EUROPEAN FERTILIZER BLENDERS ASSOCIATION

HANDBOOK OF SOLID FERTILISER BLENDING

Code of Good Practice for Quality

Third Edition

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This handbook is also available in French or German

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1. INTRODUCTION

This Handbook of Solid Fertiliser Blending has been prepared by the European Fertilizer Blenders Association (EFBA) to help members achieve their targets:

1 The identification of crop needs, in consultation with farmer customers.

2 The production of high quality blended fertilisers for the agricultural market to satisfy these needs.

3 The accurate delivery and spreading of these fertilisers.

This handbook sets out advice which, if followed, should lead to improved quality of finished products, and a wider acceptability of blended fertilisers in the European marketplace.

1.1 BLENDED FERTILISERS

The blending of solid granular materials to produce a wide range of compound fertilisers has been successfully practised for over 40 years. In some countries blends form by far the major proportion of compound fertilisers sold.

Blended fertilisers have three main advantages over complex fertilisers. The first is that of versatility, the second is a simple matter of economics and the third is a better protection of the environment. Using a limited number of base materials, an almost infinite range of compounds can be produced. These can be specifically matched to local soil conditions and plant needs, thus avoiding excess nutrients which may enter the environment. Multiple handling of the basic raw materials such as phosphate rock, is avoided by maintaining the primary production processes at the source of the nutrients. The materials which then need to be transported to the agricultural areas contain the highest possible amounts of nutrients, thus reducing overall costs.

The main disadvantage of blended fertilisers, in comparison with complex fertilisers, is the risk of segregation of the components during handling or spreading. However, this book shows how to avoid segregation.

1.2 FERTILISER BLENDERS

Blenders, in common with other fertiliser producers, have a clear responsibility to ensure that their products are of high quality. Raw material producers have a clear responsibility to ensure that the materials they supply to the blenders are also of a consistently high quality and conform to the agreed specifications. That is to say, they supply materials of guaranteed analysis in terms of their nutrient contents and physical properties.

The blender is responsible for his choice of equipment and for specifying the quality of raw materials he is purchasing. In addition he has a responsibility to check the quality of the raw materials he receives and to monitor his production at all stages to ensure finished products are of the highest quality.

Fertiliser blenders are handling materials which, if mismatched or mishandled, can result in very poor quality finished fertiliser which will not transport, store or spread satisfactorily.

There is a clear economic benefit to suppliers and users alike in improving the quality of blended fertilisers. Poor quality products can have serious consequences, both legal and financial.

The Solid Fertiliser Blending Handbook contains practical advice to help manufacturers achieve high quality blends.

1.3 SERVICES AND SOIL ANALYSIS

Whilst the blender's responsibility for the product effectively finishes with its delivery to farm, the responsibility to provide as complete a service as possible does not. Indeed, with modern systems of custom blending, the responsibility starts and finishes on the farm.

Blending can be considered to be a complete system provided for the farmer, starting with soil sampling, analysing its nutrient requirements, and finishing with the accurate spreading of the correct blend of nutrients.

2. DEFINITIONS

Throughout this handbook the general terms **blend** and blending are used to denote the product and the process of mixing granular materials. Blended fertilisers may be supplied to farmers in bags or in bulk. The term **bulk-blend** is reserved for situations where the blend is handled (stored, transported) in bulk rather than in bags.

Blends do not necessarily consist of mixtures of straight fertilisers or single compounds. A blend of two or three granular complex fertilisers will still be a blended fertiliser.

A number of other specific terms are used and these are defined as follows:

Blended fertiliser: Fertiliser obtained by dry physical blending of various raw fertilisers, without any chemical reaction.

Complex fertiliser: Compound fertiliser obtained by chemical reaction, by liquid solution or, in the solid state, by granulation and having a declarable content of at least two of the major nutrients.

Note 1: For the solid granules, each particle contains all the nutrients approximately in their declarable content.

Note 2: Some Companies use the term "uniform" to mean a complex fertiliser and to indicate the product is not a blend.

Compound fertiliser: Fertiliser having a declarable content of at least two of the nutrients nitrogen, phosphorus and potassium, obtained chemically or by blending, or both.

Note: With these definitions, mono and di-ammonium phosphates and potassium nitrate are not "straights" but are NP and NK complex fertilisers respectively.

d_{50} (Mean particle size): That size such that half the particles, by mass, are larger than that size and half are smaller.

Granular fertiliser: Solid fertiliser formed into particles of a predetermined mean size by granulation.

Note: In some countries, this term is often (wrongly) used to mean complex fertilisers.

Granulation: Technique using processes such as agglomeration, accretion, compaction, to modify the particle size.

Granulometric spread index (GSI): Measure of the spread of particle sizes and a means of expressing the granulometric spread.

Increment: Representative quantity of material taken from a sampling unit.

Lot: Total quantity of material, assumed to have the same characteristics, to be sampled using a particular sampling plan.

Mean particle size (d_{50}): That size such that half the particles, by mass, are larger than that size and half are smaller.

Particle size: Dimension which corresponds to the smallest sieve aperture size through which a particle will pass if presented in the most favourable attitude.

Particle size analysis by sieving: Division of a sample by sieving into size fractions.

Raw material: Solid, granular material used as a component in a blended fertiliser.

Note: Some of these materials are not the basic source materials which provide the nitrogen, phosphate and potash. In these cases, they are often known as intermediates or pre-mix.

Segregation: Differential movement of particles within a mixture due to differences in their size, shape or density, resulting in their separation.

Sieving: Process of separating a mixture of particles according to their sizes by one or more sieves.

Size guide number (SGN): 100 times the d_{50} measured in millimetres

Spreading width: Distance between the extreme left and right points where the fertiliser arrives on the ground.

Straight fertiliser: Qualification generally given to a nitrogenous, phosphatic or potassic fertiliser having a declarable content of only one of the plant nutrients nitrogen, phosphorus or potassium.

Note: It is possible for a straight fertiliser to be a blend. For example a mixture of granular ammonium nitrate and granular ammonium sulphate would be a straight nitrogen fertiliser.

Test sieving: Sieving with one or more test sieves.

Working width: Distance between each passage (generally between 12 and 48 m) when spreading fertilisers.

Definitions of other technical terms may be found in EN 12944 Fertilisers and Liming Materials, Vocabulary, Part 1 [8].

3. RAW MATERIALS

The quality of the finished product depends almost entirely on the raw materials. Good blending starts with good raw materials. It is not realistic to expect to make good quality blended fertilisers from poor raw materials. The specification, purchase and checking of raw materials must be the first priority of the blender.

Every raw material should be bought to as tight a specification as possible. Deliveries must be checked regularly, preferably by independent inspectors, to ensure consistent quality. All sampling and testing should be carried out using methods agreed between supplier and purchaser, based either on National or European legislation or on accepted International Standards (CEN, ISO etc.). More information about sampling will be found in section 8.

Whilst many fertiliser raw materials may be considered to be commodities, rather than speciality chemicals, the opportunistic purchase of spot consignments of doubtful origin and quality is NOT RECOMMENDED. No raw material should be purchased without an agreed contractual specification covering, as a minimum, the registration to REACH, the chemical analysis and the particle size details.

3.1 CHEMICAL PROPERTIES

The nutrient content of each raw material used must be known in order to prepare formulations for the different compounds required. Raw material suppliers should be asked to supply certificates of analysis for each large consignment. Where consignments differ markedly they should be stored separately and the formulations adjusted to take account of the true analysis figures (see Chapter 9)

The water content of each raw material used must be known in order to ensure compatibility between raw materials.

In all cases it is advisable to make occasional random checks by arranging for representative samples to be taken by independent inspectors. These samples should be analysed as soon as possible and before the consignment is used.

3.1.1 Compatibility

Some raw materials are not compatible with others and blends containing such mixtures will be of very poor quality. The compatibility data are presented in the table below in three categories (Figure 1).

Further information regarding compatibility may be found in Reference 13

	Ammonium Nitrate		Calcium Ammonium Nitrate	Calcium nitrate (fertilizer grade)	Ammonium sulphate nitrate	Potassium Nitrate / Sodium nitrate	Ammonium sulphate	Urea	Rock Phosphate	Partially acidulated rock phosphate	Single/Triple super phosphate	Monoammonium phosphate	Diammonium phosphate	Mono potassium phosphate	Potassium chloride	Potassium sulphate/ magnesium sulphate (kieserite)	NPK, NP, NK (AN based)	NPK, NP, NK (Urea based)	Limestone/dolomite/calcium sulphate/Calcium carbonate	Sulphur (elemental)
Ammonium Nitrate				1	2		3	NC 1		4	4				5		5	NC 1		NC 2
Calcium Ammonium Nitrate				6	2		2	NC 1			7				5		5	NC 1		NC 2
Calcium nitrate (fertilizer grade)	1		6		8	8	8	8			8	8	8	8	8	9	8	8		8
Ammonium sulphate nitrate	2		2	8		2		NC 1	10	4	7				5		5	NC 1		NC 2
Potassium Nitrate / Sodium nitrate				8	2		11										12	13		NC 2
Ammonium sulphate	3		2	8		11											5			
Urea	NC 1		NC 1	8	NC 1					14	15				16		NC 1			
Rock Phosphate					10															
Partially acidulated rock phosphate	4				4			14					17							
Single/Triple super phosphate	4		7	8	7			15					17				4	14	17	
Monoammonium phosphate				8																
Diammonium phosphate				8						17	17									
Mono potassium phosphate				8																
Potassium chloride	5		5	8	5			16									5			
Potassium sulphate/magnesium sulphate (kieserite)				9																
NPK, NP, NK (AN based)	5		5	8	5	12	5	NC 1			4				5			NC 1		NC 2
NPK, NP, NK (Urea based)	NC 1		NC 1	8	NC 1	13					14						NC 1			
Limestone/dolomite/calcium sulphate/Calcium carbonate											17									
Sulphur (elemental)	NC 2		NC 2	8	NC 2	NC 2											NC 2			

Figure 1. COMPATIBILITY OF VARIOUS SOLID INORGANIC FERTILIZERS

	Compatible
	Limited compatibility linked to quality issues
	Limited compatibility related to safety or regulatory issues
NC	Not Compatible

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Notes for the numbers in the boxes in the compatibility table

Limited Compatibility

1. Due to the hygroscopic behaviour of both products, the type of stabilisation of the ammonium nitrate grade could influence storage properties.
2. Consider the safety implications regarding detonability of the blend (AN/AS mixtures) and legislative implications.
3. Consider the safety implications regarding detonability of the blend (AN/AS mixtures), impact of free acid and organic impurities, if present, and legislative implications.
4. If free acid is present it could cause very slow decomposition of AN, affecting, for example, packaging.
5. Consider the possibility of self-sustaining decomposition and the overall level of oil coating.
6. Due to the hygroscopic behaviour of both products, the type of stabilisation of the ammonium nitrate based fertilizer could influence the storage properties.
7. Consider the moisture content of the SSP/TSP.
8. Consider the relative humidity during blending.
9. Risk of formation of gypsum.
10. No experience but this can be expected to be compatible. Confirm by test and/or analysis.
11. Consider impurities in AS and the drop in the critical relative humidity of the blend.
12. Consider the likely impact of additional nitrate.
13. Consider the possibility of ammonium phosphate/potassium nitrate reaction with urea and relative humidity during blending to avoid caking.
14. If free acid present, there is a possibility of hydrolysis of urea giving ammonia and carbon dioxide.
15. Formation of very sticky urea phosphate.
16. Potential caking problem due to moisture.
17. If free acid is present, consider the risk of a reaction e.g. neutralisation with ammonia and acid attack with carbonates.

