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

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National foreword

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

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

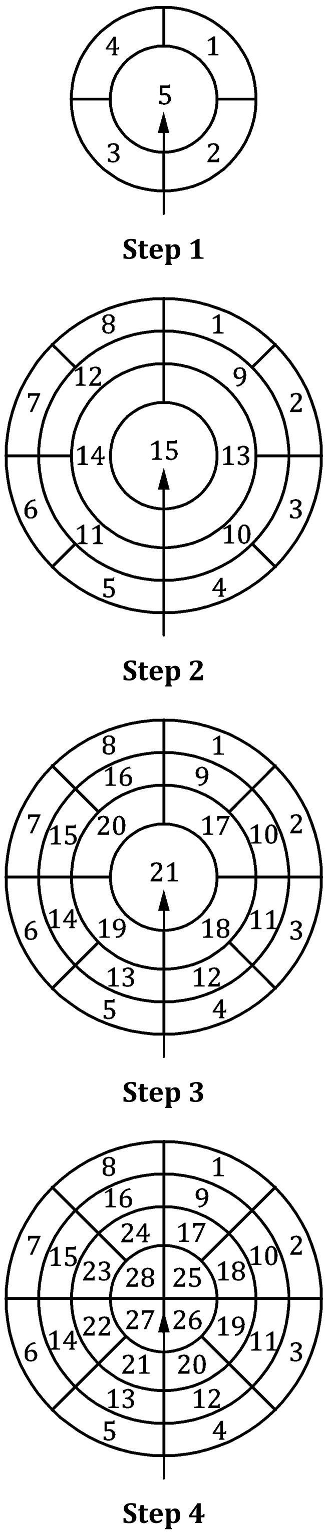


Figure 1 — Distribution of the sample units on the intermediate cones (top view)

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.

4.4 The 1st experimental trial (see A.1)

The experimental trial No I was performed with a granulated "straight" fertilizer: YaraBela® SULFAN® 24+18. This trial took place in the storage plant of YARA in Aunay-Sous-Crécy (France) from 4th to 7th June 2012. For the detailed description, results and conclusions see A.1.

4.5 The 2nd experimental trial (see A.2)

The experimental trial No II was performed with a granulated "complex" NPK fertilizer: SECOFlex 13-6-23 + SO_3 (12). This trial took place in the storage plant of Leseur SA in Carhaix (France) from 24th to 26th June 2013. For the detailed description, results and conclusions see A.2.

4.6 The 3rd experimental trial (see A.3)

The experimental trial No III was performed with a liming material: wet chalk (particle size of 0 mm to 12 mm), 80 % passing at 2 mm sieve. The trial took place in the production and storage plant of GROUPE MEAC SAS in Villeau (France) from 05th to 07th February 2014. For the detailed description, results and conclusions see A.3.

5 Development of sampling protocols

5.1 Simulation of sampling protocols

The complex procedure of sampling which was developed within the framework of the project aims at:

— generating a reference for the comparative analyses for each parameter (chemical content/characteristics and particle size distribution) with the average of the results of the 4 representative samples taken from the fertilizer/liming material flow according to EN 1482-1;

— generating a cartography in three dimensions of the conical heap with the chemical and granulometric characteristics of the fertilizer/liming material;

— being able to simulate different protocols of representative sampling in the conical heap and to compare their results with the reference;

— being able to study the influence of the size of the conical heap on the representativeness of the studied protocols of sampling.

The objective being the determination of the best protocol for sampling the static heap, five simulations of sampling the conical heap were developed as follows:

— The complete sampling: this simulation has to be considered as an ideal sampling plan in the conical heap. Indeed, the "sampler" takes as many samples as the number of units of sampling defined in the conical heap.

— The partial sampling around the base of the cone: this simulation can be considered as a classic sampling in the conical heap. The "sampler" takes samples around the base of the cone.

— The partial sampling by following an edge of the cone: this simulation takes samples from the conical heap by rising from the base up to the top. The "sampler" takes 2 to 4 samples according to the size of the cone (2 or 4 layers, 2 or 4 sample units).

— The reduced random sampling (5 samples): this simulation takes samples from the conical heap in a random way. The "sampler" takes 5 representative samples distributed in a random way over the whole heap.

— The widened random sampling (10 samples): This simulation takes samples from the conical heap in a random way. The “sampler” takes 10 representative samples distributed in a random way over the whole heap.

The five simulations have been applied to the intermediate conical heaps corresponding to the four constitutive steps of the formation of the conical heap so as to highlight any possible influence of the size of the static conical heap.

5.2 Statistical analysis

The objective was to determine the best protocol of sampling the conical heap. This protocol has to supply a result equivalent to the reference sampling taken from the flow during the formation of the cone according to EN 1482-1.

On the basis of samples taken from the conical heaps, the various protocols of sampling (complete, partial, random etc.) of the cones were simulated and their results were compared with the reference constituted by the granulometric (D16, D84, GSI…) and chemical (main nutrients, Neutralizing Value for the liming material) analyses of samples taken from the flow (according to EN 1482-1) during the four constitutive steps.

The statistical analysis bases itself on a multiple comparison of averages, which allows determination of any significant differences between the series of observations. It is, however, necessary to verify the relevance of these differences from a practical and/or regulatory point of view.

5.3 Discussion and conclusions

5.3.1 General

The project’s partners asked CEN/TC 260/WG 1 to take into account the following considerations for the elaboration of a standard method for sampling of static heaps.

5.3.2 Basic principle of sampling

The basic principle for sampling fertilizer is that each particle has the same chance of becoming part of the sample. In practice, it is not possible to apply this basic principle in the sampling of a static conical heap. We can consider only the upper layer of the heap as being the sampling zone (see Figure 2).

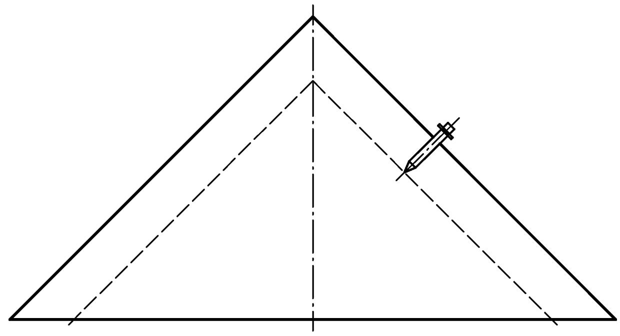


Figure 2 — Sampling of a static heap using a spear

The depth from which a sample can be obtained is dependent on the length of the sampling device and the nature of the product being sampled. During the trials, a spear (double tubes and multiple openings) for granulated fertilizers and a tube shovel for the liming material have been used. It was demonstrated that it is physically impossible to drive the spear/tube shovel into the heap more than 0,9 m but to at most 1 m deep in the granulated fertilizer and 0,30 m in the liming material.

Consequently, the sampling zone of the static conical heap is the upper layer of the heap having a thickness of at most 1 m for granulated fertilizers and 0,30 m for liming materials.

It has, therefore, to be recognized that the principle for sampling a static heap is that each particle of the upper layer has the same chance of becoming part of the sample. In this way it can be considered that the upper layer is representative of the whole static heap.

5.3.3 Segregation

During trials, segregation has been observed during the building up of the heap, even where the fertilizer is well homogeneous (e.g. trial I). Segregation is a natural process which occurs when the fertilizer is transported or piled onto a heap. Large granules rise to the top of the load during the transport and roll to the bottom during heaping (see Figure 3).

