SLOVENSKI STANDARD
SIST-TP CEN/TR 17040:2017

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Gnojila in sredstva za apnjenje - Vzorčenje statičnih kupov - Tehnično poročilo o postopkih eksperimentalnega vzorčenja v okviru mandata M/454

Fertilizers and liming materials - Sampling of static heaps - Technical report on experimental sampling trials performed under mandate M/454

Düngemittel und Kalkdünger - Probenahme aus statischen Haufwerken - Technischer Bericht über Probenahmeversuche im Rahmen des Mandats M/454

Engrais et amendements minéraux basiques - Échantillonnage des tas statiques - Compte-rendu technique des essais d'echantillonnage réalisés sous le mandat M/454

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European foreword

This document (CEN/TR 17040:2017) has been prepared by Technical Committee CEN/TC 260 “Fertilizers and liming materials”, the secretariat of which is held by DIN.

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This document has been prepared under a mandate given to CEN by the European Commission and the European Free Trade Association.

Introduction

With mandate M/454 of October 2009 the EC asked the European Committee for Standardization (CEN) for a second extension to the standardization mandate M/335 concerning the modernization of the methods of analysis of fertilizers.

This extension concerns the framework of Regulation (EC) No 2003/2003 relating to fertilizers and liming materials [1].

The establishment of European Standards for methods of sampling and analysis is of utmost importance to guarantee a uniform application and control of the European legislation in all member states. Standardized methods of sampling and analysis are an indispensable element in guaranteeing a high level of quality and safety of EC fertilizers for the benefit of purchasers.

In order to avoid any improper use of the term EC-fertilizer Member States are required to check the compliance of such fertilizers or liming materials with the Regulation. To do this effectively, representative sampling is a prerequisite for reliable analytical results.

Within the framework of mandate M/335, CEN/TC 260 developed EN 1482-1 which applies only to the sampling of bulk material while it is being moved i.e. when any part of the fertilizer has an equal chance of being part of the incremental sample, and EN 1482-2 which specifies the sample preparation. In March 2009, a meeting of the Fertilizers Working Group of the EC took place to better define the current sampling practices in the different Member States. Two Member States recommended further improvements of EN 1482-1 as regards the sampling of static heaps.

Further enforcement authorities have limited resources for conformity assessment, and these are most efficiently deployed at the downstream end of the supply chain, i.e. at retailer or farmers premises. Therefore, nutrient content compliance should be ideally controlled at the point of sale to the end user, i.e. at the end of the supply chain. The fertilizer or liming material may be delivered or stored at this point in a bulk heap. Therefore EN 1482-1 might not fully satisfy the needs of Member States and an evaluation should be carried out by CEN to see whether a representative sample can be obtained from bulk heaps and if so what size of fertilizer heaps could be sampled at affordable costs.

Therefore mandate M/454 from the EC asked the European Committee for Standardization (CEN) to provide standardized methods for sampling static heaps.

In resolution BT C093/2009, the CEN Technical Board (BT) accepted mandate M/454 and allocated the work to CEN/TC 260, more specifically to its working group WG 1 “Sampling”.

1 Scope

This document covers reports on three experimental sampling studies which have been performed under mandate M/454 in order to check the accuracy of the developed sampling method for sampling of static heaps by comparing it to the sampling of the same fertilizer product in motion according to EN 1482-1 and to determine which sizes of static fertilizer heap, if any, can be sampled using existing sampling equipment.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

EN 1482-1:2007, *Fertilizers and liming materials — Sampling and sample preparation — Part 1: Sampling*

EN 1482-3, *Fertilizers and liming materials — Sampling and sample preparation — Part 3: Sampling of static heaps*

3 Background

3.1 General

Both producers and traders of fertilizers and liming materials have to guarantee a high level of quality in the nutrient amount and physical parameters of fertilizers they put on the market. EU Member State's official fertilizer controls are required to check the nutrient contents and the composition of fertilizers placed on the market. The purpose of Regulation (EC) No 2003/2003 [1] is to guarantee the farmer the quality of the fertilizer.