Not Compatible

NC1. Mixture will quickly become wet and absorb moisture resulting in formation of liquid or slurry. There could also be safety implications.

NC2. Sulphur is combustible and can react with nitrates e.g. AN, KNO_3 and $NaNO_3$.

From the chart, it is clear that urea and ammonium nitrate should never be used together as the mixture will quickly become wet and absorb moisture. Blends containing urea and single or triple superphosphate may also become sticky and cake. Such blends should never be bagged. Mixtures of di-ammonium phosphate and superphosphates should be avoided as chemical reactions may take place which can lead to caking or changes in the solubility of the phosphate.

For reasons of safety, it is very important to avoid blending ammonium nitrate or raw materials containing ammonium nitrate with any organic materials.

3.2 PHYSICAL PROPERTIES

The most important physical property - as far as blending is concerned - is **the particle size distribution**.

The particle size distribution must be known in some detail and the specification must include a full description of this property. At the very least, **the mean particle size (as measured by the d_{50}) must be specified**. Ideally, the specification should include a measure of the granulometric spread index (GSI) and should also include maximum values for the amounts of oversize (for example > 5 mm) and fines (for example < 1 mm).

The shape and the density of particles could have an influence on the behaviour of the fertiliser during spreading.

Other physical characteristics to be specified must include "free flowing" and "dust free" and possibly hardness and impact resistance. However, these properties are much more difficult to assess using standard test methods. More realistically, it is better to specify that suitable anti-caking and/or anti-dust treatment is applied to the raw material and that it should not break down during handling.

In all cases it is advisable to make occasional random checks by arranging for representative samples to be taken and assessed by independent inspectors. It is also recommended that samples be taken during the delivery and tested for size distribution at the blending plant. See Section 5.4)

3.2.1 Particle Size

The key factor in producing quality blends is the **size compatibility of the raw materials**. Unless all the ingredients are well matched, segregation will take place every time the blend is handled in bulk. This will lead to unevenness of chemical analysis and possibly, uneven spreading of nutrients on the crops.

The particle size distribution can be expressed in a number of ways but all rely on a sieve analysis of the material. **It is essential therefore that blenders should have the facility to carry out a full sieve analysis of their raw materials** (see chapter 8).

A number of simple field test devices are available but these are limited in their ability to measure particle size distribution adequately. They are however, very useful to carry out spot checks on raw materials being fed to the blender.

The official method of test sieving is fully described in European Standard EN 1235 and Amendment A1 [3]. A number of numerical parameters may be calculated from the sieve analysis. These include the mean particle size (d_{50}) and granulometric spread index (GSI). In view of the importance of size distribution, full descriptions of these parameters and the use of the various systems are described below.

However, for the best results, it is recommended that a full size distribution curve is plotted for all raw material samples tested. A description of the test sieving technique is given in paragraph 8.3.1. Size distribution curves can readily be superimposed to give a quick comparison and an indication of compatibility.

The mean particle size (d_{50}) is determined using the following equation: (1)

$$d_{50} = z_n + \frac{(50 - c_n)}{(c_{n+1} - c_n)} (z_{n+1} - z_n)$$

Where:

1. z_n is the nominal sieve mesh in mm for which the cumulative undersize is nearest to but below 50% by mass
2. z_{n+1} is the nominal sieve mesh in mm for which the cumulative undersize is nearest to but above 50% by mass
3. c_n is the cumulative percentage undersize for sieve n
4. c_{n+1} is the cumulative percentage undersize for sieve n+1

NOTE: d_{84} and d_{16} are calculated in the same way by substituting 84 and 16 respectively for 50 in equation (1) above.

An excellent measure of the spread of particles sizes can be obtained using the whole of the linear part (between d_{84} and d_{16}) of the distribution curve obtained from the sieve analysis. The values of d_{84} and d_{16} may be found directly from the graph or by calculation. The spread is the difference between the two:

$$\Delta = d_{84} - d_{16}$$

An important value, known as the **Granulometric Spread Index (GSI)**, is derived from the following formula:

$$GSI = \frac{\Delta}{2\ \mathrm{d}_{50}}\ 100 \qquad \text{or} \qquad GSI = \frac{d_{84} - d_{16}}{2\ \mathrm{d}_{50}}\ 100$$

3.2.2 Bulk Density

The bulk density of the fertiliser may be measured in accordance with EN 1236 (Loose density) [4] or EN 1237 (tapped density) [5]. The general principle is to weigh the contents of a cylinder of a known volume. For the tapped density, the cylinder is subject to vibrations and compaction occurs. This value is always higher than the loose density.

The density of the fertiliser can have an influence on the behaviour of the particles during spreading on the field. Severe segregation may occur if the densities are very different (see section 5.3).

Generally, the loose bulk density of fertilisers is between 900 and 1100 kg/m³ but extreme values can be between 750 and 1350 kg/m³. In practice these extreme values rarely occur simultaneously.

3.2.3 Shape

The measurement of the shape of the fertiliser particles is not easy. Generally, it is necessary to use image analysis techniques. However, the measurement of the angle of repose of a heap formed by a fertiliser flowing from a funnel can be a useful guide to this parameter. The method is standardised in EN 12047 [6] and described in paragraph 8.3.3. Angles of repose vary from about 30° for the most spherical products to 40° for the most angular.

3.2.4 Particle Hardness

During handling and spreading, the fertiliser will be submitted to stresses which can break the particles, for example the impact with the vanes during spreading. This process leads to the production of small grains which cause some problems (segregation, caking). For this reason the particles should be of a sufficient hardness. Unfortunately, the test methods are not standardised because of the variability of the measurement and the evolution of these parameters with time. If the particle hardness is low, the fertiliser may contain too many small particles and thus may no longer meet the quality criteria (see section 5.4).

3.2.5 Dust Content

Some fertilisers have the tendency to produce large amounts of dust. This can cause problems in the neighbourhood of the plant and accentuates the risk of caking. There is no standard test method but with some experience the raw materials presenting this problem are easily detected.

3.2.6 Flow Rate

Normally, the fertiliser must flow freely. If there is any caking or some reaction between the components or with moisture, the flowability of the fertiliser can be reduced. This can lead to problems for all the handling operations.

A standard method for the measure of the flow rate has been developed as EN 13299 [7]. About 2 kg of fertiliser is placed in a standard funnel which has a closed aperture of 25mm diameter. Then the aperture is opened and the time for 2 kg to flow out of the funnel is measured. The apparatus is calibrated with defined glass spheres.

Table 1: Common blending raw materials

Name	Abbr	Formula	N	P_2O_5	K_2O	SO_3	MgO	CaO
Ammonium Nitrate	AN	NH_4NO_3	33-34,5					
Calcium Ammonium Nitrate	CAN	$CaCO_3/NH_4NO_3$	26-28					11
Ammonium Sulphate Nitrate	ASN	$(NH_4)_2SO_4/NH_4NO_3$	26			35		
Ammonium Sulphate	AS	$(NH_4)_2SO_4$	21			60		
Urea		$CO(NH_2)_2$	46					
Superphosphates:								
Single Superphosphate	SSP	$Ca(H_2PO_4)_2$*		18-20		30		
Triple Superphosphate	TSP	$Ca(H_2PO_4)_2$*		45-48		3		
Potassium Chloride	MOP	KCl			60-62			
Potassium Sulphate	SOP	K_2SO_4			50	45		
Korn-Kali®		$KCl/MgSO_4$			40	12	6	
Potassium Magnesium Sulphate		$K_2SO_4 + MgSO_4$			30	42	10	
Ammonium Phosphates :								
Di-ammonium Phosphate	DAP	$(NH_4)_2HPO_4$	18	46-48				
Mono-ammonium Phosphate	MAP	$NH_4H_2PO_4$	12	52-53				
Calcium Carbonate		$CaCO_3$						52
Compacted Dolomite		$CaCO_3$ - $MgCO_3$					20	30
Magnesium Carbonate		MgCO3					10	40
Kieserite		$MgSO_4$				50	25-28	

3.3 STORAGE

Raw material storage must be arranged to avoid:

1. segregation within the materials
2. cross contamination
3. deterioration of the physical quality.

Storage must be arranged to ensure adequate identification of the raw materials.

The recommended type of storage is the horizontal or open bin layout. Ideally the bin should be fed from a conveyor belt system with the discharge fitted with an anti-segregation system such as a spinner or flow splitter (Figure 5).

The European Fertiliser Manufacturers' Association (EFMA) has published detailed guidance on the safe storage of fertilisers. [12]

3.3.1 Moisture Pick-up

Some fertiliser raw materials are hygroscopic which means they can pick up moisture from humid air. Stores holding these materials should be air-conditioned or the material should be covered when not being used. Figure 2 shows the critical relative humidity for a number of common blend components and mixtures. The lower the critical relative humidity, the more moisture will be taken from the air. Generally the phosphates including the ammonium phosphates have a high critical relative humidity and thus almost never present hygroscopic problems. The opposite applies to nitrates such as calcium ammonium nitrate, ammonium nitrate, and especially calcium nitrate.

For blended and complex fertilisers the critical relative humidity in most cases is **below the average** derived from its components. This can be seen when looking at the data for PK and NPK fertilisers. An extreme example for this is demonstrated by the critical relative humidity of a blend consisting of urea and ammonium nitrate. Such a blend would pick up moisture so quickly that it would be impossible to handle it in a dry state, even if spreading occurs immediately after blending.

COMPONENT	CRIT. REL. HUMIDITY
Triple Superphosphate	93,6 %
Mono-ammonium Phosphate	91,6 %
Di-ammonium Phosphate	82,5 %
Ammonium Sulphate	79,9 %
Potassium Chloride	77,0 %
Urea	74,6 %
Sodium Nitrate	72,4 %
Calcium Ammonium Nitrate	61,3 %
Ammonium Nitrate	59,4 %
Calcium Nitrate	46,7 %
BLENDS OR COMPOUNDS	
PK Fertilizers	69,4 %
NPK Fertilizers	64,7 %
Urea - Ammonium Nitrate	18,1 %

Average data for 30 °C from Adams, Merz et al., Runge et al., Silverberg, Löhner.

Figure 2: Critical relative humidity of fertiliser salts and mixtures [15]. Values are % relative humidity at 30°C.

3.3.2 Contamination

Cross contamination of raw materials should be avoided as this will obviously affect their chemical analysis and hence the final analysis of the blends.

Ammonium nitrate and other materials containing ammonium nitrate must be kept well clear of organic materials.

Good housekeeping is vital to any blending operation. All spillages should be swept up as soon as possible and all equipment kept clean. Overhead conveyors must be kept in good condition to minimise spillage into other storage areas. The use of special chutes to avoid excessive dust is strongly recommended.

