

PHYSICAL METHOD

Assessment of free floating dust and abrasion particles of treated seeds as a parameter of the quality of treated seeds

HEUBACH TEST

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HEUBACH TEST

1. Scope

The described method is intended to assess the amount of free floating dust and abrasion particles of treated seeds under defined mechanical stress conditions.

2. Principle

Treated seeds are mechanically stressed inside a rotating drum. A vacuum pump creates an air flow through the rotating drum, the connected glass cylinder and the attached filter unit. By the air flow, abraded dust particles are transported out of the rotating drum through the glass cylinder and subsequently through the filter unit. Coarse non-floating particles are separated and collected in the glass cylinder while floating dust particles are deposited onto a filter. The amount of floating dust collected on the filter is determined gravimetrically.

3. Apparatus

3.1 Analytical balance (accuracy 0.1 mg). As the last digit of a scale carries a larger error it is recommended to use a 5-decimal scale to achieve an accurate reading of the 4th decimal.

3.2 Heubach Dustmeter device (Heubach GmbH, Heubachstrasse 7, 38685 Langelsheim, Germany)

3.2.1 Metal rotating drum

3.2.2 Glass cylinder

3.2.3 Non-electrostatic filter housing with conditioned glass fiber filter disc (Whatman GF 92 or

Macherey Nagel Type MN 85/70 BF, or equivalent specification)

3.2.4 Drive & control unit with touch screen control panel

3.3 Constant climate chamber (e.g. Binder, KBF 720)

3.4 Paper bags (not airtight)

3.5 Air ionizer (e.g. Sartorius, STAT-FAN YIB-01, or PRX U field ionizner from Haug GmbH, Germany, or equivalent)

3.6 Seed Counter (e.g. Pfeuffer, Contador or GTA Sensorik, Marvin, or equivalent)

4. Sample

A sample must consist of at least 500 g +/- 5% of seeds taken from the seed flow in a representative manner, at best by automatic continuous sampling. The thousand grain weight (TGW) of the seeds must be known and registered.

5. Procedure

5.1 Safety Advice

This test is designed to potentially produce significant amounts of fine dust particles which are loaded with active ingredients (a.i.'s). Therefore, before performing this test it must be ensured that all measures are taken to prevent exposure of any personnel to dust liberated during the test (incl. preparation, cleaning procedure and disposal of any material). Special care must be taken that potentially polluted exhaust air does not harm people and environment.

In addition, technical personnel must be properly trained in the safe use of all equipment and must be instructed on possible hazards resulting from the test (see also note 8.5). Adequate protective equipment must be used in order to avoid exposure to dust.

5.2 Laboratory Conditions

The test has to be performed in a laboratory (separated from the treating area) at 20°C to 25°C and 30% to 70% relative humidity which is free of free floating dust particles. Any other testing which could interfere with the analytical scales (electrostatics, vibrations etc.) should be avoided.

5.3 Calibration

No calibration is necessary before measurement. It is recommended that the air flow rate, time of measurements and rotational settings are checked on a routine basis (every 2-3 years) by the technical service of the manufacturer or an equivalent qualified technical service. If dust has been sucked by accident through the flow meter, it must be cleaned and a new calibration may be necessary.

5.4 Apparatus preparation

During initial installation of the Heubach equipment make sure that the same is horizontally levelled.

After disconnecting the vacuum tube from the filter unit, the Heubach device is stepwise disassembled: the filter unit is removed and opened, the glass cylinder removed and finally the metal drum removed and opened.

Make sure that all components which are in contact with seed or dust (i.e. rotating drum, glass cylinder, filter unit including rubber O-ring) have been thoroughly cleaned. Cleaning is routinely done using a vacuum cleaner with a pointed nozzle. (see 8.1).

Note: If the drum is either used for the first time in this test or has been cleaned with alcohol there is a need to run 2 cycles with treated seeds before starting the actual measurements on your samples. This ensures a constant occupancy of the pores in the metal surface.

Switch on the main power of the Heubach device min. 30 minutes before starting any measurement in order to allow proper warm-up of the flow meter. For setting the parameters on the control panel choose the program "User Method" in which the parameters are manually set to the values given in 6.2. It is recommended to set the parameters after full assembly of the device.