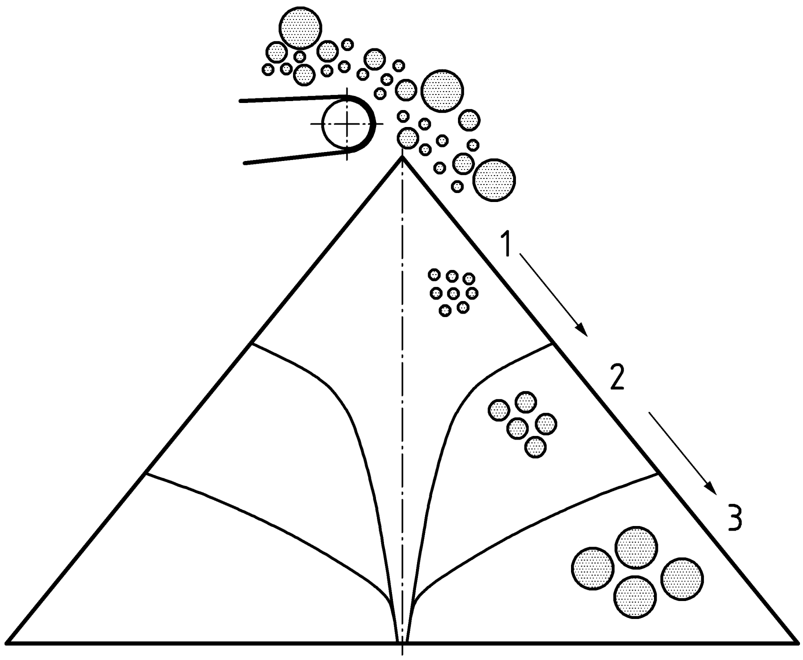


Figure 3 — Segregation during the building up of the static heap

Segregation is caused by the granulometric characteristics of the product and affects the granulometric distribution of the particles in the heap. The more the granulometric distribution of the fertilizer is extended, the more the particles will be segregated in the heap. Consequently, if the nutrient content is a function of the granulometry (e.g. in the case of blended fertilizers), the nutrient distribution inside the conical heap is also affected by the segregation.

The aim of the project was to determine a sampling protocol which mitigates the effects of the segregation and reconstitutes the initial mixing of particles.

As mentioned above, the segregation phenomenon has been observed during all trials, but was more pronounced during the last trial with the liming material. This is the result of the use of a less homogeneous product. However the granulometric parameters of the samples taken from the heaps have been affected by segregation much more strongly than the chemical parameters of the samples, provided that the products were granulated (straight or complex) or chemically very homogenous (liming material) independent of the granulometry.

Because of the segregation the effect of heap's size has also been observed. The sample units are defined on the surface area of the heap. The greater the heap the larger the surface area and smaller the area of each sampling unit which represents the same mass. Consequently there are more sampling units the greater the heap. Therefore the influence of one sample unit has a lower impact on the result of the sampling when the heap is greater.

5.4 Scope of the experimental trials

The initial scope of the experimental trials covered more and more heterogeneous (chemically and granulometrically) products as: granulated straight fertilizers (1st trial), granulated complex fertilizers (2nd trial) and blend complex fertilizers (3rd trial).

However, a wide discussion was conducted between the CEN/TC 260/WG 1 experts during the 12th meeting (January 2013) and the 13th meeting (December 2013) about the opportunity to conduct the 3rd trial with a blended fertilizer since these products are not stored typically in large static heaps. Normally blended fertilizers are delivered directly to the user in small quantities or stored in big-bags for a short time. Therefore, the question was raised whether another heterogeneous product, a static heap of which is more likely to occur in practice should be selected for the 3rd trial.

Therefore, it was proposed to select a liming material as these products are very often sampled in practice from static heaps and may be heterogeneous. Moreover liming materials have been covered by the EU-Fertilizers Legislation since 2013 and it will be essential that these products are covered by the scope of EN 1482-3 as they are covered also by EN 1482-1 and EN 1482-2.

It was, therefore agreed by the CEN/TC 260/WG 1 experts that the scope of the experimental trials and their conclusions should cover a single nutrient granulated fertilizer, a uniform complex granulated fertilizer and a milled or granulated liming material.

6 Conclusions

The partial sampling around the base of the heap does not supply a representative sample because of the phenomenon of size segregation. During the formation of the fertilizer heap, bigger diameter particles run down to the base of the heap while the fine particles concentrate on the top of the heap. This phenomenon generated significant differences between the described sampling protocol and the reference.

Under the conditions of the experimental trials and for the fertilizer types tested (granulated “straight” or “complex” fertilizers and liming materials), it is possible to realize:

— a partial sampling constituted by at least 40 to 50 increments (= sampling points) taken along an edge by rising from the base to the top of the heap, or,

— a partial sampling constituted by at least 40 to 50 increments (= sampling points) taken randomly from the whole surface of the heap.

The results of chemical analyses of the samples obtained following these protocols will not be statistically different from the inflow reference samples (EN 1482-1). The results of the granulometric analyses of the samples obtained following these protocols were not statistically different from the inflow reference samples (EN 1482-1) for the two kinds of granulated fertilizers (granulated “straight” or “complex” fertilizer) but not for the liming material due to the high segregation propensity of this product.

Within the 1st and 2nd experimental trial, the conical heaps were transferred to another storage cell with a loader on wheels so as to form a static rectangular heap. This rectangular was sampled according to Regulation (EC) No 2003/2003 [1].

The samples taken in the flow according to EN 1482-1 from the conical heap and from the rectangular heap have been re-mixed so as to provide three representative samples of the fertilizer lot provided by three different ways of sampling. These samples were analyzed (chemical and granulometric analysis) and the results were reported. Although there are some differences, on a practical point of view and regarding the measurement uncertainty, it can be considered that the results are similar. However, a statistical study has not been conducted since it was not within the purpose of the trial.

Annex A
(informative)

Reports about the experimental trials

A.1 The 1st experimental trial

A.1.1 General

The first comparative test was realized in association with the industrial partner YARA in the storage site of Aunay-Sous-Crécy (France). The product used during this first trial was a granulated nitrogenous sulfured “straight fertilizer” (YaraBela® SULFAN® 24+18) chosen for its chemical and granulometric homogeneity.

A.1.2 Description of the site

The plant is classified Seveso, comprised of two main storage buildings with a total storage capacity of 30 000 t. This site is equipped with a weighing machine for the trucks. The hall 1 (called “HR1”, with a capacity of 15 000 t) was chosen for the trial due to the fact that it is equipped with a conveyor and is subdivided into cells.

A.1.3 Constitution of the conical and rectangular heaps

A.1.3.1 General

The constitution of the conical and rectangular heaps was a complex operation. The total mass of fertilizer was 430 t and was transported by 17 trucks with an average capacity of 25,4 t. The conical heap was transferred to an adjacent cell with a wheel loader to form the rectangular heap. The execution of these two steps was possible due to the collaboration and support of YARA and the employees of this storage plant.

A.1.3.2 Conical heap

The constitution of the conical heap was conducted in four steps and needed two days. For each step, the dimensions of the conical heap were measured (see Table A.1). At the end, the conical heap had a height of 4,37 m, an average diameter of 18 m and a total mass of 431,44 t. The height of the cone has been measured with a laser pointed on the top of the conical heap. The diameter of the heap was measured with a target drawn beforehand on the floor.

Table A.1 — Characteristics of the conical heap for each step of its constitution

Steps	1	2	3	4
Downloading time (hh:mm:ss)	00:05:26	00:29:29	01:28:55	03:02:23
Downloading speed (t/h)	99,8	86,9	85,2	83,4
Added quantity (t)	9,04	42,70	126,30	253,40
Height of the cone (m)	1,20	2,19	3,17	4,37
Radius of the cone(m)	2,50	4,48	6,70	9,00
Added volume (m^3)	7,85	38,14	103,02	219,11

The objective of the step by step constitution of the conical heap is to establish a comparison between the sampling procedure (in the flow or in the heap) for increasing lot sizes.

A.1.3.3 Rectangular heap

The conical heap was transferred into an adjacent cell using a loader on wheels and formed into a rectangular heap which had the following approximated size: height of 2,25 m, width of 13,5 m, length of 23,5 m and the same total mass of 430 t.

This transfer aims at comparing the classical sampling procedure for rectangular heaps with the sampling in the flow and in the conical heap.