Further information on the prevention of contamination is given in Reference 12

3.4 SCREENING

Raw materials should be screened before being fed to the blending unit to remove any lumps or fines which may have formed during storage. If screening is performed after weighing and blending, some of the raw material will be lost and this will alter the composition of the final mix. In such cases there can be no control on the final analysis of the blend. In addition, the screening process may seriously un-mix the components. The use of a scalping screen (for example 10mm mesh) to remove any lumps which may have formed in the system, is acceptable.

3.5 SAMPLING AND TESTING

Random spot checks of the raw materials being fed to the blender are recommended. These may be simple checks of the particle size distribution but if there is any doubt, representative samples should be taken and examined fully.

For further information about sampling and physical test methods, refer to Chapter 8.

3.6 MICRONUTRIENTS AND CONDITIONING AGENTS

3.6.1 Micronutrients

It is relatively simple to add micronutrients to blended fertilisers either as granular or powder materials, but the homogeneity of distribution is particularly important. As a guide, granular materials should not be used if the micronutrient carrier represents less than 5% of the total weight. In these cases it is preferable to add the micronutrient in powder form with a binding agent to ensure good adherence to the granules. Suitable binding agents include heavy viscosity oils, UAN solutions and water. Alternatively, the micronutrients may be added as a solution which is sprayed directly onto the blend in the mixer. Note however, that oil should not be used when ammonium nitrate forms part of the formulation.

Some national regulations do not allow the use of oil: please check this point carefully.

3.6.2 Conditioning Agents

Anti-caking agents are not normally required if the product is to be used immediately. Products which are to be stored or bagged may require the addition of a small amount of anti-caking agent. It is recommended that a dust-suppressant is added to the blend.

Conditioning agents may be applied in the same operation as the micronutrients.

4. BLENDING EQUIPMENT

All blending plants should include the following equipment:

1 Weighing or other proportioning device(s)

2 Dry mixer

3 Loading or bagging equipment

In addition, some control equipment is desirable but not essential if the other equipment is reliable.

4.1 SELECTION

This handbook does not set out to recommend equipment over any other but there are various types and their characteristics and advantages are different. The capacity of the equipment should be chosen to match output requirements in terms of tonnes per hour and tonnage per week.

4.1.1 Weighing

In batch operations, the whole equipment may be mounted on a load cell or belt weighers may be used. For continuous blending, individual belt weighers may be fitted to the belts feeding the raw materials to the mixer. The size and type of such equipment should be chosen to match the plant size and outputs required.

4.1.1.1 Unit Batch Weighing

These are the most popular systems. The outputs range from 20 to 100 tonnes per hour. Smaller systems have a floor mounted hopper with load-cell digital read out, fed from a front end loader. The raw materials are weighed in the proportions determined by the formulation. Batches are then conveyed to the mixing unit.

Larger units are normally fed from overhead holding hoppers.

4.1.1.2 Continuous Weighing

These systems provide a continuous feed to the mixer by belt conveyors and may be continuous belt weighers or constant rate feeders. In the former the raw materials are fed onto fixed speed extractor weighbelts fitted with load-cells. Variations in weight are transmitted to the control unit which adjusts the hopper gate opening. Constant rate feeders have a fixed hopper gate opening and the belt speed is continuously adjusted to the predetermined rate of output. A load cell continuously monitors the amount of material on the extractor conveyor and sends signals to the motor speed control unit.

In both systems, information from the individual weight or speed controllers is fed to a central control unit which can be pre-set for the required output.

4.1.2 Volumetric Feeding

For some blending operations it is sufficient to use volumetric measuring of the raw materials to obtain the correct proportions in the blend. A separate hopper, usually fed by front end loader, must be provided for each raw material. All the components are handled simultaneously and the units are controlled to give the required blend ratio.

This system however, relies on consistent raw material densities and many uncontrollable variables may cause changes in the bulk density. One of these factors will be the particle sizes of the granules and these may vary throughout a storage heap.

4.1.3 Mixers

The blending equipment is a major determining factor in the production of good quality blends.

Different designs of mixers are available such as rotating drums, cone ended tilting mixers, stationary mixers with internal paddles, vertical mixers and volumetric mixers with variable speed screws. In addition, blending may be carried out without the use of a specific mixer. In this system all raw materials are fed onto a collector belt conveyor and, as each material is constantly fed at the correct weight, all materials are constantly layered onto the belt in the correct formulation ratio. Thus a cross section of that belt will at any time have the correct nutrient content. Subsequent mixing at belt changeovers as the blend moves through the remainder of the plant ensures a correct mix at all times.

The choice of design will depend on individual circumstances and is outside the scope of this handbook. However, one aspect of mixing which is of importance is the mixing time. Mixing times are normally around two minutes and times above 5 minutes are not recommended because of the risk of particle breakdown and deterioration of the mixing quality.

After installation, the mixer must be tested to determine that satisfactory mixing is achieved. It is recommended that a coefficient of variation of less than 10% is achieved. Descriptions of mixing tests can be found in references [1] and [11].

4.1.4 Loading Equipment

At all stages after the mixing, great care must be taken to avoid segregation of the blend components. This means that coning must be avoided at all stages.

The final section of the blending plant may include receiving hoppers for bulk loading or bagging but in the simplest case the blended material may be loaded directly onto trucks from a conveyor system.

Further information on equipment may be obtained from manufacturers or by reference to the literature [16], [18], [19], [27] and [29].

4.2 TESTING/CALIBRATION

As a minimum control, records must be kept of all formulations, batch records, weight or volume settings and raw material analyses and sources. In addition the following are to be recommended:

1 Regular checks of weighing or volumetric measuring equipment (minimum frequency, once per month)

2 Frequent check weighing for bagged material

3 Random checks of the chemical analysis of finished products (**minimum frequency, once a week**)

Further information is given in Chapters 7 and 8

4.3 OPERATIONS

Full written operating instructions must be available for all personnel. Operating procedures should be well documented and reviewed at regular intervals. Accreditation to ISO 9000 is not essential but is recommended as it offers an independent assessment of all procedures.

5. SEGREGATION

Segregation is the separation of particles due to differences in physical characteristics. For segregation to take place there must be movement between the particles.

Various characteristics may cause segregation but the most common are **particle size, particle density and particle shape**. The size is certainly by far the most important factor to explain segregation during flow. During spreading, the three properties play a role, the shape being the least important and the size again having a dominating effect.

From the above it is seen that segregation may occur during manufacture, bulk transport (and handling) or during application to the soil or crops. The effects as seen by the crops may be the same in all cases unless remixing takes place during later handling stages.

The following extract from Stairmand [26] deals with the problems of storage in hoppers and this is relevant to bulk handling, bagging operations and spreading.

"The two main factors are avoidance of segregation in filling and emptying and ensuring the hopper will be "self-clearing." In examining the question of segregation in filling… if the material is charged into the hopper from a single point, the coarser particles will migrate to the outer edges and a central core of material containing an excess of fines will form. If now filling is discontinued and the hopper is allowed to discharge it will do so in zones… If the zones contain particles of different sizes due to segregation in filling, no device fitted at the cone discharge to remix can possibly be effective. If however, the hopper is filled without segregation, little segregation will occur when emptying."

By applying the above to blending, it can be seen that the avoidance of segregation and the subsequent handling of the product, is the most important part of any blending process. This may be achieved in three ways:

1 The careful matching of the blend components

2 The design of hoppers (see Leonard [19])

3 The loading methods of the hoppers and vehicles, etc.

The origin of the segregation is always a difference of physical properties leading to a physical separation of the particles which may or may not result in a chemical segregation. Size segregation may also occur within raw materials or complex fertilisers but this will have no (or very little) effect on the chemical content. However, in bulk blends, physical segregation often leads to chemical differences.

5.1 FLOW SEGREGATION

Care must be taken when storing any material to avoid size segregation in the storage heaps. Whenever granular material falls freely to form a cone or part cone, the larger particles will tend to run down the outside with the smaller particles remaining near the centre of the cone. Should this happen, there may be quite large variations in size characteristics between various parts of a heap and this could have serious effects on all fertilisers (Figure 3). Batches taken from a segregated heap can

thus differ in particle sizes, and this will affect the spreading width of a centrifugal spreader with straight, blended and complex fertilisers.

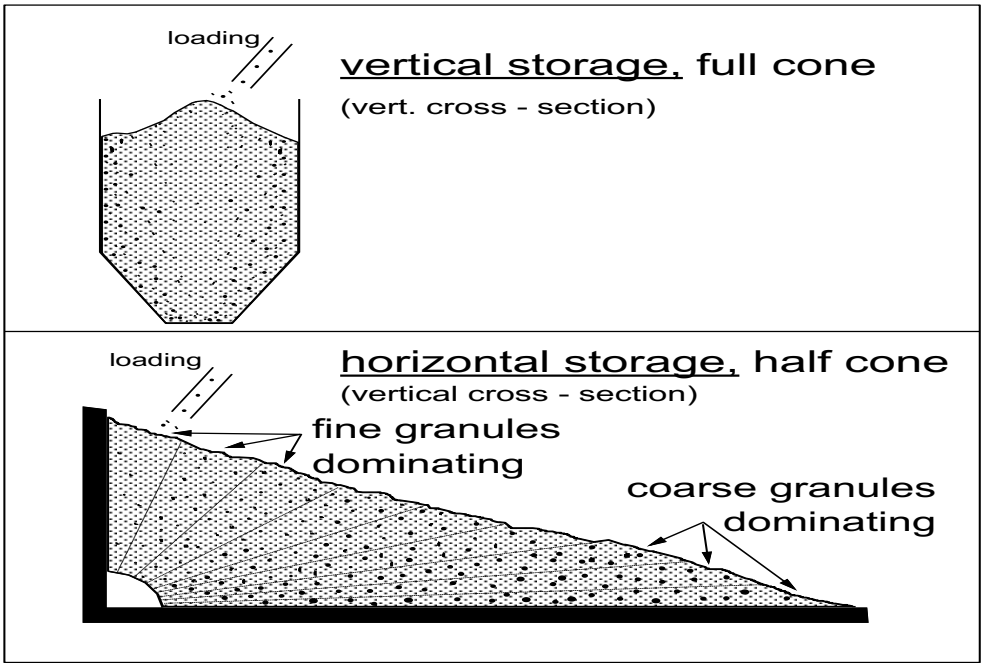


Figure 3. Illustration of flow segregation when coning occurs during loading [15]

There may be some remixing at later handling stages. Such remixing may be caused simply by tipping a bag of fertiliser out into a hopper or even more simply by turning a loosely filled bag over. Remixing may also occur during loading and unloading of bulk fertiliser (Figure 4). However, systematic remixing can rarely be counted on.