5.5 Sample Preparation

Prior to testing, seed samples have to be stored in a constant climate chamber for at least 48 hours (2 days) at $20^{\circ}\text{C} \pm 2^{\circ}\text{C}$ and at $50\% \pm 10\%$ relative humidity. To allow equilibration, seeds must be kept in paper bags (not airtight) when entering the climate chamber.

For obtaining a working sample a soft method should be used to reduce the submitted sample in size to the size needed for the test. This to avoid damage to the treated seed which could lead to artificially enhanced dust levels. Examples of a soft methods are the modified halving method, the spoon method and the hand halving method described in the ISTA Rules.

5.6 Measurement

Carefully transfer (avoid dust) 100 ± 1 grams of the conditioned seeds (weight seeds w_s [g]; accuracy: 0.01 gram) into the metal drum of the Heubach device, then correctly close and reassemble the drum and connect the glass cylinder. The system has to be leveled perfectly horizontally and no obstruction of the rotating parts and of the internal or external airflow must take place.

The time for transferring and analyzing the sample is to be kept as short as possible in order to avoid a change in its relative humidity. A contamination with non-seed dust particles must be excluded.

Place a glass fiber filter disc (Whatman GF 92 or similar specification) in the filter unit according to the description in the manual. For equilibration with the laboratory conditions, the filter discs will be stored in an open box next to the Heubach device. In order to prevent effects resulting from electrostatic charging, the use of a non-electrostatic filterhousing offered by HEUBACH is compulsory to use. The filter unit including the filter disc is weighed (weight filter assembly w_0 [g]; accuracy: 0.1 mg; see note 3.1), placed on the glass cylinder and connected to the vacuum tube.

On the control panel pre-select the "time" option. Start the rotation cycle by pressing "1" on the control panel. After completion of the run, the rotation must have fully stopped before any parts of the apparatus may be disassembled. Remove the filter unit including the filter disc carefully from the glass cylinder and weigh it in the same manner as described before (weight filter assembly w_1 [g]; accuracy: 0.1 mg).

If significant amounts of dust have passed *through* the filter disc (by visible inspection), the test must be stopped immediately and the filter unit checked for incorrect assembly or damages. If necessary, it has to be replaced and the test has to be repeated.

The test has to be performed twice. After each measurement, the apparatus must be cleaned as described in section 5.4.

If the rotation speed (rpm) displayed on the control panel during the measurement deviates more than $\pm 10\%$ from the pre-set value or if the total air volume sampled during the measurement deviates more than $\pm 10\%$ from the expected volume of 40 L (20 L/min for 2 min) the measurement has to be redone.

As a back-up control for the air volume a separate flow-meter [e.g. DFM Typ SVB (Uniflux 1/4") from VAF-Fluid-Technik GmbH, Germany; www.vaf-fluidtechnik.de] can be inserted in the plastic air hose, as shown in Appendix 6.

5.7 Evaluation and Calculation of Results

The Heubach dust value is expressed in g / 100 kg of treated seeds. Depending on requirements and seed type tested, the result can be also expressed in g / 100.000 kernels, taking into account the Thousand Seed Weight (TSW) of the tested sample.

Use the following formula to convert the measured result to the Heubach dust value:

$$\text{Heubach dust value} = \frac{(W_1 - W_0) \cdot 100.000}{W_s} \quad [\text{g} / 100 \text{ kg}]$$

or alternatively expressed in g / 100.000 kernels :

$$\text{Heubach dust value} = \frac{(W_1 - W_0) \cdot 100 \cdot \text{TGW}}{W_s} \quad [\text{g} / 100.000 \text{ kernels}]$$

where:

W_1 = weight of the loaded filter unit incl. filter disc [g]

W_0 = weight of the empty filter unit incl. filter disc [g]

W_s = weight of the treated seeds [g]

100.000 = conversion factor a []

100 = conversion factor b []

TGW = Thousand Grain Weight [g / 1000 kernels]

The final result is the mean of the two measurements. If a threshold value is defined the test must be repeated if one test result is higher than 50% of the threshold value and if the two test results differ more than 20% from each other. In case of experimental or voluntarily purposes without a mandated threshold value (e.g. small seeded crops) the test must be repeated if the two test results differ more than 20% and at least one test result exceeds 1 g/100 kg. If both test results are below 1 g/100 kg and the two results differ more than 0.2 g the test must be repeated.