A.1.4 Sampling in the flow

For each of the four steps of the constitution of the conical heap, an independent sample was taken from the flow according to EN 1482-1:

— use of a stream sampling cup according to EN 1482-1;

— incremental sampling in the fall of the fertilizer;

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

— the number of sampled increments for each step (see Table A.2) was always greater than the number specified in EN 1482-1, in Regulation (EC) No 2003/2003 and in the composite protocol;

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

Table A.2 — Determination of the number of increments sampled for each step

Step	**Mass (t)**	**Number of increments according to the standards for each step**			
		EN 1482–1	**Regulation (EC) No 2003/2003**	**Composit protocol**	**Trial 1**
1	9,04	10	14	-	**15**
2	42,70	14	30	-	**35**
3	126,30	23	40	-	**45**
4	253,40	33	40	40	**45**

For each step, the increments were mixed and then divided using riffle and rotating dividers. At the end of the division procedure, for each step, eight final samples (± 500 g/sample) remained. They were distributed as follows:

a) 1 sample for the chemical analysis (Laboratoire Départemental d'Analyses et de Recherche) (LDAR);

b) 1 sample for the granulometric analysis (Centre wallon de Recherches agronomiques) (CRA-W);

c) 2 samples mixed again with the final samples of the other steps of the constitution of the conical heap (see below);

d) 4 samples kept in stock (CRA-W).

Two final samples of each step have been mixed again to obtain a sample of the whole heap of fertilizer. This remixed sample has been also divided following the same procedure and the eight final samples of the whole heap have been distributed as follows:

e) 1 sample for the chemical analysis (LDAR),

f) 1 sample for the granulometric analysis (CRA-W), and

g) 6 samples kept in stock (CRA-W).

A.1.5 Sampling of the conical heap

For each step of the build-up of the conical heap, a specific sampling plan was undertaken. For each intermediate cone, the number of sample units was defined beforehand (see Table A.3).

These sampling units have been then distributed on each intermediate cone (see Figure 1). Taking into account the actual size of the cone, the geometrical dimensions of the sample units have been calculated so that they have the same approximate mass (see Table A.3). The calculation takes into account the previous cone.

Table A.3 — Number and size of the sample units for the 4 steps of the conical heap constitution

Step	**Mass** (t)	**Number of sample units**	**Mass of the sample unit** (t)
1	9,04	5	1,81
2	42,70	15	2,85
3	126,30	21	6,01
4	253,40	28	9,05

The dimensions and the distribution of the sample units have been visualized on the cone with the laser. So as to avoid walking on the heap and disturbing its structure, a bucket lift has been used to locate the sample units and to sample. For each intermediate cone corresponding to the four steps of the conical heap constitution, the distribution of the sample units has been defined beforehand as specified in Figure 1. The arrow represents the conveyor belt and the direction of the fertilizer flow.

Sampling of the conical heap was undertaken following the proposed protocol and taking into account the composite protocol:

— use of a probe with double tube and multiple openings;

— sampling of the increments in each sample unit as pre-defined. The sample units have the same approximate mass;

— random sampling in the sample units;

— taking of 10 to 12 increments per sampling unit so as to obtain a total mass of approximately 4 kg.

Samples were taken from the bottom up to the top of the heap.

For each step, the increments were mixed and then divided using riffle and rotating dividers. At the end of the division procedure, for each step, there were eight final samples (± 500 g/sample). They have been distributed as follows:

a) 1 sample for the chemical analysis (LDAR),

b) 1 sample for the granulometric analysis (CRA-W),

c) 1 sample mixed again with the final samples of the others sample units of the heap, and

d) 5 samples kept in stock (CRA-W).

One final sample of each sample unit of the intermediate cone was mixed again to obtain a sample of the whole intermediate heap. This remixed sample was also divided following the same procedure and the eight final samples of the whole intermediate heap were distributed as follows:

e) 1 sample for the chemical analysis (LDAR),

f) 1 sample for the granulometric analysis (CRA-W), and

g) 6 samples kept in stock (CRA-W).

A.1.6 Sampling of the rectangular heap

After the transfer of the conical heap to an adjacent cell, the rectangular heap was sampled following Regulation (EC) No 2003/3003 [1]. The top of the heap was subdivided into sampling units (40) using the frames of the roof and the concrete slabs as marks. A grid was drawn defining the sample units. In the 40 sample units, one increment was taken in a random way using the probe double tubes and multiple openings. The increments were mixed and then divided using riffle and rotating dividers. At the end of the division procedure, for each step eight final samples (± 500 g/sample) were obtained. They were distributed as follows:

a) 1 sample for the chemical analysis (LDAR),

b) 1 sample for the granulometric analysis (CRA-W), and

c) 6 samples kept in stock (CRA-W).

A.1.7 Chemical analysis

The chemical analysis was undertaken by the LDAR. After a milling to a particle size of 500 µm according to EN 1482-2, the following parameters were determined:

— total nitrogen, determination by element analyzer,

— total phosphorus: determination by UV-VIS spectrophotometry after an extraction using mineral acids,

— water soluble potassium: determination by Flame Emission spectrophotometry after an extraction using boiling water, and

— total sulfur, determination by spectrometer ICP (Inducted Coupled Plasma) after an extraction using diluted hydrochloric acid.

A.1.8 Granulometric analysis

The granulometric analysis was performed by the CRA-W using a protocol according to EN 1235:1995 and EN 1235/A1:2003. The final sample (± 500 g) taken during the sampling on site was divided so as to obtain a quantity between 250 g and 300 g. The choice of the seven sieves was on the average diameter of the fertilizer specified by the producer (3,4 mm ± 0,25 mm), the guide for the optimization of solid mineral fertilizer spreading (Comifer 2009), the "Handbook of solid fertilizer blending – code of good practice for quality (EFBA 2007) and an agreement between the experts of the consortium with the following mesh sizes: 5 mm, 4 mm, 3,55 mm, 3,15 mm, 2,80 mm, 2,50 mm and 1 mm.

A.1.9 Conclusions of the 1st trial

The objective of the project was to determine if it is possible to undertake sampling of a static heap of fertilizer having a representativeness equivalent to sampling taken from the flow according to EN 1482-1. The product used during this first trial was a granulated nitrogenous sulfured "straight fertilizer" (YaraBela®SULFAN®24+18).

A conical heap of fertilizer (431,44 t) was built up in 4 steps. One of the objectives of the trial was to consider whether the mass (size) of the heap had any effect on the results of the comparison of the sampling method with the in-flow method:

— Sampling from the flow was undertaken according to EN 1482-1 and constitutes the reference sample for the comparative analysis. Four representative samples were constituted and analysed for the flow. They correspond to four stages of the build-up of the conical heap.

— Samples in the intermediate conical heaps were taken using of probe (double tubes and multiple openings). Sampling units of the same approximate mass (size) were defined for every intermediate cone. The number of units corresponded with the size of the cone. A final sample was established for each sampling unit. All in all, 69 final samples were analysed for the four intermediate cones.

The chemical analysis undertaken by the LDAR supplied the contents of total N and total SO_3 of the final samples. The sieve analysis undertaken by the CRA-W supplied the granulometric parameters of diameters D16, D50, D84 and the GSI (Granulometric Spread Index) indicating the particle size distribution in the product from the samples, according to EN 1235/A1.

The complex procedure of sampling which was developed within the framework of the project aims at:

1) generating a reference for the comparative analyses which corresponds to each parameter (chemical content and granulometry) with the average of the results of the four representative samples taken from the fertilizer flow at the level of the conveyor belt;

2) generating a cartography in three dimensions of the conical heap of the chemical and granulometric characteristics of the fertilizer;

3) being able to simulate different protocols of representative sampling in the conical heap and to compare their results with the reference;

4) being able to study the influence of the size of the conical heap on the representativeness of the studied protocols of sampling.