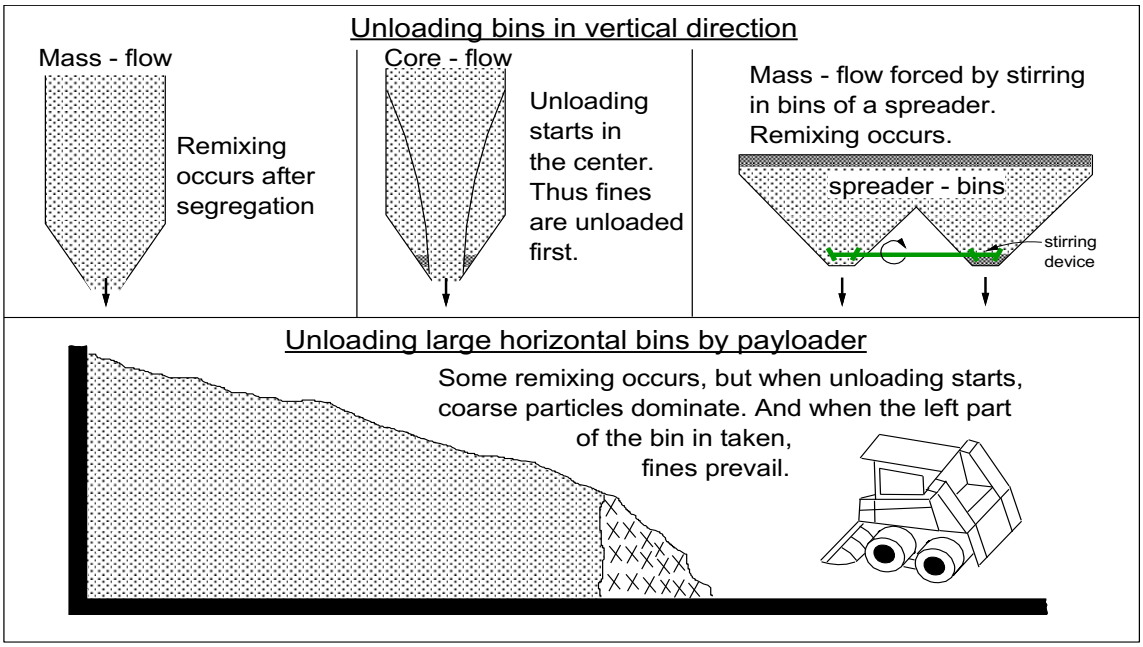


Figure 4. Remixing of segregated fertiliser when unloading bins [15]

It is essential that all handling equipment is constructed to minimise segregation. The use of specially designed chutes is recommended to avoid segregation and excessive amounts of free dust.

Examples of techniques which may be used to avoid segregation include (Figure 5):

"Egg crate" baffles in square or rectangular hoppers,

Concentric cone distributors for cylindrical hoppers,

Flexible spouts for direct loading equipment.

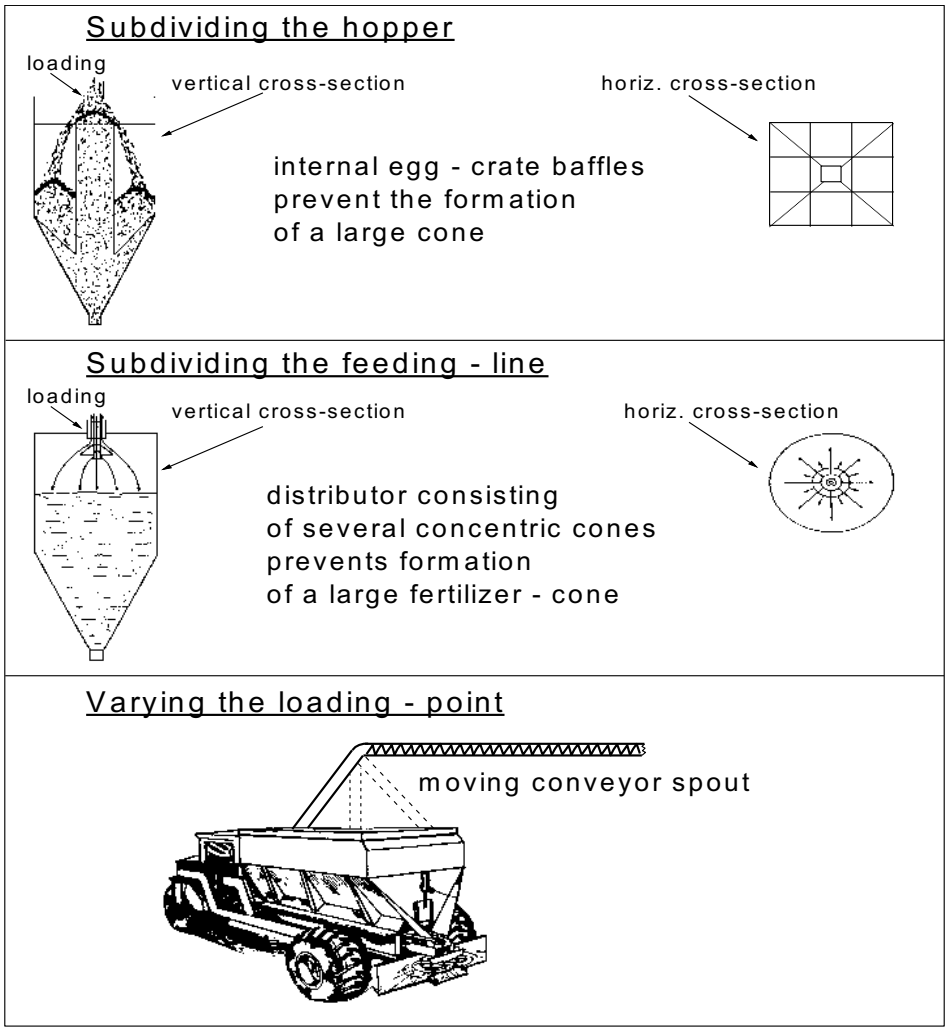


Figure 5 – Prevention of segregation in hoppers [18]

In the case of raw materials, the segregation is mainly a size segregation which has little effect on the chemical content. For blends, the risk is to have both size and chemical segregation.

Tests of filling and emptying containers have shown that granulometric segregation is well linked with the granulometric spread index (Figure 6). It is recommended that the granulometric spread index

of the blend is limited to below 20. This means that the raw materials must have an even lower GSI (see Table 2 page 30)

The figures below distinguish three ranges of segregation: **Low** segregation means that for the given difference of property the final segregation has little impact – **Medium** is the situation where the final segregation begins to be significant but if there is another unfavourable condition, for example low overlapping during spreading, the final result may be bad – **High** is the worst situation where there are negative economic and/or environmental consequences.

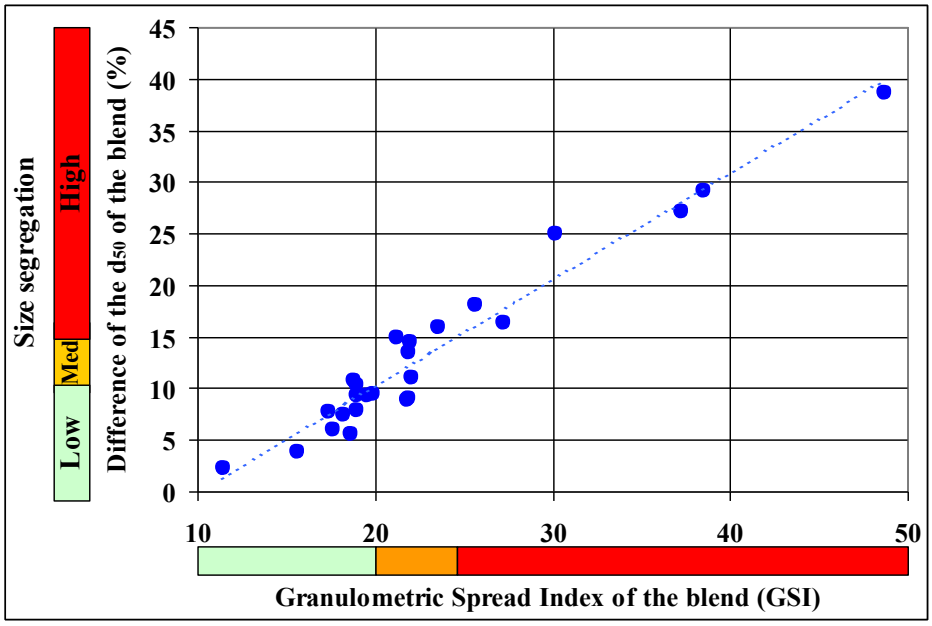


Figure 6: Relation between size segregation and GSI of bulk blends when emptying a container (segregation is the difference of d_{50} between the first and the last third of emptying a container) [20]

Chemical segregation is related to the absolute sum of the difference of d_{16} and d_{84} of each component (Figure 7). This means that if the fine particles come from one raw material and the coarse particles from another, the size segregation induces a chemical segregation.

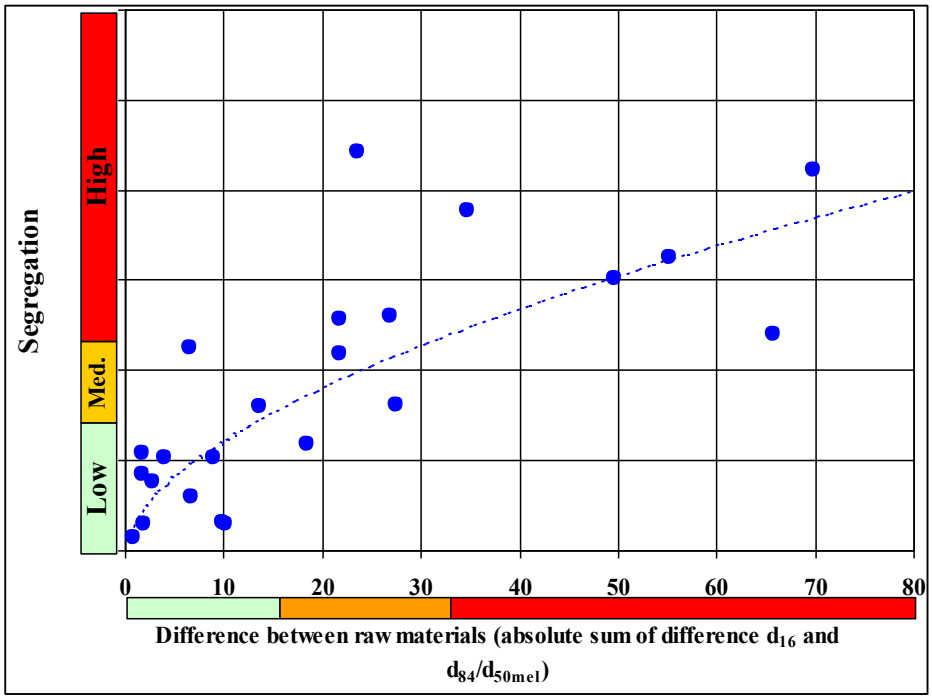


Figure 7: Chemical segregation in relation with the absolute sum of the differences of d_{16} and d_{84} of the components. [20]

5.2 TRANSPORT SEGREGATION

It is often said that blends are subjected to serious segregation during vehicle transport but this is not true. The level of vibration in transport vehicles is not high enough to induce a movement of the fertiliser. In fact, the segregation occurs when filling (and emptying) the container as described above.