6. Results

6.1 Results from the measurement

Heubach dust value = *** [g / 100 kg] or
Heubach dust value = *** [g / 100.000 kernels]

6.2 Parameter settings

Rotation speed = 30 [rpm]
Rotation time = 120 [seconds]
Airflow rate = 20 [liters per minute]

Pre-Selection: set to "time"; set to "rotation speed"; set to "airflow rate".

6.3 Accuracy and Measurement Errors

The measurement accuracy can be strongly influenced by electrostatic charging, contamination with non-seed dust and absorption of humidity from the air. In addition, loose dust particles which are already contained in the seed sample can strongly affect the result.

7. Analysis report

The minimum requirements to report in the analysis report are:

- Name and coordinates of the laboratory
- Reference number of the sample
- Date of receipt/arrival of the sample in the laboratory
- Date of analysis
- Date of issuance of the analysis report
 - condition (packaging, i.e. sample not tightly packed or damaged)
- Results as described in 6.1
- Signature or electronic validation by responsible person of the laboratory

The analysis report has to be released.

8. Notes

8.1 The vacuum cleaner must qualify for the safe use with hazardous chemicals (especially hazardous dust particles, e.g. Kärcher Nt 35/1 Eco H).

8.2 The handling of the filter unit during assembly and weighing should always be done wearing laboratory gloves (e.g. nitrile type) to avoid grease depositions from hands which would affect the weight.

8.3 Warning: the seeds you are working with are treated with active ingredients. Always wear adequate Personal Protective Equipment (PPE). Please take the necessary actions which are determined by your local laws to work with these type of products. For the disposal of the seeds and other contaminated materials check the local requirements by law.

8.4 Method extension: In case the content of active ingredient(s) in the floating dust should be determined this is possible by appropriate extraction of the filter disc and subsequent chemical analysis (e.g. HPLC) using an appropriate analytical method. Such analytical method(s) which has to be developed and validated in accordance with valid guidelines are specific for one or several active ingredient(s) and can not be described in frame of this method.

8.5 Material Safety Data Sheets for each product used in the test item (treated seeds) must be available at the test location and must be consulted before any test. Furthermore, safety requirements concerning exposure to dust must be strictly observed.

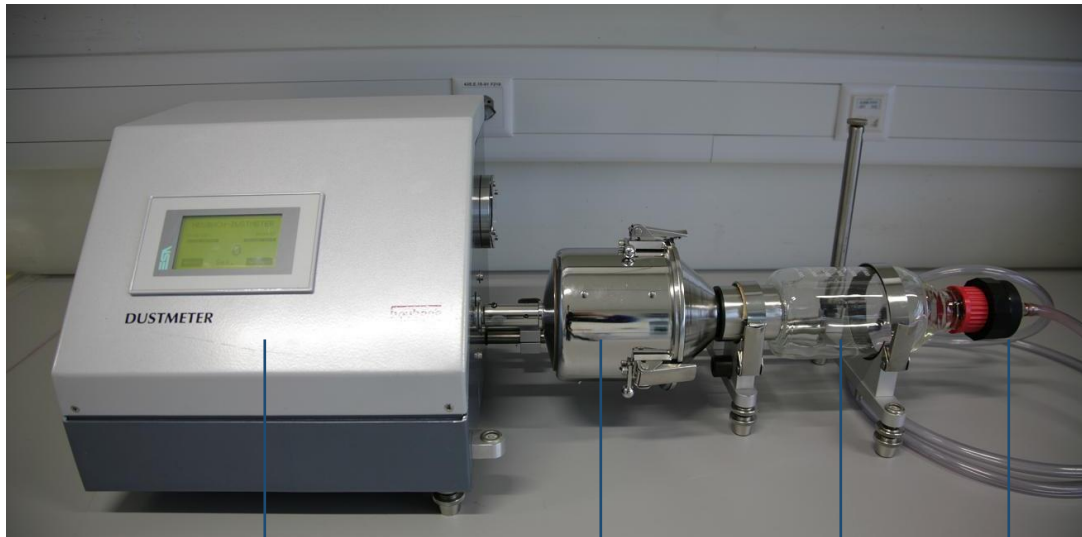
9. LITERATURE

Manual to the Heubach device (“Dustmeter Instruction Manual”)