The first step of the fertilizer's control is the sampling in order to deliver a representative sample of a fertilizer placed on the market. Any bias during the sampling could lead to great economical and/or environmental consequences.

Sampling according to EN 1482-1 requires that a static heap has to be put in motion and this requires time and effort to be spent by the sampling officials. Official control authorities cannot always be present when static heaps are being formed or loaded for transport.

Consequently, the EC asked CEN/TC 260 "Fertilizers and liming materials" with Mandate M/454 for investigation of the possibility of the development of a European Standard and, if appropriate, to develop such a standard giving a sampling method of static fertilizer heaps for official controls that guarantees reliable analytical results.

3.2 Requested tasks

The following main tasks were requested:

a) Monitoring the literature as well as International and European Standards in similar fields and an evaluation of their relevance to this project (see Annex B);

b) elaboration and technical description of a method protocol to sample static heaps;

c) organization, performance and evaluation of experimental sampling studies in order to check the accuracy of the elaborated sampling method as compared with the sampling in motion of the same fertilizer according to EN 1482-1;

d) determine which size of static fertilizer heap could be sampled using existing sampling instruments, and which fertilizer types could be covered by the scope of the new sampling method.

4 Experimental sampling studies

4.1 General

The objective of the experimental sampling studies was to check if it is possible to take samples from static heaps of fertilizers which have an equivalent representativeness to samples which have been taken from the product in motion in accordance with EN 1482-1.

Basically it was proposed to undertake three comparative experimental trials using three mineral fertilizer types with chemical and granulometric characteristics more and more heterogeneous as follows:

1) Granulated “straight” fertilizer → 2) granulated “complex” NPK fertilizer → 3) “blend” NPK fertilizer.

The experimental trials were undertaken in collaboration with industrial partners in their facilities in order to be closer to reality.

After the presentation of the results of the 1st and the 2nd trial CEN/TC 260/WG 1 decided to perform the 3rd and last trial on a liming material product.

4.2 Sampling protocol

4.2.1 Protocol

The same protocol was followed during each of the three trials. Firstly a static conical heap was built up in **4 steps** using the transport chain of the storage plant as follows:

Receiving pit → Elevator → Several conveyor belts → Discharging in the storage cell onto the heap.

Secondly the conical heap was transferred to an adjacent storage cell with a loader on wheels so as to form a static rectangular heap. The constitution of this static rectangular heap was performed in the cases of the 1st and the 2nd experimental trials. This transfer was not performed in the case of the 3rd trial because of:

— heap’s transfer from storage cell to another isn’t usual for liming materials,

— the plant doesn’t lend itself to this transfer.

4.2.2 Mass of the heap to be sampled

The final mass of the static conical heap was 430 t for the 1st experimental trial and was reduced to 250 t for the 2nd and 3rd trial according to the advice of the CEN/TC 260/WG 1 after consideration of the results of the first trial.

4.2.3 Types of sampling

4.2.3.1 General

During the building up of the heap, three types of sampling were performed:

1) sampling in the flow, and

2) sampling from the static conical heaps,

3) sampling from the rectangular heap (1st an 2nd experimental trials).

4.2.3.2 Sampling in the flow (product in motion)

Independent sampling in the flow was performed according to EN 1482-1 as follows:

— use of a stream sampling cup as described in EN 1482-1:2007, 5.4.2;

— sampling of the increments in the fall of the product;

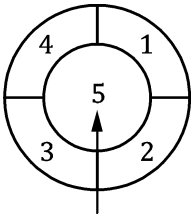
— random sampling during the whole period of the product downloading;

— the number of sampling points was always higher than the number specified in EN 1482-1, in Regulation (EC) No 2003/2003 [1] and according to CEN/TC 260/WG 1;

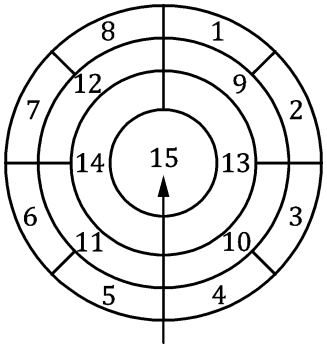
— the total mass of the aggregate samples was always higher than 4 kg.