During transport, only percolation of very small grains between the larger ones can occur. With common fertilisers, this phenomenon appears if there are particles smaller than 0,5 mm. Above this size, the process is too slow to have a real impact on the final product.

5.3 SPREADING SEGREGATION

Blenders should have some knowledge of the kinds of spreading equipment available and the advantages and disadvantages of these when using blended fertilisers. Blenders should be prepared and able to advise farmers on the type of equipment to use and the setting up, calibration and maintenance necessary. This means that they should be prepared to give as much information as possible about the physical quality of their products. Such information may include the composition, bulk density and flow characteristics as well as the particle size distribution. This is another reason for keeping adequate records of raw materials and product batches.

Centrifugal, pneumatic, and auger spreaders are available. The segregation induced by physical differences of the fertiliser particles may be due to trickling, as is the case with auger spreaders, or may be caused by the aerodynamic resistance of the particles when thrown through the air, as with centrifugal and pneumatic spreaders.

However, any segregation during spreading may be completely compensated for by the overlapping of adjacent spreading swaths. This leads to the question, how much overlapping?

As a general guide, pneumatic spreaders will not induce segregation because of the large amount of overlapping (from the spouts). The effects of segregation from centrifugal spreaders will be reduced by overlapping.

Auger spreaders will segregate heavily. Since these spreaders, when correctly adjusted have a rectangular lateral mass distribution, no overlapping occurs and thus also no reduction of segregation takes place. For this reason, auger spreaders are not recommended for blended fertilisers. Further information may be found in reference [2].

In Western Europe, the most common devices are centrifugal spreaders with two discs. Their **working widths** (width between tractor tracks) vary between 12 and more than 36 m. The **spreading width** can reach more than 50 m.

Segregation occurs during spreading due to physical differences between the components of the blended fertiliser. The larger, heavier and more spherical the particles are, the further they are spread. The size and density are the major factors (Figure 8, Figure 9 and Figure 10).

The charts show the segregation as a function of the difference of a given physical property (size, density or shape). The considered blends have two components in a proportion of 50% and these are physically similar except for the analysed property.

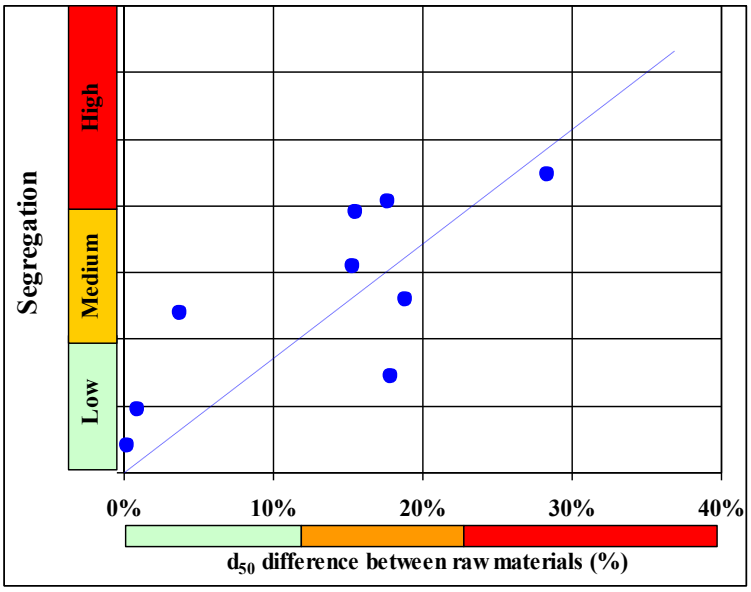


Figure 8: Evolution of the segregation with the difference of d_{50} between the two components [20]

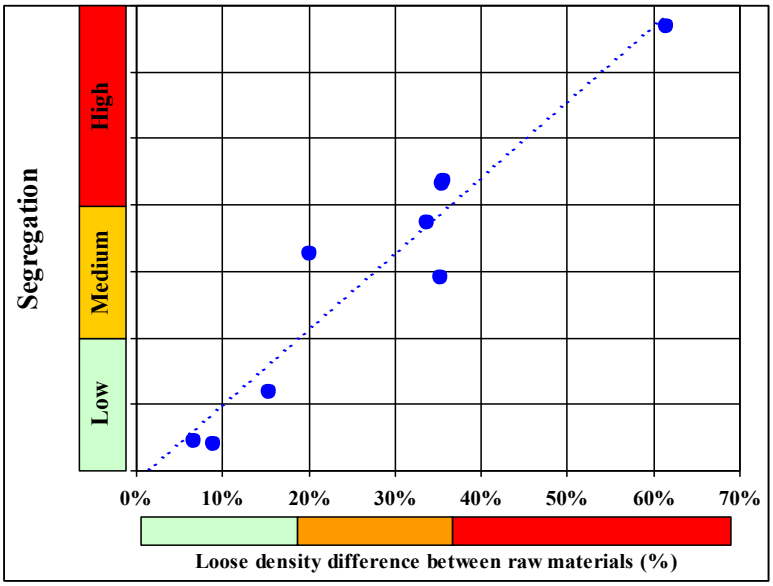


Figure 9: Evolution of the segregation with the difference of density between the two components. [20]

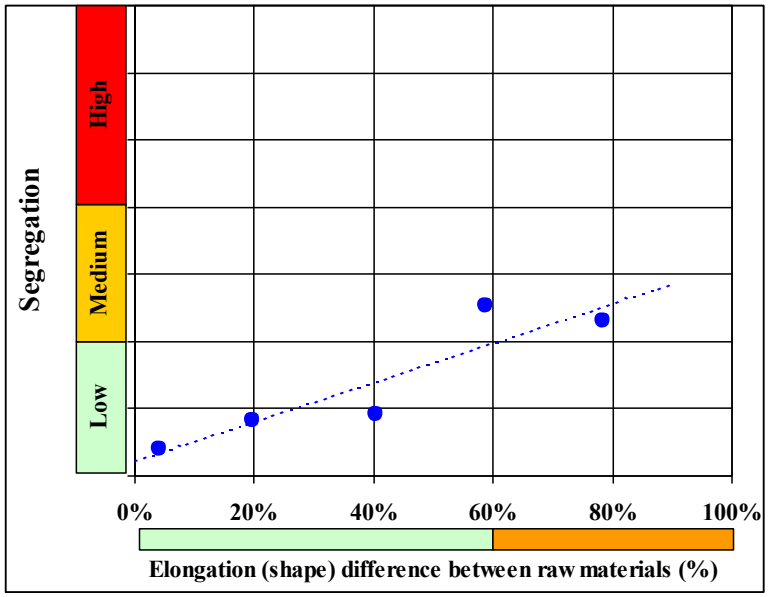


Figure 10: Evolution of the segregation with the difference of shape between the two components. [20]

In practice, the components present differences for more than one physical property and each difference can accentuate or reduce the final segregation. For example, size difference between components can compensate for a density difference and vice versa. The larger, heavier and more spherical the particles are, the further they are spread. **For example, urea which has a low density in comparison with other fertilisers should ideally have large sized particles with a d_{50} near the maximum tolerance in order to limit spreading segregation**.

The final impact of segregation during spreading can be significantly reduced by appropriate overlapping between the passes. Indeed, the segregation is generally marked in such a way that one component tends to accumulate behind the spreader and another tends to be spread further. If the overlapping between the passes is sufficient, at each place in the field the final quantity of fertiliser comes from two passes (Figure 11, top and centre). With a good spreader correctly adjusted, it is even possible for working widths up to 24 or 28 m. However, for badly adjusted spreaders the segregation remains. Practically, a correct overlapping means that the spreading width must be about double the working width. So with a working width of 24 m, it means that the spreading width must approach 48 m. Figure 11, below, shows an example of insufficient overlapping, resulting from a working width which is much greater than half the spreading width.

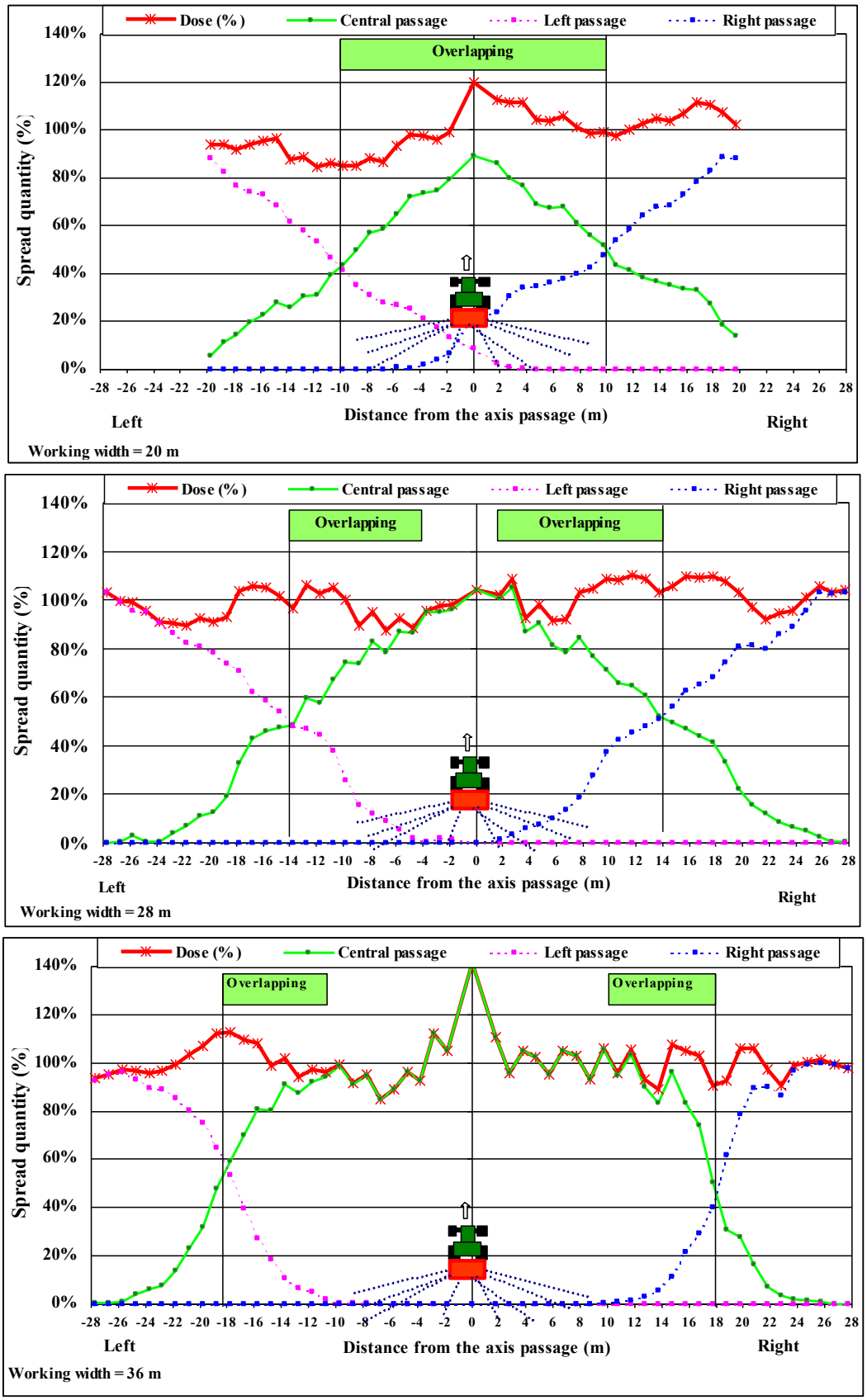


Figure 11: Illustration of the overlapping for the same bulk blend but for working widths of 20, 28 and 36 m. [21]

Another important point to mention is that with higher working widths, it becomes difficult to find the correct adjustment. This is not only the case with blends but also with straight or complex fertilisers. The use of a control kit is certainly recommended for the farmer (Figure 12 below).

