore expressed in logs in order to correct this effect and convert them into a useable format. Once this was done, the variances tended towards greater homogeneity.

This improves the results to a slight degree, although the enhanced accuracy still lies below 5% (11.1%). The method is both sensitive and reproducible. Observing the variation from one day to the next revealed that the variations in additive 2 (low dust content) exceeded those recorded for additive 5 (high dust content) prior to the log-transformation. This transformation therefore maximises the weighing errors that occur in products that are low dust generators.

The inaccuracy of this method, and the variation in dust concentrations in relation to the size of the test portion, makes it unreliable. However, there is no better method currently available for measuring dust quantity directly.

7. Range of additives

A sample of 30 additives that are representative of the additive population used in animal feeds showed a fairly even distribution around a mean dust concentration of 1013.6 mg/kg (Table 2 and see i'Tec_Q2) and 5.8 when the results for this population are converted to a logarithmic scale.

	mg/kg	log
Mean	1013.6	5.8
Standard deviation	1508.4	1.8
Minimum	6.7	1.9
Maximum	6985.3	8.9
Min./max. difference	6978.6	7.0
Median	364.7	5.9

Table 2: Statistical report on the range of 30 representative products

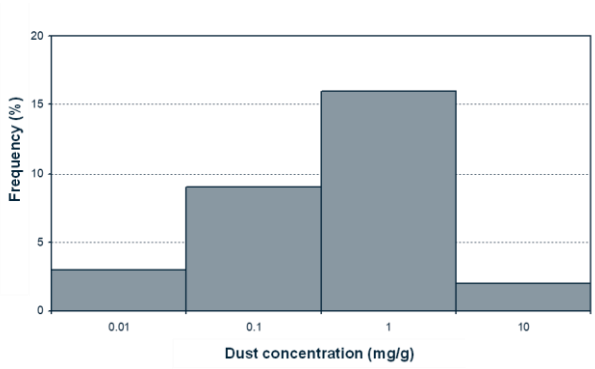

Figure 4: Frequency histogram

If not shown as a log, there would be over 1000 degrees between the minimum and maximum values. The two additives that generate approx. 10 mg of dust per gram are both vitamins (Figure 4).

8. Redundancy

Particle size measurement (laser diffraction) was the only link identified during a review of the relationships with other measurements of physical powder properties. This suggested that products with the smallest particle size are most likely to generate dust. Given the tenuous nature of the relationship, this particle size measurement does not, however, take all the parameters into account. It will, therefore, mainly be used for exploratory purposes in the dust detection domain.

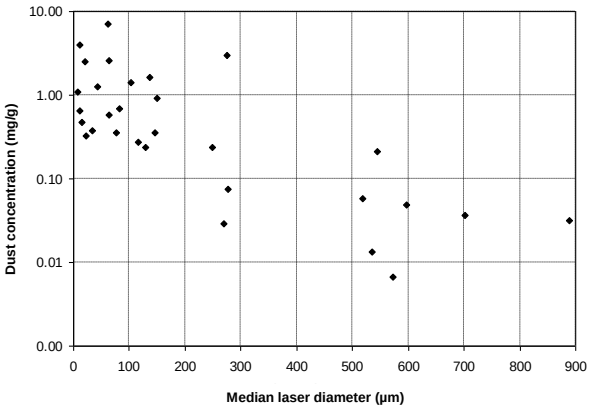

Figure 5: Relationship between dust concentration (mg/g) and median diameter (µm)

9. Conclusions

Firstly, it should be noted that there is a defect in the measuring apparatus. The position of the flowmeter makes it impossible to detect a leak on the air intake circuit.
Secondly, the necessity of cleaning the whole apparatus between measurements allied with the poor level of accuracy and the handling difficulties means that, even with improvements, this method would be fairly unworkable for plant acceptance tests.
However, this conclusion does not prejudice the method being used to collect dust intended for measuring a product's active substance content. This method is also the only one that currently provides a means of measuring dust directly. At least three measurements are made in order to keep results costs low, which might not be sufficient to get truly reliable results.

10. Bibliography

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