Five protocols of sampling on the conical heap were simulated and their results compared with the reference: complete sampling, partial sampling around the base of the cone, partial sampling by following a cone edge, reduced random sampling (5 samples), widened random sampling (10 samples). The statistical analysis bases itself on a multiple comparison of averages which allows determining the significant differences between the series of observations. **It is, however, necessary to verify the relevance of these differences from a practical and/or regulatory point of view**.

The conclusions of the comparative analysis are the following ones:

5) For the studied fertilizer, the content of total N determined on the basis of the samples taken from the conical heap is not statistically different from the content of the reference determined on the basis of samples taken from the flow, irrespective of the followed protocol or the size of the conical heap.

6) From a statistical point of view, the SO_3 contents of the samples taken from the conical heap are systematically different from the reference (flow samples), whatever the protocol is. This report shall, however, be put in perspective. On one hand, the difference in the measurement of the content of SO_3 is 10 %, that is ± 1,7 g/100 g. On the other hand, the tolerance permitted within Regulation (EC) No 2003/2003; Annex II) is 0,9 per unit of SO_3. In practice, the average content of SO_3 measured on the heap cannot thus be considered as significantly different from the content obtained from the samples taken from the flow according to the reference protocol.

7) The partial sampling around the base of the heap **does not produce** a representative sample for the determination of the granulometric parameters of the fertilizer (D16, D84 and GSI) because of the phenomenon of **size segregation**. During the formation of the fertilizer cone, bigger diameter particles run down to the base of the heap while the fine particles concentrate at the top of the cone. **Although the tested fertilizer is very homogeneous, this phenomenon generated significant differences between this sampling protocol and the reference**. This phenomenon is even more significant for larger heaps.

8) The fertilizer delivered for the formation of the second cone (51,74 t) had a particle size lower than the rest of the stored fertilizer. This had an impact on the comparative analysis of the granulometric parameters, mainly of D84. Whatever the sampling protocol in the studied conical heap, the average D84 of the fertilizer particles of this cone was significantly different from D84 from the samples taken from the flow. Among the studied granulometric characteristics, sometimes the D16 was also impacted, never the GSI.

9) Given the above, in the conditions of the trial, and the fertilizer type used (granulated straight fertilizer), it is possible to realize a partial sampling constituted from 40 to 50 increments **taken along an edge** by rising from the base to the top of the cone **or taken randomly from the whole surface of the heap** for which the results of chemical and granulometric analyses will not be statistically different from the reference sample taken from the flow (EN 1482-1). These two protocols (partial sampling by following a cone edge, reduced random sampling) will generate representative samples of conical heaps of large capacity (>150 t), while being realized by only one person.

A.2 The 2nd experimental trial

A.2.1 General

The 2nd comparative trial was realized in collaboration of the industrial partner Leseur SA on its storage plant in Carhaix (France). The product used for this second trial was a granulated complex NPK fertilizer. The trial took place on the 24th to 26th June 2013.

A.2.2 Description of the site

The plant is of middle size, comprised of several storage buildings with a total storage capacity of 12 000 t in bulk plus 6 000 t in packaging. This site is very like the previous one used for the first trial, it is equipped with a weighing machine for the trucks and its main hall is equipped with a conveyor and is subdivided into cells.

A.2.3 Constitution of the conical and rectangular heaps

A.2.3.1 General

The constitution of the conical and rectangular heaps was a complex operation. The total mass of fertilizer was ± 250 t and was already stored in the plant. The transfer of the fertilizer from one cell to another needed the use of a tractor, a dumper and a loader. After sampling the conical heap was transferred to an adjacent cell with a wheel loader to form the rectangular heap.

A.2.3.2 Conical heap

The constitution of the conical heap was conducted in four steps and needed two days. For each step, the dimensions of the conical heap were measured (see Table A.1). At the end, the conical heap had a height of 4,08 m, an average diameter of 14,72 m and a total mass of ± 250 t. The height of the cone has been measured with a laser pointed on the top of the conical heap. The diameter of the heap has been measured with a target drawn beforehand on the floor.

Table A.4 — Characteristics of the conical heap for each step of its constitution

Steps	1	2	3	4
Downloading time (hh:mm:ss)	00:03:01	00:18:18	00:55:30	01:34:17
Downloading speed (t/h)	60	80	80	100
Added quantity (t)	3,02	25,58	73,22	146,38
Height of the cone (m)	0,87	1,89	3,01	4,08
Radius of the cone(m)	1,64	3,53	5,46	7,36
Added volume (m^3)	2,43	22,16	69,46	137,15

The objective of the step by step constitution of the conical heap is to establish a comparison between the sampling procedure (in the flow or in the heap) for increasing lot sizes.

A.2.3.3 Rectangular heap

The conical heap was transferred into an adjacent cell and formed into a rectangular heap which had the following approximated size: height of 2,5 m, width of 5,85 m, length of 20 m and the same total mass of ± 250 t.

This transfer aims at comparing the classical sampling procedure for rectangular heaps with the sampling in the flow and in the conical heap.

A.2.4 Sampling in the flow

For each of the four steps of the constitution of the conical heap, an independent sample was taken from the flow according to EN 1482-1:

— use of a stream sampling cup according to EN 1482-1;

— incremental sampling in the fall of the fertilizer;

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

— the number of sampled increments for each step (see Table A.2) was always greater than the number specified in EN 1482-1, Regulation (EC) No 2003/2003 and the composite protocol;

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

Table A.5 — Determination of the number of increments sampled for each step

Step	**Mass (t)**	**Number of increments according to the standards for each step**			
		EN 1482–1	**Regulation (EC) Nr 2003/2003**	**Composit protocol**	**Trial 2**
1	3,02	10	8	-	**15**
2	25,58	11	23	-	**35**
3	73,22	18	39	-	**45**
4	146,38	25	40	40	**45**

For each step, the increments were mixed and then divided using riffle and rotating dividers. At the end of the division procedure, for each step, eight final samples (± 500 g/sample) remained. They were distributed as follows:

a) 1 sample for the chemical analysis (LDAR),

b) 1 sample for the granulometric analysis (CRA-W),

c) 2 samples mixed again with the final samples of the other steps of the constitution of the conical heap (see below), and

d) 4 samples kept in stock (CRA-W).

Two final samples of each step have been mixed again to obtain a sample of the whole heap of fertilizer. This remixed sample has also been divided following the same procedure and the eight final samples of the whole heap have been distributed as follows:

e) 1 sample for the chemical analysis (LDAR),

f) 1 sample for the granulometric analysis (CRA-W), and

g) 6 samples kept in stock (CRA-W).

A.2.5 Sampling of the conical heap

For each step of the build-up of the conical heap, a specific sampling plan was undertaken. For each intermediate cone, the number of sample units was defined beforehand (see Table A.3).

These sampling units have been then distributed on each intermediate cone (see Figure 1). Taking into account the actual size of the cone, the geometrical dimensions of the sample units have been calculated so that they have the same approximate mass (see Table A.3). The calculation takes into account the previous cone.

Table A.6 — Number and size of the sample units for the 4 steps of the conical heap constitution

Step	Mass (t)	Number of sample units	Mass of the sample unit (t)
1	3,02	5	0,60
2	25,58	15	1,71
3	73,22	21	3,49
4	146,38	28	5,23

The dimensions and the distribution of the sample units have been visualized on the cone with the laser. So as to avoid walking on the heap and disturbing its structure, a bucket lift has been used to locate the sample units and to sample. For each intermediate cone corresponding to the four steps of the conical heap constitution, the distribution of the sample units has been defined beforehand as specified in Figure 1. The arrow represents the conveyor belt and the direction of the fertilizer flow.

Sampling in the conical heap was undertaken following the proposed protocol and taking into account the composite protocol:

— use of a probe with double tube and multiple openings;

— sampling of the increments in each sample unit as pre-defined. The sample units have the same approximate mass;

— random sampling in the sample units;

— taking of 10 to 12 increments per sampling unit so as to obtain a total mass of approximately 4 kg.