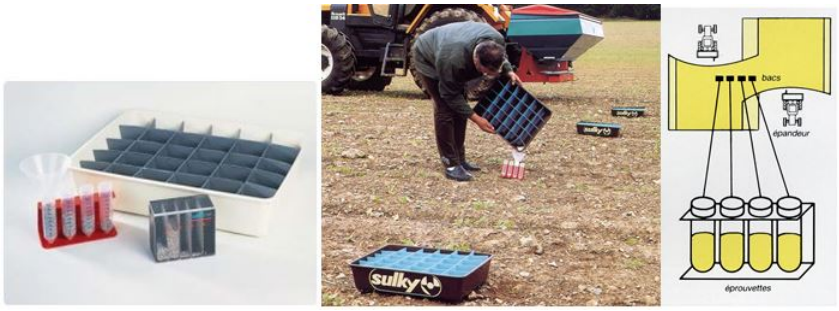


Figure 12 : Illustration of a kit to measure spreading quality

Good advice for farmers is to **limit the working width in order to obtain an important overlapping**. In practice this means choosing a working width lower than 24 or 28 m with a high performance spreader.

5.4 EFBA QUALITY RECOMMENDATIONS

To reduce segregation problems, it is recommended to blend raw materials **having similar physical properties**, the most important being the size of the particles. The European Fertilizer Blenders Association (EFBA) has defined quality recommendations for the particle sizes (Table 2). The recommendations concentrate on a d_{50} around 3,25 mm with a limited granulometric spread index (GSI). If the raw materials comply with these recommendations, **flow segregation is not significant and spreading segregation only occurs if there are important density and/or shape differences**. This happens only when certain specific fertilisers are used.

Table 2 - Targets and tolerances for the granulation of blend components.

EFBA Targets and Tolerances for the granulation of raw materials for fertiliser blending			
Verbal notation	Physical dimension		
Mean Particle Size	d_{50} in mm	3,25 mm	±0,25 mm
Fine Particles	< 1 mm, % of mass	0%	0,25%
Coarse Particles	> 5 mm, % of mass	0%	1%
Main Range	2,5 – 4,0 mm, % of mass	90%	±5 %
Granulometric Spread Index (GSI)	$GSI = \frac{d_{84} - d_{16}}{2\ \mathrm{d}_{50}}\ 100$	<18	

It is assumed that the sieve analysis is carried out according to European Standard EN 1235/A1.
EFBA recommends the use of these seven sieves : 1,00 mm – 2,50 mm – 2,80 mm – 3,15 mm – 3,55 mm – 4,00 mm – 5,00 mm (the justification for these sieves is linked to the recommendations)

6. SAFETY CLASSIFICATION – LABELLING - DISTRIBUTION

Blenders must be aware of the hazards associated with raw materials and fertilisers, especially those containing ammonium nitrate. Recognition of the hazards is made simpler by classification systems such as those prepared by the United Nations (UN), International Maritime Organisation (IMO) and the European Commission (EC). All products must be labelled according to National and European Regulations and in some circumstances only packaged fertilisers may be supplied to the end user.

Full account should be taken of all industry guidance and Codes of Practice such as those prepared by Fertilizers Europe [12, 13]

6.1 – Labelling and marketing of fertilisers

Any fertilisers placed on the European or national market must be correctly labelled in accordance with either national or EC regulations, dependant on the market in which it is to be sold. These regulations normally cover matters such as nutrient content, safety and protection of the environment.

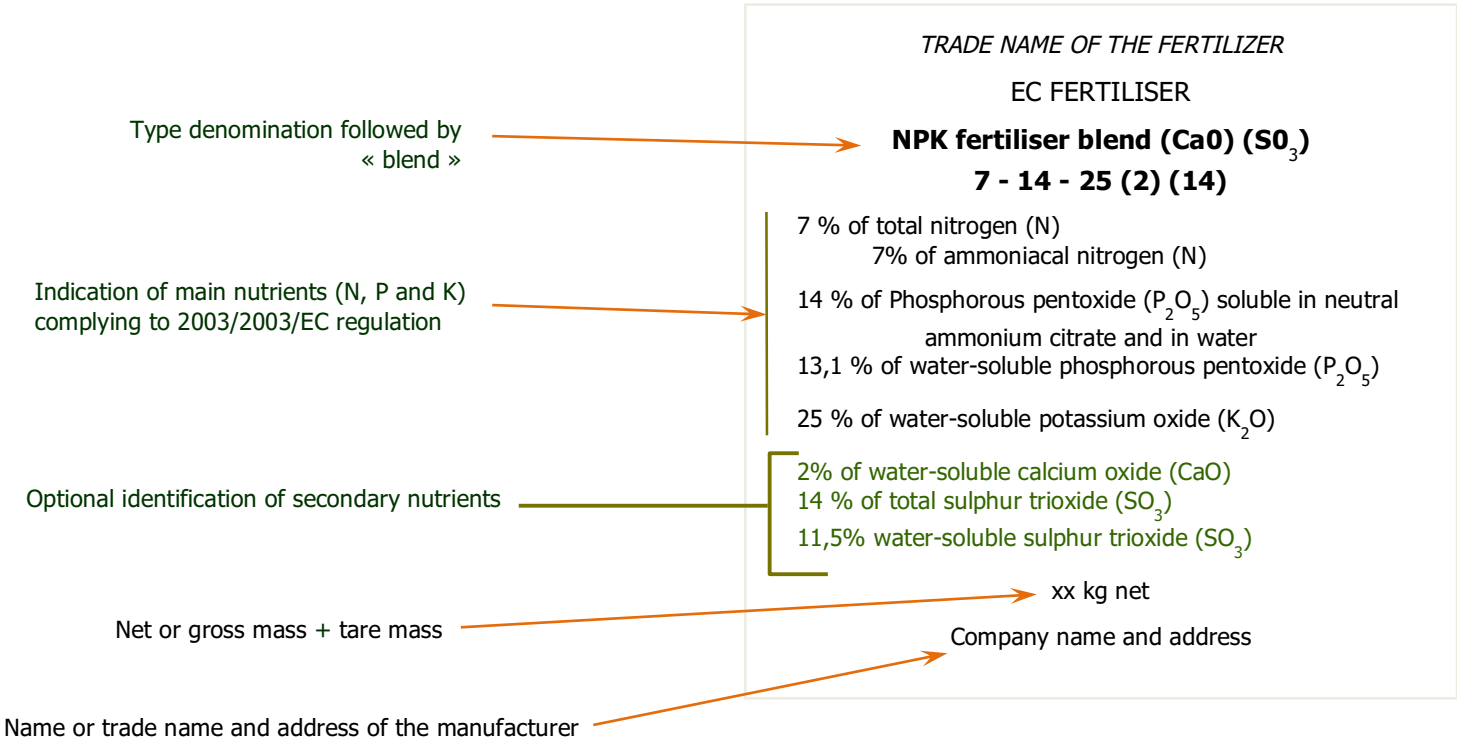
Mineral fertilisers, offered for sale on the European market, need to comply with European Council Regulation 2003/2003/EC [25].

For packed blended fertilisers, the labelling information must be placed on the packaging in a conspicuous position. Labels must be attached to the package or to whatever system is used for closing it. Markings must be, and must remain, indelible and clearly legible (2003/2003/EC, Art. 10).

For bulk blended fertilisers, these markings may appear on the accompanying documents (2003/2003/EC, Art. 7).

Markings must appear in at least the national language or languages of the Member State in which the EC fertiliser is marketed.

Figure 13 : Example of EC label for a solid mineral fertiliser according to EC 2003/2003.



6.2 – Labelling and classification of hazardous substances and mixtures (CLP regulation) : hazard labelling

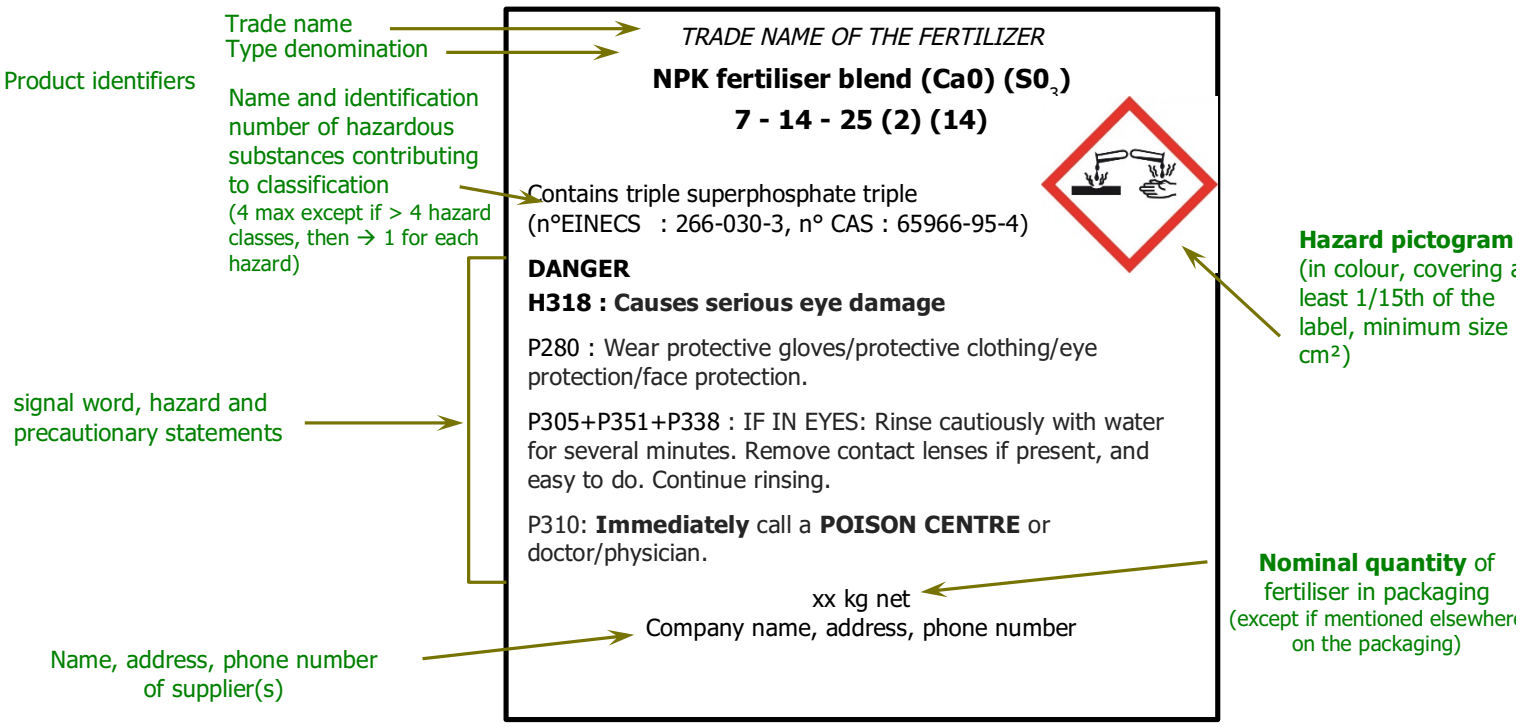
European Regulation 1272/2008/EC concerns the classification, labelling and packaging of substances and mixtures [23]. It is known by its abbreviated form, 'the CLP Regulation' or just plain 'CLP'.

