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Gnojila - Določanje odpornosti granul gnojil proti drobljenju

Fertilizers - Crushing strength determination on fertilizers grains

Düngemittel - Bestimmung der Kornfestigkeit auf Düngemittelkörnern

Engrais - Détermination de la résistance à l'écrasement des engrais

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Fertilizers - Crushing strength determination on fertilizers grains

Engrais - Détermination de la résistance à l'écrasement des engrais

Düngemittel - Bestimmung der Kornfestigkeit auf Düngemittelkörnern

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EUROPEAN COMMITTEE FOR STANDARDIZATION
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European foreword

This document (CEN/TR 12333:2021) 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 12333:1996.

Significant changes between this document and CR 12333:1996 are as follows:

a) 24/8/8, 13/13/21 and 0/10/20 changed to NPK 24+8+8, NPK 13+13+21 and PK 10+20, respectively;

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

The present report arises from work done by CEN/TC 260/WG 2 under the work item “Crushing strength determination” with the view of producing a European Standard.

Unfortunately, after several ring tests, results appeared to be so randomly distributed that a statistically significant interpretation showing good reproducibility and repeatability could not be obtained.

Tests on correlation have also shown that no significant correlation as to the causes of the dispersion of the results could be obtained within the proposed time scale and probably not without expending too much effort, time and money.

Therefore, since the results obtained may be added to those of past publications in the literature, CEN/TC 260 decided the results obtained should not be ignored by the scientific community.

Introduction

The resistance to crushing (crushing strength) of fertilizer is an important property used in quality control of fertilizer production. It influences the storage and handling as well as spreading properties of fertilizer.

However, crushing strength of fertilizer grains is not a constant property *stricto sensu*. Crushing strength is significantly influenced by the content of free water in the fertilizer, humidifying and drying during storage, as well as by temperature changes. Time dependent processes in the grain and bulk during the lifetime of the grains may also affect the crushing strength.

Nevertheless, there are several “in house” methods used for the measurement of crushing strength of fertilizer grains which give different results. There is also a need for a standardized method.

This document reports the results of the international ring test, organized by CEN/TC 260/WG 2 (physical properties), carried out 1992, with the purpose of standardizing the measurement technique of crushing strength.

1 Scope

This document is applicable to crushing strength measurement as applied to grains of fertilizer obtained in prilling or wet-granulation process. Compacted or crystalline materials were not considered.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 565:1990, *Test sieves — Metal wire cloth, perforated metal plate and electroformed sheet — Nominal sizes of openings*

ISO 5725:1986, *Precision of test methods — Determination of repeatability and reproducibility for a standard test method by inter-laboratory tests*[1]

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

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/

3.1
crushing strength of grains
force necessary to destroy the grain as such

4 Principle

The measurement of the force applied for the destruction of the grains may be carried out by constant load or constant speed.

NOTE Not all manufacturers of apparatus can give information about the principle (constant speed or constant load) used.

It is not known which of the first "peak" or maximum of force applicable on the grain is measured.

The grains undergoing the measurement were of the same size.

5 Apparatus

5.1 Sieves

The sieve is in accordance with ISO 565:1990 for the grain size chosen. In this ring test, the grain size was 2,8 mm (retained in the mesh of the sieve).

[1] ISO 5725:1986 has been withdrawn and replaced by the ISO 5725 (series) released between 1994 and 1998.

5.2 Crushing strength measuring apparatus

In this ring test, the following apparatus as given in Table 1 were used:

Table 1

	Compression speed mm/min		Direction
	low	high	
Alvetron	10		vertical
Dinamometro Ceast	5, 10, 20		vertical
Erweka THB 28	8	38, 48, 55	horizontal
Forcemeter	10		vertical
lnstrou 4502	10	55	vertical
Matec Accuforce II	10		vertical
Mecmesin	10	55	vertical
Salter EFG-2	10	55	vertical

To investigate the influence of compression speed and/or direction of the crushing force applied, in this ring test both low and/or high compression speed were allowed in both directions: horizontally and vertically.

6 Sample preparation

The ring test included products with high and low elasticity. The following products or formulations have been chosen:

Urea; AN; NPK 24+8+8; NPK 13+13+21; PK 10+20.

The test was taken with 5 × 25 = 125 grains of each formula. The grain size was d = 2,8 mm (in order to be retained in the mesh of the sieve).

To carry out the test at two different compression speeds, $N \geq 250$ grains were sent to the participant laboratories in February 1992. In this season no danger of damage by temperature change is expected.

