

**SLOVENSKI STANDARD**  
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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'échantillonnage réalisés sous le mandat M/454

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

Engrais et amendements minéraux basiques -  
Échantillonnage des tas statiques - Compte-rendu  
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mandat M/454

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Haufwerken - Technischer Bericht über  
Probenahmeversuche im Rahmen des Mandats M/454

This draft Technical Report is submitted to CEN members for Vote. It has been drawn up by the Technical Committee CEN/TC 260.

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EUROPEAN COMMITTEE FOR STANDARDIZATION  
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**CEN-CENELEC Management Centre: Avenue Marnix 17, B-1000 Brussels**

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## European foreword

This document (FprCEN/TR 17040:2016) has been prepared by Technical Committee CEN/TC 260 “Fertilizers and liming materials”, the secretariat of which is held by DIN.

This document is currently submitted to the Vote on TR.

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## 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;

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- 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 1<sup>st</sup> and the 2<sup>nd</sup> trial CEN/TC 260/WG 1 decided to perform the 3<sup>rd</sup> 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 1<sup>st</sup> and the 2<sup>nd</sup> experimental trials. This transfer was not performed in the case of the 3<sup>rd</sup> 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 1<sup>st</sup> experimental trial and was reduced to 250 t for the 2<sup>nd</sup> and 3<sup>rd</sup> 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 (1<sup>st</sup> and 2<sup>nd</sup> 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.

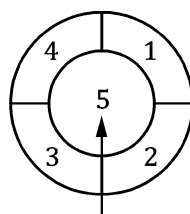
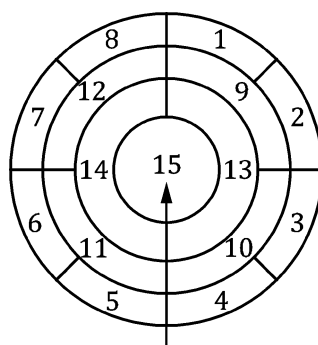
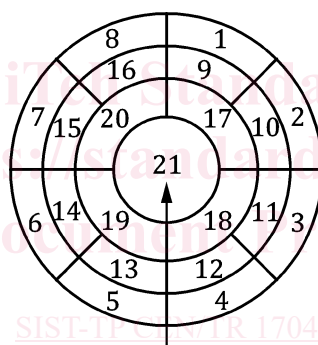
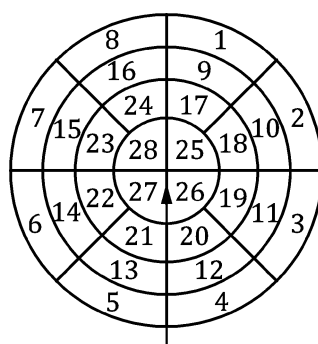
#### 4.2.3.3 Sampling of the static conical heaps

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 blue 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.

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**Step 1****Step 2****Step 3****Step 4****Figure 1 — Distribution of the sample units on the intermediate cones (top view)**