The CLP Regulation adopts the United Nations' Globally Harmonised System on the classification and labelling of chemicals (GHS) across all European Union countries.

Before manufacturing, each blended fertiliser containing hazardous substance from raw material must be classified according to the CLP regulations.

The classification of the blended fertilisers depends on the concentration of hazardous substances in the final product. All classified fertilisers require hazard labelling.

Figure 14: Example of hazard labelling according to CLP regulation for a fertiliser containing over 3% of triple superphosphate.



The size of the label is also defined according to packaging volume :

Packaging volume	Label size (mmm)	Pictogram size (mm)
> 500 liters (Big Bag)	Mini 148 x 210	Mini 46 x 46
> 50 liters and ≤ 500 liters	Mini 105 x 148	Mini 32 x 32
> 3 liters and ≤ 50 liters	Mini 74 x 105	Mini 23 x 23
≤ 3 liters	Mini 52 x 74 if possible	Mini 10 x 10 (16 x 16 if possible)

The label of a fertiliser which does not meet the criteria for classification as hazardous but contains a hazardous substance in a concentration level with safety data sheet available on demand shall bear the statement : EUH210 — 'Safety data sheet available on request'.

For ammonium nitrate, the Oxidizing pictogram from CLP can be replaced by the ADR pictogram (5.1 class) on the label but H272 hazard statement shall remain.

6.3 – Regulation related to hazardous substances and mixtures (REACH regulation) : Safety data sheet (SDS)

REACH Regulation 1907/2006/EC on Registration, Evaluation, Authorisation & Restrictions of Chemicals) defines Safety Data Sheet as the main information vector on product hazards and good practices (Art. 31) [24].

A Safety Data Sheet (SDS) is mandatory and is to be pro-actively provided to the client if the final product is classified as hazardous in accordance with CLP Regulation.

A SDS is to be provided to the recipient at his request when the fertiliser does not meet the criteria for classification as hazardous in accordance with CLP, but contains:

- 1 % or more of a substance classified as hazardous
- 0.1% or more of a substance classified as persistent, bioaccumulative and toxic or very persistent and very bioaccumulative which is on the list of substances submitted to authorisation (REACH)
- a substance for which there are Community workplace exposure limits.

Any SDS shall be provided free of charge.

For fertilisers classified as hazardous, the SDS shall to be provided on paper or electronically no later than the date on which the fertiliser is first supplied (REACH Art. 31-8). There is no need to provide it at each delivery.

Updates of SDS shall be provided to all former recipients to whom fertiliser has been supplied within the preceding 12 months (REACH Art.31-9)

SDS shall be provided in the language of the country where the product is sold.

SDS shall follow the product from its manufacture to it's end use :

Distribution → forwarding of supplier's SDS to the client.

Packaging → Transmission of a SDS in the name the company responsible for placing the fertiliser on the market.

Blending → Transmssion of a SDS specific to the blended fertiliser in the name the company responsible for placing the fertiliser on the market.

The safety data sheet shall be dated and shall contain the following headings:

1. identification of the substance/mixture and of the company/undertaking;
2. hazards identification;
3. composition/information on ingredients;
4. first-aid measures;
5. fire-fighting measures;

6. accidental release measures;
7. handling and storage;
8. exposure controls/personal protection;
9. physical and chemical properties;
10. stability and reactivity;
11. toxicological information;
12. ecological information;
13. disposal considerations;
14. transport information;
15. regulatory information;
16. other information.

A set of Standardised Safety Data sheets for the most common fertilisers are available on request.

Key steps according to hazardous substances regulation :

⇨ Check if presence of hazardous substances in raw materials : superphosphates, ammonium nitrate…
⇨ Classify the new fertiliser in accordance to hazardous substances regulation (CLP)
⇨ Label the new fertiliser in accordance with hazardous substances regulation (CLP) when needed
⇨ Create the Safety Data Sheet in accordance with REACH if needed

7. QUALITY CONTROL

Blenders should draw up a routine quality control schedule to include the sampling and analysis of raw materials and products as well as checks on raw material weighers and/or feeders and check weighing of finished bagged products.

7.1 RAW MATERIALS

The amount of quality control of raw materials depends on the reliability of the suppliers. If supplies are received from single sources, experience will soon establish the degree of control needed. If raw materials are purchased from a variety of sources, extra control is essential.

Random samples from each consignment should be taken as described in Chapter 8, for reference purposes. These samples can be sent for analysis whenever there is reason for doubt but they should be carefully labelled with date and origin and kept for at least three months.

As mentioned in section 3.2, the **particle size distribution of the raw materials is the most important property to be controlled**. Representative samples of raw material feeds should be taken and tested as detailed in Chapter 8 at regular intervals with a minimum frequency of once per shift. A full sieve analysis should be carried out on each raw material.

7.2 FINISHED PRODUCTS

All blenders have an obligation to meet the requirements of their National Fertiliser Regulations and for those fertilisers which are marketed as EC fertilisers, common Regulation EC 2003/2003 [25] applies throughout the Community. National Regulations will cover products which are not declared as EC fertilisers. It should be noted that in cases where the farmer does not purchase a fertiliser with a specified nutrient ratio, for example, if he purchases his own raw materials and contracts the blender to mix them for him, all official controls for the blended fertiliser may not apply.

The regulations set out the tolerances on the declared **chemical analysis** for all fertilisers. Fertilisers not meeting these tolerances may result in prosecution and penalty. It is therefore recommended that some degree of quality control is exercised by blenders (auto-control).

The amount of quality control required will depend very much on the scale of the blending operation. For simple small scale batch blending operations it may be sufficient to rely on the principle that “what goes in must come out”. However, even these operations will need some regular control of the weighing equipment.

For large scale continuous operations, consideration should be given to full automated on-line sampling and analysis.

In between these two extremes, it is recommended that one representative random sample is taken from one of the grades made each day. If laboratory facilities are available on site, these samples should be analysed daily. If outside contract laboratory services are used, the frequency of analysis should be **at least one sample per week**, selected at random from the week’s collection.

Records should be kept of all samples taken and analyses carried out.

8. SAMPLING AND ANALYSIS

8.1 SAMPLING

Representative sampling of any material requires special techniques and equipment. Full details are outside the scope of this handbook but it is recommended that blenders follow recognised sampling methods such as those set out in European Standard EN 1482 Part 1 [9].

It is essential that all samples are correctly taken to ensure their representativity. There is no point in taking and analysing unrepresentative samples. The quality of the measurements made on the samples depends on their representativity. This is particularly important for blended fertilisers where the risk of heterogeneity is higher.

As stipulated by CEN, **blended samples must always be taken from material in motion**, either in free fall or on conveyor systems. It is essential to sample **through the whole stream** rather than from the same part of the stream. Suitable automated equipment is available for most circumstances and is to be recommended whenever possible.

For the blend producer it is important to know the sampling and measurement methods and the necessary equipment. Generally, all of this is described in standards which are regularly updated. Blenders are recommended to study the latest versions of sampling and measurement standards.

When a sample of any granular matter must be taken, some questions should be asked:

1 What quantity must I take?

2 How many increments to obtain this quantity?

3 How to take the samples?

Finally the sample must be reduced in order to obtain a final sample for analysis (usually about 250 g for sieving test and 500 g for a chemical analysis). Thus after the sampling there is also a reduction step. Full details of reduction methods are given in EN 1482 Part 2.

8.1.1 Sampling Quantity

The minimum quantity of sample recommended is given in EN 1482 Part 1. The recommendations are summarised as follows:

Table 4: Recommended quantity to sample for bagged products

Number of bags	Minimal number of increments
< 5 bags	A sample from each bag
4 < number of bags < 11	4 bags
10 < number of bags < 401	Whole number above the square root of the number of bags
> 400 bags	20 bags

If the weight of a bag is less than 5 kg, a complete bag is considered as a sub-sample. The number of selected bags can be higher if the final quantity of the sample is not sufficient. For bags of 50 kg, the content must ideally be divided in order to obtain a representative sample (see paragraph 8.1.3). Sampling the contents of an IBC (big bag) is not easy. The best solution is to sample during emptying, but this is not always possible. Further information is given in EN 1482 Part 1.

Table 5: Number of sampling units from which incremental samples are to be taken from a bulk lot

Lot Size	Minimum number of sampling units
25 t or less	10
More than 25 t and less than 401 t	The nearest whole number above the square root of 4 times the number of tonnes present
More than 400 t	40

The data from this table can be expressed in a more practical way (Table 6).

Table 6: Number of increments by vehicle to sample for bulk products.

	Number of increments to be taken during emptying of each delivery truck or wagon		
Lot Size	**Truck**	**Wagon 25 t**	**Wagon 60 t**
25 t	10	10	
50 t	7		15
75 t	6		
100 t	5		
150 t	4		
200 t	4		
300 t	3		
500 t	2		
600 t	2	2	4
1000 t	1	1	3
1250 t	1	1	1
2500 t	½	½	1

The amount of fertiliser for an increment is about 250 g for raw materials and 500 g for blends.

8.1.2 Sampling Equipment

Appropriate sampling equipment is essential. There are various automated systems for use with conveyor belts. These are certainly recommended if there are large amounts of fertiliser handled. For smaller deliveries, manual sampling is more suitable. A suitable type of sampling cup is described in EN 1482 Part 1 [9] and is essential for sampling from falling streams of materials (Figure 15). Information about the number of increments to be taken must be given to the operator (Table 6).

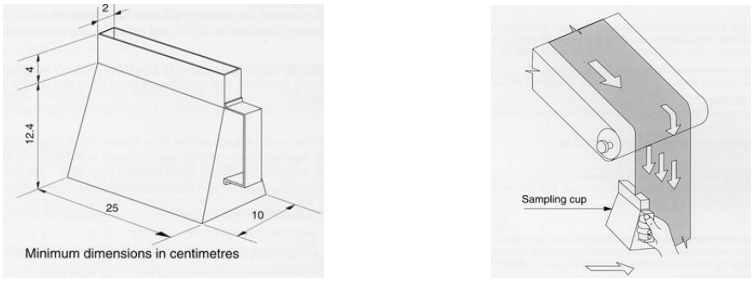


Figure 15: Example of a sampling cup and its use.

