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Fertilizers - Determination of dust content

Düngemittel - Bestimmung des Staubgehaltes

Engrais - Détermination de la teneur en poussière

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Fertilizers - Determination of dust content

Engrais - Détermination de la teneur en poussière

Düngemittel - Bestimmung des Staubgehaltes

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EUROPEAN COMMITTEE FOR STANDARDIZATION
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European foreword

This document (CEN/TR 14061:2021) has been prepared by Technical Committee CEN/TC 260 “Fertilizers and liming materials”, the secretariat of which is held by DIN.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN shall not be held responsible for identifying any or all such patent rights.

This document supersedes CR 14061:2000.

Significant changes between this document and CR 14061:2000 are as follows:

a) modification of the figures to contain neutral language;

b) adaption to current principles and rules for structure and drafting.

This document is published by the European Committee for Standardization. It is published for information only and does not have the status of a European Standard.

The Annexes A and B are informative.

Introduction

0.1 General

In production and handling of fertilizers dust generation is of great concern by both producers and users of the fertilizer products. For health and environmental reasons, it is of great interest to control and reduce the amount of dust generation. In the fertilizer industries there are a wide variety of apparatus for dust determination, most being used as "in-house" methods in plants and laboratories.

The content of this document was developed by CEN/TC 260/WG 2 between 1991 and 2000 in order to develop a standard dust test. A spouting bed apparatus was designed for gravimetric determination of dust, and after two preliminary ringtests a conclusive ringtest involving six laboratories was carried out. Not being able to develop a statistical significant method for the determination of dust, TC 260 decided by resolution 105/1997 to change the deliverable of this work item into a CEN Technical Report. The change of deliverable has been approved by CEN/BT with its resolution BT C172/1999.

0.2 General background

When handling fertilizer grains, dust is at every moment generated on the surface. The fertilizer thus contains more or less free dust, and has a potential for generating more dust (abrasion dust) when subject to subsequent handling.

In all existing gravitational test methods dust will be generated during the testing time, and the two types of dust will be measured simultaneously. The scope of the method is expressed in Annex A and the aim is to:

"...specify a method for the determination of the ***dust potential*** *of solid fertilizers and is applicable to granular and prilled fertilizers.*

Dust particles, which cause reduced visibility in air are too small to be determined by this method."

0.3 Background for choice of method

Fluidized particle powders are generally divided into four characterizing groups (A, B, C, D) [1]. Group C particles are small, cohesive and are difficult to fluidize. Aeratable powders belong to group A, and many fluidized bed catalysts characterize this group. Sand typifies group B, in which inter-particle forces are negligible, in contrast with group A powders. Large and/or dense particles in general belong to group D, and fertilizer particles (2 mm to 4 mm) in air are in this group. A flow diagram can be used to broadly identify flow regimes appropriate to combinations of gas velocity and particle properties. It can be shown that the fertilizer system is in the lower part of the spouted bed regime.

A criterion that can be used to distinguish between group B and D is the numerical inequality that classifies a powder as spoutable if:

$$(\rho_p - \rho_f) \cdot d_p^{1,24} > 0,23$$

For a typical fertilizer this value will be about 1,4 and about 0,5 for an urea prill.

From previous experiments with other methods based on a fluidized bed and the above calculations, it was decided to base the method upon the spouted bed principle.

1 Scope

This document is applicable to the determination of dust potential of solid fertilizer, obtained in prilling or granulation process. Compacted or crystalline materials were not considered.

2 Normative references

There are no normative references in this document.

3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

4 Symbols and abbreviated terms

4.1 Technical Symbols

C_D	drag coefficient
d_p	particle diameter, expressed in metres (m)
d_s	average spout diameter, expressed in metres (m)
D_p	average particle diameter, expressed in metres (m)
D	diameter of spouting section, expressed in metres (m)
D_i	inner orifice diameter, expressed in metres (m)
g	gravity, expressed in kilograms per metres per square seconds (kg/m s^2)
H	bed height, expressed in metres (m)
Re	Reynolds number
v_t	terminal velocity, expressed in metres per seconds (m/s)
v_{ms}	minimum spouting height
ρ_p	particle density, expressed in kilograms per metres to the third power (kg/m^3)
ρ_f	fluid density, expressed in kilograms per metres to the third power (kg/m^3)
μ	viscosity, expressed in Newton seconds per square metres (Ns/m^2)