For each step, the increments were mixed and then divided using riffle and rotating dividers. At the end of the division procedure, for each step, there were eight final samples (± 500 g/sample). They have been distributed as follows:

a) 1 sample for the chemical analysis (LDAR),

b) 1 sample for the granulometric analysis (CRA-W),

c) 1 sample mixed again with the final samples of the others sample units of the heap, and

d) 5 samples kept in stock (CRA-W).

One final sample of each sample unit of the intermediate cone was mixed again to obtain a sample of the whole intermediate heap. This remixed sample was also divided following the same procedure and the eight final samples of the whole intermediate heap were distributed as follows: 1 sample for the chemical analysis (LDAR), 1 sample for the granulometric analysis (CRAW), 1 sample mixed again with the other final samples of the intermediate cones and 5 samples kept in stock (CRA-W).

Finally the four final samples of the four intermediate cones were remixed and also subdivided so as to constitute the eight final samples of the whole heap. These samples have been distributed within the labs to be analysed.

A.2.6 Sampling of the rectangular heap

After the transfer of the conical heap to an adjacent cell, the rectangular heap was sampled following Regulation (EC) No 2003/3003 [1]. The top of the heap was subdivided into sampling units (40) using the frames of the roof and the concrete slabs as marks. A grid was drawn defining the sample units. In the 40 sample units, one increment was taken in a random way using the probe double tubes and multiple openings. The increments were mixed and then divided using riffle and rotating dividers. At the end of the division procedure, for each step eight final samples (± 500 g/sample) were obtained. They were distributed as follows:

a) 1 sample for the chemical analysis (LDAR),

b) 1 sample for the granulometric analysis (CRA-W), and

c) 6 samples kept in stock (CRA-W).

A.2.7 Chemical analysis

The chemical analysis was undertaken by the LDAR. After a milling to a particle size of 500 µm according to EN 1482-2, the following parameters were determined:

— total nitrogen, determination by element analyzer,

— total phosphorus: determination by UV-VIS spectrophotometry after an extraction using mineral acids,

— water soluble potassium: determination by Flame Emission spectrophotometry after an extraction using boiling water, and

— total sulfur, determination by spectrometer ICP (Inducted Coupled Plasma) after an extraction using diluted hydrochloric acid.

A.2.8 Granulometric analysis

The granulometric analysis was performed by the CRA-W using a protocol according to EN 1235:1995 and EN 1235/A1:2003. The final sample (± 500 g) taken during the sampling on site was divided so as to obtain a quantity between 250 g and 300 g. The choice of the seven sieves has been based on the average diameter of the fertilizer specified by the producer (D50: 3,28 mm ± 0,25 mm), the guide for the optimization of solid mineral fertilizer spreading (Comifer 2009), the "Handbook of solid fertilizer blending – code of good practice for quality (EFBA 2007) and an agreement between the experts of the consortium. They were the same as in the first trial with the following mesh sizes: 5 mm, 4 mm, 3,55 mm, 3,15 mm, 2,80 mm, 2,50 mm and 1 mm.

A.2.9 Conclusions of the 2nd trial

The objective of the project was to determine if it is possible to undertake sampling of a static heap of fertilizer having a representativeness equivalent to sampling taken from the flow according to EN 1482-1. The product used during this second trial was a granulated complex NPK fertilizer. A conical heap of fertilizer (248,20 t) was built up in 4 steps. At every step of the building up of the static heap, samples were taken from the flow of fertilizer at the end of the conveyor belt and from the static heap:

— Sampling from the flow was undertaken according to EN 1482-1 and constitutes the reference sample for the comparative analysis. Four representative samples were constituted and analysed for the flow. They correspond to four stages of the build-up of the conical heap.

— Samples in the intermediate conical heaps were taken using of probe (double tubes and multiple openings). Sampling units of the same approximate mass (size) were defined for every intermediate cone. The number of units corresponded with the size of the cone. A final sample was established for each sampling unit.

The chemical analysis undertaken by the LDAR supplied the contents of total N, P_2O_5, K_2O and SO_3 of the final samples. The sieve analysis undertaken by the CRA-W supplied the granulometric parameters of diameters D16, D50, D84 and the GSI (Granulometric Spread Index) indicating the particle size distribution in the product from the samples, according to EN 1235/A1.

The complex procedure of sampling which was developed within the framework of the project aims at:

1) generating a reference for the comparative analyses which corresponds to each parameter (chemical content and granulometry) with the average of the results of the four representative samples taken from the fertilizer flow at the level of the conveyor belt;

2) generating a cartography in three dimensions of the conical heap of the chemical and granulometric characteristics of the fertilizer;

3) being able to simulate different protocols of representative sampling in the conical heap and to compare their results with the reference;

4) being able to study the influence of the size of the conical heap on the representativeness of the studied protocols of sampling.

Five protocols of sampling on the conical heap were simulated and their results compared with the reference: complete sampling, partial sampling around the base of the cone, partial sampling by following a cone edge, reduced random sampling (5 samples), widened random sampling (10 samples). The statistical analysis bases itself on a multiple comparison of averages which allows determining the significant differences between the series of observations. **It is, however, necessary to verify the relevance of these differences from a practical and/or regulatory point of view**.

The conclusions of the comparative analysis are as follows:

5) During this second trial, the size segregation was found to be more significant and had a greater effect on the results than during the first trial. During the formation of the fertilizer cone, larger diameter particles run down to the base of the heap while the fine particles concentrate at the top of the cone. This observation is probably due to the use of a less homogeneous fertilizer. However, the size segregation affected the granulometric parameters more than the chemical content of the samples taken from the conical heaps.

6) Due to the size segregation, a heap's size effect was observed. The sample units were defined on the surface area of the heap. The larger is the heap, the larger the surface area and smaller the area of each sampling unit which represents the same mass. Consequently, there are more sampling units the larger the heap. Therefore, the influence of one sample unit has a lower impact on the result of the sampling when the heap is larger.

This observation affected mainly two types of sampling from the heap:

— the complete sampling,

— the partial sampling by following a cone edge.

These two sampling protocols are able to give comparative results to the reference sampling in the flow for heaps larger than or equal to 100 t for the complete sampling and larger than or equal to 250 t for the partial sampling by following a cone edge.

7) The partial sampling around the base of the heap **does not supply** a representative sample. This observation has been already highlighted during the first trial even though the tested fertilizer was very homogeneous.

8) The random sampling from the heap (reduced and widened) seems to be able to mitigate the size segregation. This observation is important because the underlying idea is to give the same weight to each sample unit in the final sample. During the sampling, the surface of the heap has been divided in sample units of equal mass. This implies that the bottom sampling units of the heap are more numerous than the top ones. If the sampling protocol doesn't take into account this fact, more samples will be taken at the bottom of the heap where the granules are bigger due to the size segregation. In this way the coarser granules will be overrepresented in the final sample and this would not be representative. Thus the sampling protocol should mitigate this issue.

9) Following the previous observation, the size segregation affected the granulometric parameters. For the chemical contents in the samples taken on the heap, although from a statistical point of view, they were sometimes different from the reference ones taken in the flow, they were not significantly different from a practical and legal point of view. The observed differences were significantly lower than the tolerances given in Annex II of Regulation (EC) No 2003/2003 [1].

10) Finally, the same conclusion as from the first trial can be taken: in the conditions of the trial and for the fertilizer type used (granulated NPK fertilizer), it is possible to realize a partial sampling constituted from 40 to 50 increments **taken along an edge** by rising from the base to the top of the cone **or taken randomly from the whole surface of the heap** for which results of chemical and granulometric analyses will not be statistically different from the reference (EN 1482-1). These two protocols (partial sampling by following a cone edge, reduced random sampling) will generate representative samples of conical heaps of big capacity (>100 t), while being undertaken by only one person.

A.3 The 3rd experimental trial

A.3.1 General

Following the 13th meeting of CEN/TC 260/WG 1, the 3rd experimental trial was performed with a liming material. After proposals and exchanges within CEN/TC 260/WG 1, it was agreed to organize the trial in the plant of the GROUPE MEAC SAS, located in Villeau (France). The plant has a covered building where the product was stored and which provided good experimental conditions. The trial took place from 5th to 7th February 2014.

