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Straight ammonium nitrate fertilizers - Comparative study on the determination of porosity (oil retention)

Reine Ammoniumnitratdünger - Bestimmung der Porosität (Ölretention)

Engrais simples a base de nitrate d'ammonium - Etude comparative de méthodes de détermination de la porosité (rétention fuel)

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Straight ammonium nitrate fertilizers - Comparative study on the determination of porosity (oil retention)

Engrais simples à base de nitrate d'ammonium - Etude comparative de méthodes de détermination de la porosité (rétention fuel)

This CEN Report was approved by CEN on 13 April 2002. It has been drawn up by the Technical Committee CEN/TC 260.

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EUROPEAN COMMITTEE FOR STANDARDIZATION
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EUROPÄISCHES KOMITEE FÜR NORMUNG

Management Centre: rue de Stassart, 36 B-1050 Brussels

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Foreword

This document (CR 14539:2002) has been prepared by Technical Committee CEN/TC 260, "Fertilizers and liming materials", the secretariat of which is held by DIN.

It is published for information only and does not have the status of a European Standard.

The annexes A to D are informative.

Introduction

Straight ammonium nitrate fertilizers of high nitrogen content (> 28 %), following Directive 80/876/EEC [1] and Directive 87/94/EEC [2], are subject to the particular regulatory requirement of a maximum porosity limit of 4 %. The currently used official EC method is demanding with respect to time and skilled laboratory manpower.

Therefore CEN/TC 260 agreed to launch a new Work Item, in order to compare non-standardized alternative methods for measuring porosity with the official one, through ring testing.

Parallelly, oil retention can also be determined by ISO 5313.

1 Scope

This CEN report gives the results of inter-laboratory testing to compare the accuracy and convenience of the official EC method for porosity measurement (given as Annex B) with two non-standardized alternative methods (given as Annexes C and D) already used in some participating laboratories.

Three products, with a porosity between 1 % and 7 %, have been used in the inter-laboratory trials.

2 Test procedure

2.1 Methods for measuring porosity

All the methods tested for measurement of ammonium nitrate porosity are based on immersion of the test sample in gas oil, removing the excess of oil, and finally determining the absorbed amount of oil through weighing. The methods differ in the way of removing the excess oil.

Table 1 — Methods for measuring porosity

Method	Removal of oil	Protocol
Method 1 : EC Method [2][a]	Hand-rubbing between sheets of filter paper	see Annex B
Method 2 : Centrifuge method	Draining and centrifuging	see Annex C
Method 3 : Roller drum method	Draining and roller drum with filter thimble	see Annex D
[a] This method is based on ISO 5313, with some minor but significant differences.		

WARNING — Appropriate safety rules and procedures should be followed while handling samples and residues of Ammonium nitrate impregnated with gas oil.

2.2 Products

2.2.1 Test samples: straight ammonium nitrate

Three different ammonium nitrate samples have been provided to all the participants. Two samples were fertilizer ammonium nitrates of the 33,5 % N type, the third was a special ammonium nitrate, with a higher porosity well over 4 %.

2.2.2 Gas oils

All gas oils used in the ring test conformed to the requirements of Directive 87/94/EEC, Annex II [2]. In order to improve the accuracy of the ring test, one specific gas oil, distributed centrally in this ring test, has been used as a reference by all participants, parallel to oils from local origin.

2.3 Ring test procedure

This ring test has been performed by 17 participating laboratories, private ones as well as official ones. Three ammonium nitrate samples have been tested by three different methods, using a reference gas oil, and oils of local origin.

In order to avoid the influence of thermal cycling under different circumstances, test samples were prepared centrally without further thermal cycling by the participating laboratories.

The participating laboratories were requested to perform at least two independent replicates of each sample, according to each method.

Test results, observations and remarks have been reported on the appropriate sheets.

3 Results and statistical interpretation

3.1 General

Statistical calculations have been run on all the results, according to ISO 5725-2 and ISO 3534-1.

Repeatability and reproducibility have been evaluated for each sample coupled with each method. Evaluations of the mean result have been carried out through variance analysis.

3.2 Mean value

Mean values for a given method have been evaluated, incorporating the influence of different gas oils and the intra-laboratory variance.

See table 2.

Table 2 — Mean values

Method according to table 1	Sample number		
	1	2	3
Method 1	2,20	6,22	1,42
Method 2	2,22	6,10	1,51
Method 3	1,89	6,28	1,42

3.3 Repeatability

Table 3 gives the general evaluation of standard deviation with respect to repeatability for each couple method/sample after checking the equality of variances using Bartlett's test.

Table 3 — Repeatability

Method according to table 1	Sample number		
	1	2	3
Method 1	0,17	0,20	0,07
Method 2	0,12	0,15	0,06
Method 3	0,10	0,17	0,05

3.4 Reproducibility

Table 4 gives the general evaluation of standard deviation with respect to reproducibility for each couple method/sample after checking the equality of variances using Bartlett's test.

Table 4 — Reproducibility

Method according to table 1	Sample number		
	1	2	3
Method 1	0,64	0,73	0,14
Method 2	0,75	0,64	0,28
Method 3	0,47	0,66	0,11

3.5 Interpretation of the results

When the typology of mean results is examined, the statistical proof of the existence of two groups corresponding to the gas oil type can only be obtained in the case of the couple sample 1/method 1.

The comparison of the mean values obtained by the three methods in all cases, studied by variance analysis of intra-laboratory means for each test shows that the methods are equivalent.

4 Conclusions

Based upon the above mentioned statistical tests, no significant difference between the methods can be found; hence the accuracy of the different methods is equivalent.

From a practical point of view, the tested non-standardized alternative methods are more user-friendly and more convenient than the official EC method.

It could not be shown that there is a statistical difference between results obtained with the different gas oils conforming to the requirements of Directive 87/94/EEC, Annex II [2].

Annex A
(informative)

Characteristics of and limits for straight ammonium nitrate fertilizers of high nitrogen content as given in EC Directive 80/876/EEC

A.1 Porosity (oil retention)

The oil retention of the fertilizer, which must first have undergone two thermal cycles of a temperature ranging from 25°C to 50°C, must not exceed 4 % by mass.

A.2 Combustible ingredients

The mass fraction of combustible material in per cent measured as carbon must not exceed 0,2 % for fertilizers having a nitrogen content of at least 31,5 % (mass fraction) and must not exceed 0,4 % for fertilizers having a nitrogen content of at least 28 % but less than 31,5 % (mass fraction).

A.3 pH

A solution of 10 g of fertilizer in 100 ml of water must have a pH value of at least 4,5.

A.4 Particle size analysis

Not more than a mass fraction of 5 % of the fertilizer must pass through a 1-mm mesh sieve and not more than a mass fraction of 3 % must pass through a 0,5-mm mesh sieve.

A.5 Chlorine

The maximum chlorine content is set at 0,02 % (mass fraction).

A.6 Heavy metals

Heavy metals should not be added deliberately, and any traces which are incidental to the production process should not exceed the limit fixed by the Committee.

The mass fraction of copper shall not be higher than 10 mg/kg.

No limits are specified for other heavy metals.