4.2.3.3 Sampling of the static conical heaps

Sampling from each static conical heap was performed following the specific protocol developed for the project. For each intermediate cone, the number of sample units was defined beforehand. These sample units were then distributed on the surface of each intermediate cone (see Figure 1), the arrow representing the conveyor belt and the direction of the fertilizer's flow. Taking into account the actual size of the cone, the geometrical dimensions of the sample units were calculated so that they represent an equal quantity of fertilizer. The calculation takes into account the previous cone.



Step 1



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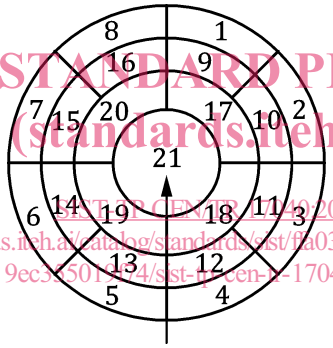

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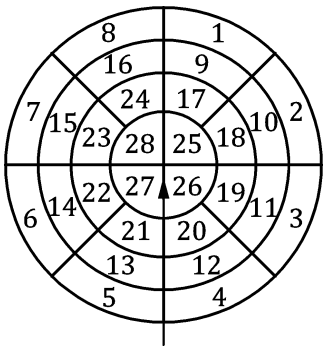

Step 4

Figure 1 — Distribution of the sample units on the intermediate cones (top view)

The sampling of the conical heap was conducted according to the sampling protocol:

— use of a spear with double tube (granulated fertilizer) or a tube shovel (wet liming material), see 4.3;

— sampling at the sampling points in each sample unit as pre-defined, the sample units represent equal quantities of fertilizer;

— random sampling in the sample units;

— taking 8 to 10 sampling points per sample unit so as to obtain a total mass of max. 4 kg per sample unit.

4.2.3.4 Sampling of the static rectangular heaps

The rectangular heap was sampled according to Regulation (EC) No 2003/2003 [1]. The surface of the heap was subdivided into sample units by drawing an imaginary grid such that each grid area represented an equal quantity of fertilizer. The number of grid areas exceeded the number of incremental samples required and each represented a sample unit.

In each sample unit, one increment was sampled in a random way using a spear with double tube. This procedure was performed within the 1st and the 2nd experimental trial (granulated fertilizer).

4.3 Sampling instruments

4.3.1 General

The equipment depends on the product (particle size and flow ability) and on the mass of samples required. The equipment has to be neutral (no influence on the sample like impurities or crushing) and should be easily cleaned to avoid contaminations between samples. For manual sampling the use of a spear makes it possible to work at some different depths inside the bulk.

Different types of manual and automatic spears and other types of equipment have been tested for their suitability to sample static heaps of fertilizers and liming materials. It was found that the spears with a double tube are the most used and adapted for sampling in bulk because they are more solid and easier to use. Spears should be used both in a vertically and horizontally way in order to take samples in different shapes of bulk.

4.3.2 Suitable instruments for granulated fertilizers

For granulated fertilizers the spear with double tubes and multiple openings was identified as the most appropriate sampling instrument: robust, simple to use, easily transportable (for one person) and not very expensive. During sampling of the static heap, it was possible to reach a depth of at most 1 m using this spear. In practice, it is difficult to go deeper than 0,9 m depending on the operator's force.

4.3.3 Suitable instruments for liming materials

For wet liming materials a tube shovel, which is a kind of shovel with a handle, the plate of the shovel being a tube, was identified to be the most appropriate sampling instrument, because the nature of the material means that only a very small amount would enter the openings in the double tube instrument. In the tube shovel the tube is about 30 cm long and has a large diameter of 12 cm to 13 cm. There is no closing system at the entrance. The sampling is done into the surface of the heap. This sampling instrument is a good compromise. It easily penetrates the material (± 30 cm deep), and avoids any bias in the sampling caused by the falling down of particles in the sample (closed system). As the liming material is quite compact the sample stays in the tube.