The tested liming material was a wet chalk (grain size 0 mm to 12 mm), 80 % passing at 2 mm.

A.3.2 Description of the site

The trial has been undertaken in the production and storage plant of GROUPE MEAC SAS in Villeau (France, 28). This is a production and temporary storage place. The quarry where the liming material was extracted is located near to the plant. The liming material was transported from the quarry to the plant using a conveyor belt. Depending on demand, the production is directed either to the drying system and process, or to the covered storage place. For the purposes of the trial, this second way was used. The liming material was stored in a large covered building and transported using a central conveyor belt. During the 3rd trial, only a conical heap has been constituted and sampled. The displacement of the conical heap into an adjacent cell in order to build up a rectangular heap and its sampling was not possible, because of the following reasons:

— The transfer of material from one storage cell to another isn't usual for liming material.

— The plant doesn't lend itself to this transfer.

A.3.3 Constitution of the conical heap

The constitution of the conical heap was undertaken in four successive steps and needed three days. For each step, the dimensions of the conical heap were measured (see Table A.7). At the end, the conical heap had a height of 4,08 m, an average diameter of 14,72 m and a total mass of ± 250 t.

The height of the cone has been measured with a laser pointed on the top of the conical heap. The diameter of the heap has been measured with a target drawn beforehand on the floor.

Table A.7 — Characteristics of the conical heap for each step of its constitution

Steps	1	2	3	4
Downloading time (hh:mm:ss)	00:01:18	00:10:57	00:31:22	01:02:44
Downloading speed (t/h)	140	140	140	140
Added quantity (t)	3,02	25,58	73,22	146,38
Height of the cone (m)	1,01	1,78	3,03	4,16
Radius of the cone(m)	1,81	3,01	4,63	6,19
Added volume (m^3)	3,47	13,92	50,96	98,91

The objective of the step by step constitution of the conical heap was to establish a comparison between the sampling procedure (in the flow or from the heap) for increasing heap sizes.

A.3.4 Sample division

Two types of sampling were undertaken: in the flow and from the conical heap. The division of the samples was undertaken on site with riffle dividers. It wasn't possible to use rotary dividers because of the moisture content of the liming material.

A.3.5 Sampling in the flow

For each four steps of the constitution of the conical heap, a sampling in the flow was performed according to EN 1482-1:

— use of a stream sampling cup in line with the standard;

— sampling of the increments in the fall of the liming material;

— random sampling during the whole period of the liming material downloading;

— the number of sampling points for each step (see Table A.8) was always higher than the number specified in EN 1482-1, by Regulation (EC) No 2003/2003 [1] and in the composite protocol);

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

Table A.8 — Determination of the number of sampling points

Step	Mass (t)	Number of sampling points			
		EN 1482–1	**Regulation 2003/2003**	**Composite protocol**	**Trial 3**
1	3,02	10	8	-	**15**
2	25,58	11	23	-	**35**
3	73,22	18	39	-	**45**
4	146,38	25	40	40	**45**

For each step, the samples were mixed and then divided using riffle dividers. At the end of the division procedure, for each step, 6 final samples (± 500 g/sample) remained. They were distributed as follows:

a) 1 sample for the chemical analysis (LDAR);

b) 2 samples for the granulometric analysis (CRA-W);

c) 2 samples mixed again with the other final samples from the other steps of the constitution of the conical heap (see below);

d) 1 sample kept in stock (CRA-W).

Two final samples of each step were mixed again to obtain a sample of the whole heap of fertilizer. This remixed sample has also been divided following the same procedure and the 6 final samples of the whole heap have been distributed as follows:

e) 1 sample for the chemical analysis (LDAR),

f) 2 samples for the granulometric analysis (CRA-W) and

g) 3 samples kept in stock (CRA-W).

A.3.6 Sampling of the conical heap

For each step, specific samples have also been taken from the conical heap. For each intermediate cone, the number of sample units has been defined beforehand (see Table A.9).

These sampling units have then been distributed on each intermediate cone (see Figure A.1). Taking into account the actual size of the cone, the geometrical dimensions of the sample units have been calculated so that they have an approximately equal (identical) mass (see Table A.9).The calculation takes into account the previous cone.

Table A.9 — Number and size of the sample units for the 4 steps of the conical heap constitution

Step	Mass (t)	Number of sample units	Mass of the sample unit (t)
1	3,02	5	0,60
2	25,58	15	1,71
3	73,22	21	3,49
4	146,38	28	5,23

The dimensions and the distribution of the sample units have been visualized on the cone with the laser. To avoid walking on the heap and disturbing its structure, a bucket lift has been used to define the sample units and to sample. For each intermediate cone corresponding to the four steps of the conical heap constitution, the distribution of the sample units has been defined beforehand as shown in Figure 1. The arrow represents the conveyor belt and the direction of the liming material flow.

Sampling of the conical heap was undertaken following the proposed protocol and taking into account the composite protocol as follows:

— use of a tube shovel (penetration of ± 30 cm);

— sampling at the sampling points in each sample unit as pre-defined. The sample units have the same size;

— random sampling in the sample units;

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

The spear with double tube and multiple openings hasn't been used for the sampling of this product. The reason for this is that the product (wet chalk) isn't fluid at all. When the spear is driven into the heap, the wet chalk settles itself and doesn't fill up the spear. Therefore a new sampling tool has been developed (see Figure A.1).

Figure A.1 — Tube shovel

The tube shovel is a kind of shovel with a handle, and the plate of the shovel is a tube. The tube is ± 30 cm long and with a large diameter of ± 12 cm to 13 cm. There is no closing system at the entrance. Sampling is done into the surface of the product. This instrument, as a compromise, allows easy penetration of 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, ensure that the sample stays in the tube.

For each step, the samples from the sampling points are mixed and then divided using riffle dividers. At the end of the division procedure, for each step, six final samples (± 500 g/sample) remain. They were distributed as follows:

a) 1 sample for the chemical analysis (LDAR);

b) 2 samples for the granulometric analysis (CRA-W);

c) 1 sample mixed again with the final samples from the others sample units of the heap;

d) 2 samples kept in stock (CRA-W).

The final samples (c) of each sample unit have been mixed again to obtain a sample of the whole intermediate heap. This remixed sample has also been divided following the same procedure and the 6 final samples of the whole intermediate heap have been distributed as follows:

e) 1 sample for the chemical analysis (LDAR),

f) 2 samples for the granulometric analysis (CRA-W),

g) 1 sample mixed again with the other final samples of the intermediate cones and

h) 2 samples kept in stock (CRA-W).

Finally the four final samples of the four intermediate cones have been remixed and also subdivided to constitute the six final samples of the whole heap. These samples have been distributed within the labs to be analysed.

A.3.7 Chemical analysis

The chemical analysis undertaken by the LDAR supplied the CaO content and the Neutralizing Value (NV) of the final samples. The determination of the CaO content was performed on the basis of a usual and robust method which is used in France in the frame of Regulation (EC) No 2003/2003 [1]. The measurement is performed in a plasma torch after extraction with hydrochloric acid and dilution in a spectrophotometric buffer.

The Neutralizing value was determined by titration with an acid which allows the determination of the basic potential of the liming material.

A.3.8 Granulometric analysis

The size distribution was determined following a wet sieving method according to EN 12948. The size distribution measurements were repeated two times for each sample unit. 166 samples have been analysed finally.

The sieve analysis undertaken by the CRA-W supplied the granulometric parameters of diameters D16, D50, D84 and the D80. The last one has been calculated by semi-log interpolation (interpolation given by MEAC). The wet sieving also supplies directly the Moisture content (M) in percent on a wet basis.