10. APPENDICES

- Appendix 1 Heubach Dustmeter Device (Overview)
- Appendix 2 Analytical Balance
- Appendix 3 Touch-screen Panel and Rotating Drum
- Appendix 4 Filter housing developed by BCS
- Appendix 5 Examples for Deposited Abraded Dust
- Appendix 6 External flow-meter

Appendix 1: Heubach Dustmeter Device (Overview)



Drive & control unit

Rotating drum

Glass cylinder

Filter unit

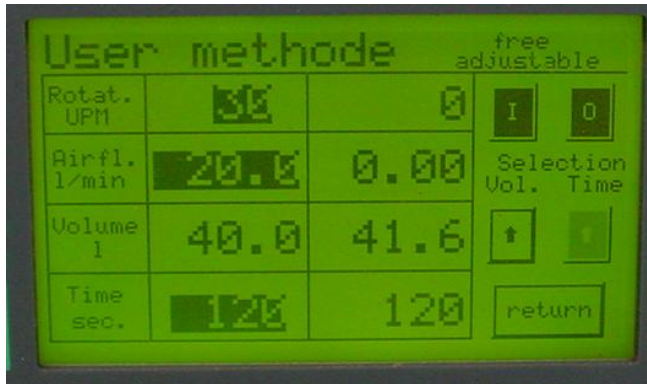
Appendix 2: Analytical Balance



Filter Unit (open, without filter disc)

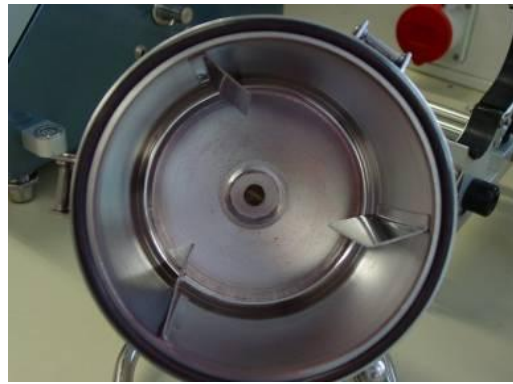


Appendix 3: Touch-screen Panel and Rotating Drum



Touch-screen for parameter setting:

- Rotation speed (rpm)
- Airflow (L/min)



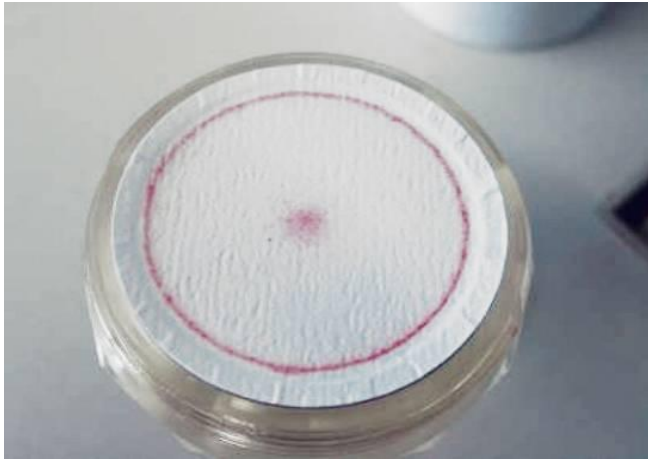
Rotating drum for seed

Appendix 4: Black Filter housing (non-electrostatic)



Filter Unit (open, with filter disc and O-seal)

Appendix 5: Examples for Deposited Abraded Dust



Little Dust



Higher Amount of Dust

Appendix 6: External flow-meter