The samples were sent to the participants well packed to avoid mechanical or wetting damage during transport.

7 Procedure

Follow the instructions of the manufacturer of the apparatus used. Carry out the measurements rapidly one by one on each of the grains.

8 Test report

The measured single values have been reported together with important details: temperature, relative humidity of the air, compression speed, type of apparatus used.

9 Statistical calculations

The references for the statistical calculations are in accordance with ISO 5725:1986[1] with some exceptions given in Annex A, Figure A.1.

The significant levels used in statistical calculations are:

Normal distribution-, in accordance with ISO 5725:1986[1], outlier- and *t*-tests are calculated, at $\alpha = 0{,}01$ significance level.

Repeatability (*r*) and Reproducibility (*R*) are calculated, in accordance with ISO 5725:1986[1], at $\alpha = 0{,}05$ significance level.

9.1 For outlier tests and the calculation of repeatability (*r*) and reproducibility (*R*) the normal-distribution is required: there is a need for a test of normal distribution.

9.2 For normal distribution test both, the Gaussian distribution and the log-normal distribution were used. As test methods, the Kolmogoroff-Smirnow test and the Shapiro-Wilk test were calculated.

9.3 For outlier test on the single values of each data set the Grubbs-test was calculated. Outlier single values were deleted before calculation of the mean values ($\bar{x}$) and standard deviation (s).

9.4 Laboratory-outlier test on the mean, standard deviation and relative standard deviation were calculated for speed groups.

9.5 Before any influence of compression speed or direction of crushing force may be postulated, the mean values are compared if they are "statistically significantly different" or not. If they are not, no influence or regression may be postulated. The statistical method to compare mean value is the *t*-test.

10 Expression of results

10.1 For the more elastic fertilizer formulas Urea, AN, NPK 24+8+8 and NPK 13+13+21 the Gaussian-distribution seems to be the better approximation and for the most rigid formula PK 10+20 the log-normal distribution seems to be a better approximation.

10.2 Outlier-test for laboratories: $\bar{x}$, s.

There is no laboratory mean outside the control levels. Outlier-test on standard-deviation indicate nearly 30 % of the data sets as outliers.

At this calculation level no outlier laboratory was deleted.

10.3 The *t*-test for compression-speed groups allow the calculation of regression only for NPK 13+13+21. It is incomprehensible why especially this fertilizer formula is sensitive to compression speed and the other formulas not. Any dependence from AN-content or elasticity of the product is not apparent.

10.4 Mean $\bar{x}$, repeatability (*r*) and reproducibility (*R*) are listed in Annex B, Table B.1. The values *r* and *R* are, related to $\bar{x}$, too high to be acceptable for a normalized test method.

10.5 Outlier-test for Laboratories: relative standard deviation (s_{rel}).

It seems that *s* is not the adequate measure for outlier and s_{rel} will give formally a better result. Apparently, the crushing strength of the most elastic fertilizer Urea and AN are measured with less s_{rel} than NPK and PK, and PK with higher s_{rel} then NPK. Not one s_{rel} is calculated below 10 %.

For further calculations one of the participant laboratories was regarded as an outlier and was deleted.

10.6 The recalculated *t*-test result was not influenced by the deletion of the “outlier laboratory”.

10.7 Mean $\bar{x}$ repeatability (*r*) and reproducibility (*R*) were recalculated without the “outlier laboratory” (see Annex C, Table C.1). The result is similar to that given in Annex B, Table B.1: the values *r* and *R*, related to $\bar{x}$, remain too high for a normalized test method. The poor result in Annex B, Table B.1 is not caused by an outlier laboratory.

10.8 For further progress a postulated “anisotropic effect” was tested alone (see Annex D, Table D.1) and in combination with compression speed (see Annex E, Table E.1 and Annex F, Table F.1). There is no combination with better results as given in Annex B, Table B.1 and Annex C, Table C.1.

10.9 The results in more details were given in Annex F, Table F.1.

11 Conclusion

The results of this ring test were highly unsatisfactory.

For all fertilizer formulas and for all groupings, the calculated repeatability (*r*) and reproducibility (*R*) are too high in relation to the calculated mean values. It means that, at 95 % significance level, the methods for crushing strength measurement used in this ring test cannot distinguish good and poor quality of a fertilizer. A lengthy discussion of apparatus properties and construction principles, as well as measuring details given in the test reports did not encourage further investigations.

The problem is closely related with the high standard deviation of all data sets. That means, it is related to the internal structure of fertilizer grains which are deemed to be inconsistently anisotropic.