The complex procedure of sampling which was developed within the framework of the project aims at:

1) generating a reference for the comparative analyses of each parameter (chemical content/characteristics and particle size distribution) with the average of the results of the 4 representative samples taken from the liming material flow at the level of the conveyor belt;

2) generating a cartography in three dimensions of the conical heap indicating the chemical and granulometric characteristics of the liming material;

3) being able to simulate different protocols of representative sampling in the conical heap and compare their results with the reference;

4) being able to study the influence of the size of the conical heap on the representativeness of the studied protocols of sampling.

Five protocols for sampling the conical heap were simulated and their results compared with the reference:

5) Simulation 1 (S1): complete sampling,

6) Simulation 2 (S2): partial sampling around the base of the cone,

7) Simulation 3 (S3): partial sampling by following a cone edge,

8) Simulation 4 (S4): reduced random sampling (5 samples), and

9) Simulation 5 (S5): widened random sampling (10 samples).

The statistical analysis bases itself on a multiple comparison of averages, which allows the determination of any significant differences between the series of observations. **It is however necessary to verify the relevance of these differences from a practical and/or regulatory point of view**.

A last protocol for sampling has been performed (and not simulated) during the trial on the final heap of 250 t. This last protocol is a variation of the sampling procedure by following a cone edge based on GOST-Standard (Russian State Standard) (see [2]) found in the literature.

A.3.9 Conclusions of the comparative analyses

The conclusions of the comparative analysis are the following ones:

1) The liming material tested has a very wide particle size distribution and is subject to great segregation. This segregation significantly affected the results. During the formation of the heap, the largest particles run down to the bottom of the heap while the finest particles concentrate at the top of the heap. However, the size segregation affected the particle size distribution more than the chemical content and characteristics of the samples taken from the conical heaps.

2) Because of the segregation, the variability between the samples taken on the heap is very high for the particle size distribution parameters. This high variability impacts on the statistical tests which are no longer able to detect differences between the tested sampling protocols and the reference one (Flow). However, from a practical point of view it can be stated that the particle size distribution is generally significantly different between the tested protocols and the reference method.

3) Contrariwise, the variability of the CaO content, the Neutralizing Value (NV), and to a lesser extent the moisture content of the samples is very low wherever the samples are taken from the heap (base, middle or top). This low variability also impacts on the statistical tests which highlight differences when from practical point of view there is none.

4) For the CaO content and the NV, all simulations, with the exception of the second one, give consistent results comparable with the reference sampling protocol. Even though the statistical test highlights a significant difference for the CaO content and NV, from a practical point of view, all the tested sampling protocols gave comparable results to the reference one. For the second simulations (sampling around the base of the cone), the differences between in flow and static heap sampling were statistically significant for the step 3 of the constitution of the conical heap (see Table A.10). But these differences are not practically different.

5) For the particle size distribution, the conclusion is the opposite. Even though the statistical test doesn't highlight any differences between the samples taken from the heap and from the flow, there are significant differences. The D50 is the most affected parameter.

6) For the moisture content the conclusions are more complex. This is explained by the delay between taking the samples and the measurement of the moisture content. The resulting storage of the samples affected the reliability of the measurements.

7) Definitely, the partial sampling around the base of the heap **does not give** a representative sample. This conclusion is confirmed both by the statistical tests and by the interpretation of the results.

Table A.10 — Differences between in flow and heap sampling

Step No	Sampling procedure	Content CaO %	NV
Step 3	In flow	47,991	47,161
Step 3	From heap	48,942	48,124

Annex B
(informative)

Literature review

B.1 Introduction

A review was undertaken to look for standards and/or best practice guidelines used by different stakeholders in the fertilizer sector and legislation in different countries.

B.2 Inventory of standards

This section concerns documents published by CEN, ISO and the American National Standards Institution (ANSI).

The main standard used in many European countries (Germany, Austria, Sweden, France, Belgium and The Netherlands) is based on EN 1482-1:2007 **"Fertilizers and liming materials – Sampling and sample preparation – Part 1: Sampling"**. This European Standard specifies the sampling plans and the sampling methods for fertilizers and liming materials that produce representative samples for chemical and physical analysis. The described sampling methods concern solid and liquid fertilizer in packages or containers and in bulk during loading/unloading or filling/emptying of storage tanks. Once the fertilizer is in motion the recommended number of sampling units is 40 where the original heap was more than 400 t; one increment per sampling unit should be taken. The minimum mass of an increment is 250 g; except for blended fertilizers and for liming materials where the minimum mass of increment should be increased to 500 g.

When consulting the online database of ANSI, which allows finding standards used in the world, the reference is EN 1482-1 also. No specific American standard was found.

Another standard that gives information about fertilizer sampling is ISO 8634:1991 **"Solid fertilizers — Sampling plan for the evaluation of a large delivery"**. This International Standard specifies a method for sampling a delivery of more than 250 t of fertilizer and, after analysis of the sample(s), presents rules for assessing the quality. For bulk storage, the sampling should be done during loading or unloading.

ISO 8633:1992**: "Solid fertilizers – Simple sampling method for small lots".** This International Standard defines a sampling plan for the control of quantities of solid fertilizer < 250 t and outlines the method to be used. It is applicable to all solid fertilizers which may be in bulk or in packages. It mentions that sampling from a running bulk stream by mechanical devices is to be preferred over manual sampling from the static heap. The minimum number of sampling units = 10 for all sample portions up to 5 t. Above 5 t, the number should be taken as the square root of 20 times the tonnage present in the sample portion. The total amount should be divided into a number of equal parts; this number corresponds to the number of sampling units to be selected. One increment per sampling unit should be taken using a shovel or a scoop.

B.3 Inventory of best practice guidelines

Beside the standards, less formal documents are used by different stakeholders of the sector who needs to obtain samples from bulk material for different purposes: process following by industry, analysis for research projects, quality control systems, certification body or association responsible for controls.

Documents from the International Fertilizer Industry Association (IFA)

Best practices for the sampling of dry bulk fertilizer shipments: In order to limit the disputes about the quality parameters of shipments between international trading parties due to the application of different methods for fertilizer sampling, IFA examines means to harmonization sampling practices. This work results in best practice recommendations prepared by the working group on the harmonization of fertilizer sampling and methods of analyses. The mechanical sampling systems (MSS) should always be the preferred procedure, when available, as these systems have been found to be the most reliable and consistent means of taking representative samples. The best practices allow the use of manual sampling in the absence of MSS, but this sampling shall be conducted when material is being transported along a conveyor belt and/or in a falling stream (from a belt or a chute). An in-depth review by the IFA working group has shown that the Association of Fertilizer and Phosphate Chemists (AFPC) has adopted a suitable manual sampling scheme that is widely used and recognized by the global fertilizer industry.

The sampling of bulk fertilizer in static heap piles (concept note from working group of IFA on the harmonization of fertilizer quality analysis and sampling methods): Samples are often taken from bulk materials in a static pile. Such sampling is prone to systematic error compared to sampling a moving product. The sampling of bulk fertilizer in static heap piles contradicts the theory of sampling in which all parts of the sampled unit should have an equal probability of being collected in the sample. It shall be noted that stockpile sampling should not be considered as a preferred method for obtaining a representative sample. The results from samples obtained in static heaps should only be considered representative of the part of the pile that was within reach during sampling. An overview was made about industry practices used by some of their members for static heap sampling, but the information given isn't precise enough to be useful for this report.

European Fertilizer Blenders Association (E.F.B.A.): Handbook of solid fertilizer blending, code of good practice for quality (second edition 2005/2007): The recommended method of sampling described in the handbook, derives from EN 1482 and concerns only fertilizers in motion.

Canadian Food Inspection Agency, 1997: Sampling procedure for sampling fertilizers: This document specifies procedures and information concerning fertilizer sampling. For the sampling of dry products in bulk, the Missouri D tube spear shall be used. The spear is to be used in the vertical plane, inserted in the open position to its full effective length where possible, closed and withdrawn.