4.2 Statistical symbols and abbreviations

df	degrees of freedom
F	mean square between groups/mean square within groups
F_{crit}	tabulated value form the F-distribution for a significance level of 0,05 confidence interval
MS	mean square
P-value	significance level corresponding to a given F (should be less than 0,05 to reject the null-hypothesis)
SS	sum of squares

5 Calculation of the spouting bed apparatus

5.1 Particle terminal velocity

A particle falling freely in a fluid will finally reach its terminal velocity. The forces acting on it are gravitational, accelerating, buoyancy force and drag (friction) force. The drag force can be expressed by a drag coefficient C_D, which is expressed by Formula (1):

$$C_D = \frac{4}{3}\frac{\left(\rho_p - \rho_f\right) d_p g}{\rho_f v_t^2} \qquad (1)$$

By calculation and plotting log C_D against log Re (Reynolds number) the so-called "standard drag-curve" can be obtained which has three broad regions:

— Laminar region, $Re < 0,2$;

— Transitional region (tr), $0,2 < Re_{tr} < 1000$;

— Turbulent region, $Re > 1000$.

The drag coefficient equation can be multiplied with $\rho_f^2 v_t^2 d_p^2 / \mu^2$ and rearranged as:

$$C_D Re_t^2 = \frac{4}{3}\frac{\left(\rho_p - \rho_f\right) d_p^3 g}{\mu^2} \qquad (2)$$

The group $C_D Re_{tr}^2$ is dimensionless containing only the physical properties of the particle/fluid system including the particle diameter d_p. The Re-number and the terminal velocity (v_t) can be estimated by graphical methods.

Calculations prove that transitional flow describes the system of fertilizer dust in air, thus giving Table 1.

Table 1 — System of fertilizer dust in air

Particle size d_p µm	$C_D Re_{tr}^2$	Re_{tr}	v_t m/s
100	88	3,0	0,5
150	300	7,7	0,8
200	704	15,0	1,3

The air velocity was chosen to be 0,75 m/s in the classification section (110 mm ⌀) of the apparatus, and irregular particles less than 150 µm will then be carried over, according to calculations.

5.2 Spouting section

The spouting section is characterized by the "minimum spouting height", v_{ms}, that depends on the particle (fertilizer) properties, spouting column geometry and the inlet orifice diameter:

$$v_{ms} = \left(\frac{d_p}{D}\right)\left(\frac{D_i}{D}\right)^{1/3}\left(\frac{2gH\left(\rho_p - \rho_f\right)}{\rho_f}\right)^{1/2} \quad (3)$$

Based on 500 g fertilizer, v_{ms} = 1,0 m/s and diameter D = 85 mm of spouting section, the theoretical expression of v_{ms} [1] was rearranged. Inner orifice diameter D_i:

$D_i^{1/3}$= 5,645 10^{-4} d_p

thus giving the figures:

d_p	2,0 mm	3,0 mm	4,0 mm
D_i	22,5 mm	6,6 mm	2,8 mm

Depending on the average particle diameter (d_p) the inner orifice diameter (D_i) should thus be varied, according to the theory.

5.3 Maximum spoutable bed height

The maximum spoutable bed height (H_s) can be estimated from the correlation:

$$H_s = 0{,}345\ (D^2 - d_s^2) \cdot D^{0{,}384} \cdot d_s^{-1{,}384} \quad (4)$$

where d_s is the average spout diameter. D = 85 mm and estimated d_s = 15 mm gives H_s ~ 38 mm, which is higher than the chosen bed height. However, the calculation assumes spherical particles, and practical maximum spoutable depth will therefore be lower than the theoretical value.

Based on the calculations above the spouting bed apparatus was designed and tested.

5.4 Design of apparatus

The column was designed with the dimensions according to Table 2.

Table 2 — Design of apparatus

Classification section		Spouting section	
Column diameter	110 mm	Column diameter	85 mm
Column height	400 mm	Column height	120 mm
Outlet diameter	40 mm	Cone height	85 mm
Air velocity	1 m/s	Total height (incl. bottom inlet)	220 mm
Fertilizer mass	400 g	Cone inlet diameter	23 mm[b]
Air rate	25 m^3/h[a]	Air velocity (overall)	1,2 m/s

[a] The air velocity in the classification section was chosen to be 0,75 m/s in order to carry 150 m particles over (see 5.1).