Some research institutes have to take samples during their activities and use specific methods for fertilizer sampling. An interesting example is the **German Agricultural Analytic and Research Institute (VDLUFA).** In comments made on EN 1482-1:2007 by this institute, we found a more detailed (compared to other references) description of sampling bulk goods, heaps and piles. More information about sampling can be found in:

— Methods Book II.1, "Fertilizer analysis";

— Methods Book II.2, "Testing of Secondary Fertilizers, Growing Media and Soil Improvers".

Both books are edited by VDLUFA and are only available in German.

B.4 Inventory of legislation

The legislation covers the sampling of fertilizers for the purpose of officials' controls.

Regulation (EC) No 2003/2003 of the European Parliament and of the council relating to fertilizers; Annex IV: Methods of sampling and analysis. (JO L304 21/11/2003): This Regulation gives two examples of apparatus that can be used for manual sampling: flat-bottomed shovel with vertical sides or sampling spear with a long split or compartments. The minimum number of increments depends on the size of the sampled portion, between 2,5 t up to 80 t, $n = \sqrt{20 \times \text{mass of sampled portion}}$. For sampling portions exceeding 80 t, the minimum number of samples is 40. No information is given about the size of one increment but the total aggregate sample should be 4 kg with incremental samples of approximately equal sizes. The samples shall be taken at random throughout the whole sampled portion.

CE 77/535 Commission Directive on the Approximation of the laws of the member's state relating to method of sampling and analysis of fertilizers – Annex 1: Methods of sampling for the control of fertilizers: See Regulation (EC) No 2003/2003.

BE AR 4 July 2004 fixing the measures as regards official sampling of fertilizers, liming materials and substrates for crops. Annex: method of sample selection. This order ensues from the Regulation (EC) No 2003/2003.

Annex C
(informative)

Sampling instruments - Equipment for static heap sampling

C.1 General

The equipment depends on the nature of the product (particle size and fluidity) and the number and 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 contamination between samples. For manual sampling the use of spears makes it possible to work at different depths inside the bulk. The minimum size of increments recommended in the previous text indicates that the spear should be capable of taking a sample of 500 g.

C.2 Manual spears

The opening in the spear should be at least two (three would be better) times the maximum diameter of the largest particle in the product. The spears are usually made of stainless steel or aluminium.

C.3 Monotube spear

This spear with one tube has a minimum diameter of 2 cm and a length from 65 cm to 90 cm. This equipment is mostly used for horizontal sampling in bags. When introducing the spear, the opening in the spear should face downwards the spear is then turned upwards to allow product to fall into it and it is then removed keeping it in the upward position.

C.4 Nobbe spear

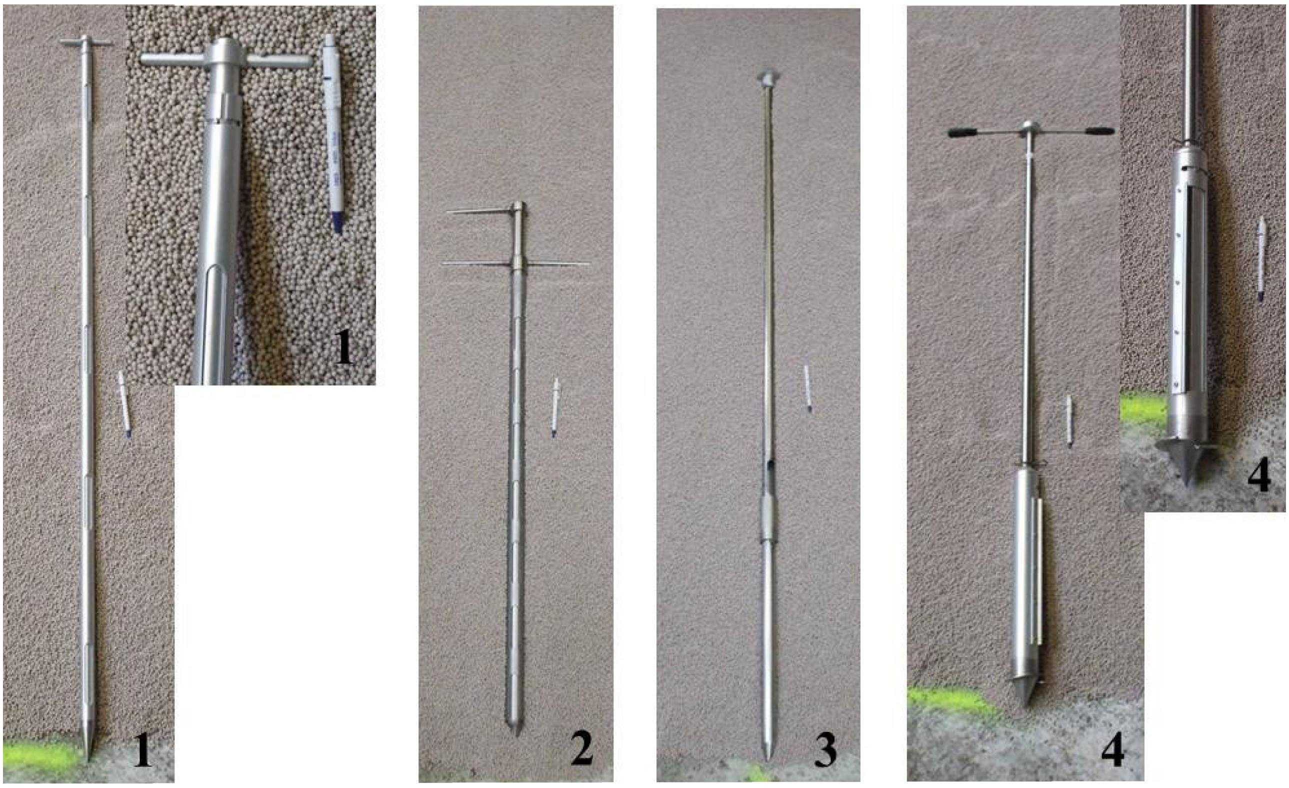
It is recommended that this spear be used with one single opening in a horizontal ways in C.3.

C.5 Double tube spear

This spear comprises one tube fitting closely inside a second one. The internal part could be moved by a handle. The main tube is ended by a hard point. Both tubes show openings in their surfaces, the spear is opened by turning the internal tube until openings are matching. The double tube spear type is suitable for sampling big packages or bulk.

C.6 Test of manual spears

During the 1st trial, four spears were tested and evaluated from a practical point of view. The tested equipment is illustrated in Figure C.1.



Key

1 double tube, 1210K-1500Cereals, Maes laboratory, L=1,5 m, Ø=40 mm, 5 openings,

2 double tube, Fertilizer, home made from Yara, L=1,17 m, Ø=30 mm, 7 openings,

3 monotube, Tekpro Cereals, Cereal Tester, L=2,0 m, Ø=28 mm, 1 opening,

4 monotube, Liming material, LDAR, L=1,5 m, Ø=55 mm, 1 opening,

Figure C.1 — Tested spears, stainless steel

The ease of use for each spear was evaluated: penetration, lock on and lock off system, sampling. Table C.1 summarizes the results of the tests.

Table C.1 — Assessment of the use of the tested spears

No of the spear	Disadvantages	Advantages
1	quite heavy, no handle, distance between two openings is to long (± 20 cm) penetration: max 90 cm	increment of 500 g locking system easy to use point is sharp
2	not commercially available point end not sharp enough penetration: max 90 cm	good handle increment of 500 g locking system easy to use
3	no handle one opening locking system penetration max 90 cm increment of 250 g	–
4	hard penetration crush the fertilizer difficult to empty	good handle increment of 750 g

C.7 Consideration

When choosing a spear, from a practical point of view, we should consider the size of the sample required, the shape and size of the handle, the penetration, the ease of taking the sample and discharging it from the spear.

The spears with double tube are the most used for sampling from bulk because they are more solid and easier to use. They can be used both vertically and horizontally in order to take samples from different shaped heaps of bulk fertilizer. The design of the spear: shape, number of openings, handle linked to the sample products had a great influence on the ease of use.

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