[b] Adapters with diameters 7, 8, ...,18 mm were made to include most fertilizers. A 440 m grid was fitted into the adapter inlet.

5.5 Flowmeter

A calibrated flowmeter is connected to the column. The flowmeter should have a capacity of approximate 40 m^3/h.

6 Initial testing

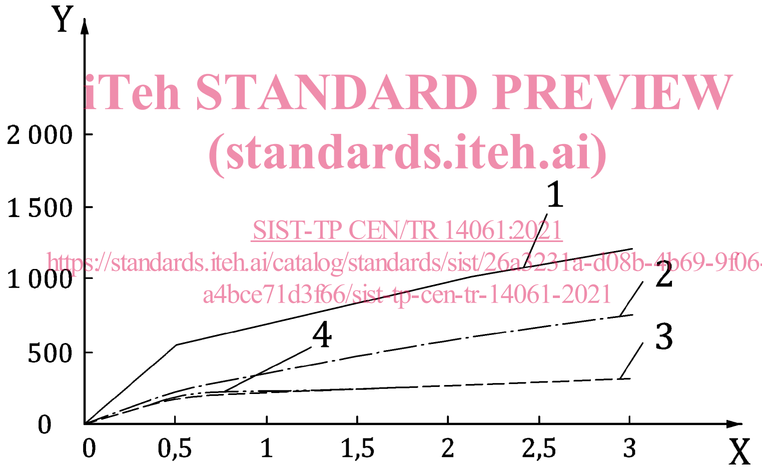
6.1 Determination of dust weight

Initially the dust was collected by a filter at the outlet of the apparatus. However, because of safety (pressurized air in the glass apparatus) and inaccuracy in measurements due to accumulation of dust on column walls, it was decided to record the difference in weight of the fertilizer sample during the test.

6.2 Setting the test time

Initial tests were carried out in order to set the test-time. Dust generation of selected NP/NPK-fertilizers were measured at increasing time intervals.

Figure 1 shows a decreasing slope at approximate 0,5 min test time which is due to a change from free dust to abrasion dust. In order to include approximately the same amount of free dust as abrasion dust, a 2 min test time was chosen.



Key

X time (min)

Y dust (mg/kg)

1 blended NPK

2 granulated NP

3 granulated NPK

4 prilled NPK

Figure 1 — Dust generation as function of time

6.3 Preliminary ringtests

Two preliminary ring tests were run in order to improve the method.

7 Conclusive ring test

7.1 General

A final and conclusive ring test was run with six participating laboratories involved. Ten replicates of five fertilizers were tested at each laboratory and statistical results calculated by ANOVA.

7.2 Apparatus

The apparatus is described in Annex A.

7.3 Sample preparation

The ring tests were conducted using the following five types of homogenous fertilizer products: granulated urea; granulated CAN; granulated PK; granulated NPK; prilled NPK. The relevant producer of each fertilizer sent 12 separate samples (10 as required for the tests plus 2 spares in case a test had to be aborted) to the participant laboratories.

7.4 Procedure, test plan

The drafted test procedure is enclosed in Annex A. Ten replicates were tested for all five fertilizers.

7.5 Statistical methods

7.5.1 Statistical model

Each test result, y, is the sum of four components: $y = m + A + B + e$

where m is the general average, A is the adapter diameter used, B is the between-laboratory variation and e is the random error occurring in every test.

The model for sample j at laboratory i is: $y_{ij} = m + b_0 A_i + b_i + e_{ij}$

where b_0 and b_i are regression coefficients and A_i is the adapter diameter used in laboratory i.

7.5.2 Outliers

Assuming that the statistical model is correct, the residuals, e are normal distributed. A normal-plot is used to check for normality.

7.5.3 Regression analysis

In the regression analysis the outliers are removed. The regression analysis gives one significant PLS component (one-component explains model).

7.5.4 Correction for adapter-effect

After regression analysis the effect of chosen adapter was removed, and variance within laboratory and between laboratories were analysed.

7.5.5 ANOVA-analysis

The ANOVA-analysis performs simple analysis of variance, which tests the hypothesis that means from several samples are equal. The confidence-level is set to 95 %. Generally, analysis of variance, or ANOVA, is a statistical procedure used to determine whether means from two or more samples are drawn from populations with the same mean. This technique expands on the tests for two means, such as the *t*-test.