8.1.3 Sample Divider

Rotary sample dividers are recommended, particularly when sieve analysis is to be carried out or when blends are being tested. Riffle dividers may be used for raw materials but are less suitable for blended products. Descriptions of these dividers (Figure 16) can be found in EN 1482 [9]. It is important to remember that for the measurement of the physical properties, the particles must not be crushed, but for chemical analysis, it is recommended to crush the particles before the final reduction of the sample.

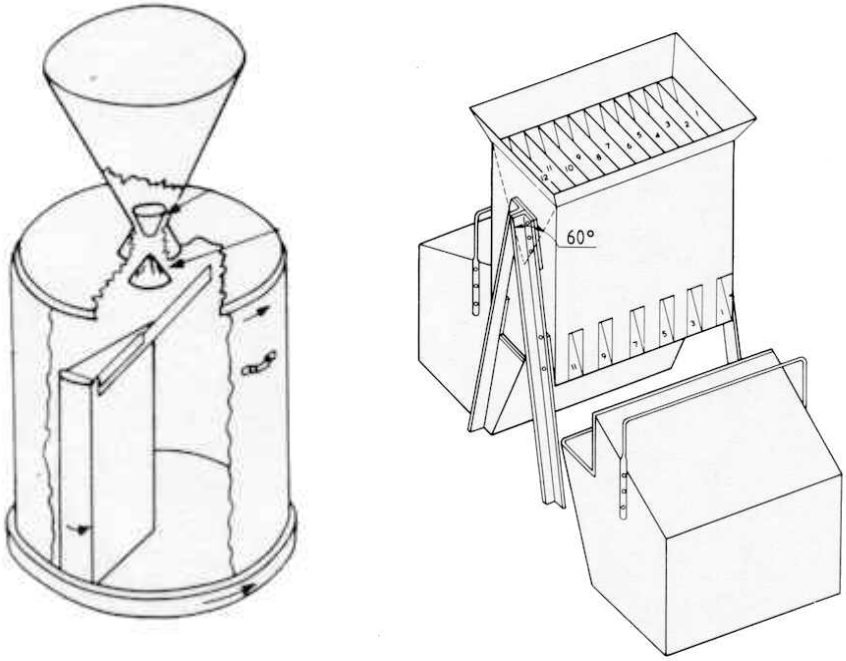


Figure 16: Illustration of rotary or riffle dividers.

8.2 CHEMICAL ANALYSIS

Standard reference test methods for most of the common chemical analyses used for fertiliser materials may be found in EC Directives, National Standards, European and International Standards and the AOAC Methods of Analysis Handbook. It is not essential that these methods are used for routine process control and simpler, often automated, methods are available. However, all such methods must be evaluated against one of the recognised standards.

8.3 PHYSICAL TESTS

Recognised standard test methods exist for several physical properties. The interest of blenders should focus on test sieving and bulk density (see section 3.2). Some laboratories have an accreditation for these special measurements. No Standard methods are available for such

properties as caking and free dust.

Sampling is also a fundamental step for physical testing because of the segregation that occurs naturally. This is particularly the case for sieve analysis because of the size segregation when there is a flow of fertiliser (which is always the case for bulk products).

Another point is that for physical testing, the quantity needed for the final sample may be more than 5 kg. This means that the number or the mass of sub-samples must be sufficient to achieve this.

8.3.1 Test Method for Sieve Analysis

(The following method is a summary of EN 1235/A1)

8.3.1.1 Principle:

Dry sieving of a sample of fertiliser material with one or more test sieves, using a mechanical sieving machine.

8.3.1.2 Apparatus:

- Balance, capable of weighing to the nearest 0,1g.
- Stainless steel woven wire test sieves, 200mm diameter, with a lid and receiver for the sieves.
- Mechanical shaker (sieving machine), capable of imparting both horizontal and vertical motion to the material on the set of sieves.
- Stopwatch
- Soft brush

8.3.1.3 Procedure:

Reduce the sample to approximately 250g, preferably using a rotary sample divider, or if one is not available, a riffle divider. Select seven sieves to cover the range of particle size expected and assemble in ascending order of aperture size on top of the receiver. (See note at the bottom of Table 2)

Weigh the test portion to the nearest 0,1g and place it on the top sieve and fit the lid. Place the set of sieves with the sample on the shaker and shake for 10 minutes.

Remove the sieves from the nest, starting from the top and weigh the quantity retained on each sieve and in the receiver, to the nearest 0,1g. Remove any particles trapped in the mesh by brushing from underneath.

Sum the masses of the fractions retained on the sieves and in the receiver and check that the total mass is within 2,5g of the original mass. Calculate each fraction mass as a percentage of the sum of the masses and draw up a table showing the cumulative percentage passing each sieve.

The percentage of material retained in the receiver (x_0) and on each sieve (x_n) is obtained from the formula :

$$X_n = \frac{m_n}{m_t} \times 100$$

Where m_n is the mass on sieve n

m_t is the total mass ($m_0 + m_1 + \ldots$)

X_n is the mass % retained on sieve n

The cumulative undersize is defined by the formula:

$$C_n = X_0 + X_1 + X_2 + \ldots + X_{n-1}$$

Where C_n is the cumulative % undersize for sieve n

8.3.2 Test Method for Loose Bulk Density

(The following method is a summary of EN1236)

8.3.2.1 Principle:

Weighing a known volume of the fertiliser.

8.3.2.2 Apparatus:

- Balance, capable of weighing to the nearest 1g.
- Cylinder of a known volume, *V (*approx. 1 litre and diameter around 60 mm).
- Standard funnel with an aperture of 25 mm diameter.

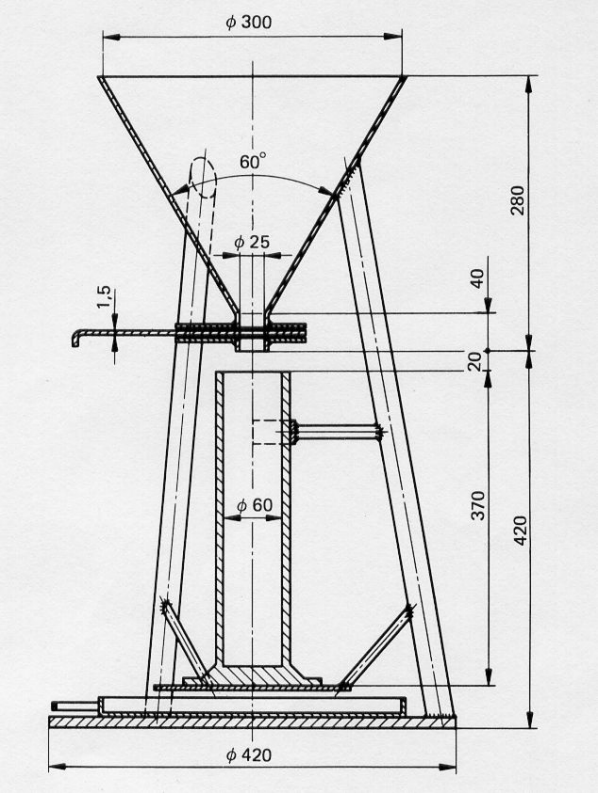


Figure 17: Illustration of the equipment for the loose density measurement.

8.3.2.3 Procedure:

Place the sample of fertiliser in the funnel with the aperture closed. Weigh the empty cylinder and place it under the funnel. Open the aperture of the funnel and allow the fertiliser to flow into the cylinder. When the cylinder is full, close the funnel and remove the excess fertiliser above the cylinder with a spatula. Weigh the cylinder and its contents and calculate the weight of fertiliser (*m* in kg).

The loose bulk density is given by the following formula:

$$\rho = \frac{\mathrm{m}}{\mathrm{V}}$$

8.3.3 Test Method for Angle of Repose

(The following method is a summary of EN 12047)

8.3.3.1 Principle:

Measurement of the diameter of a heap of a given height and calculation of the angle of the heap.

8.3.3.2 Apparatus:

- Funnel (aperture of 25 mm diameter) placed at 120 mm above a surface.
- Horizontal surface of 750 x 750 mm, with four lines presenting an angle of 45° between them and traced at the centre of the surface.

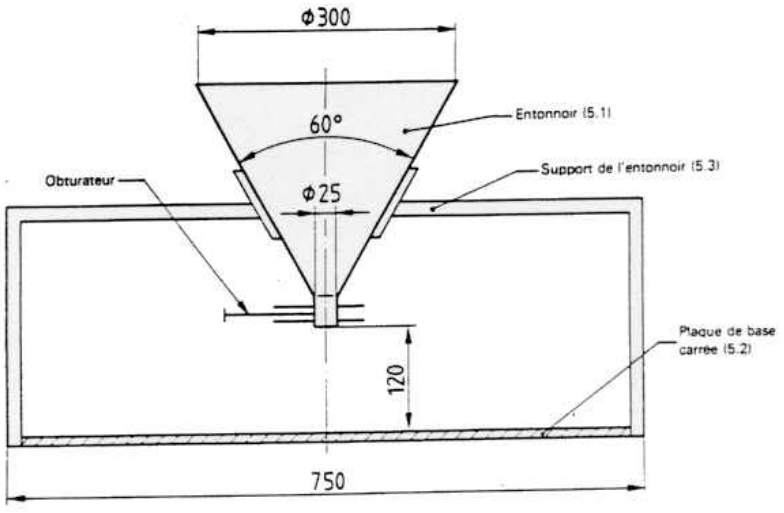


Figure 18: Illustration of the equipment for the angle of repose measurement.

8.3.3.3 Procedure:

Place about 5 kg fertiliser in the funnel with the aperture closed. Open the aperture and allow the fertiliser to flow freely onto the surface and form a heap. The flow stops when the heap reaches the bottom of the funnel. Measure the four diameters on the plate. Calculate the average diameter ($\overline{d}$ in mm). The value of the angle of repose is obtained by the following formula:

$$\alpha = \arctan\left(\frac{240}{\overline{d} - 25}\right)$$

8.4 EXAMPLES

This section gives some examples of the results of physical tests on fertilisers. The data used are realistic but the actual physical properties will vary considerably for the same product depending on its origin [14].

Using the measurement methods described in the previous section, the following results were obtained for three different fertilisers, A, B and C. (Table 7).

Table 7: Example of data from the laboratory for physical testing of three fertilisers.

	Fertiliser		
	A	**B**	**C**
Sieving test			
< 1,00 mm	0,1 g	0,5 g	8,2 g
1,00 to 2,50 mm	1,2 g	9,3 g	35,6 g
2,50 to 2,80 mm	5,3 g	19,9 g	45,2 g
2,80 to 3,15 mm	36,7 g	68,1 g	51,9 g
3,15 to 3,55 mm	115,2 g	79,6 g	46,8 g
3,55 to 4,00 mm	67,7 g	51,2 g	35,3 g
4,00 to 5,00 mm	15,9 g	13,2 g	21,7 g
> 5,00 mm	2,1 g	2,1 g	5,2 g
Total	244,2 g	243,9 g	249,9g

Loose bulk density			
Weight of 1 litre(V = 0,001 m³)	1,000 kg	0,750 kg	0,950 kg
Angle of repose			
Average diameter base of the heap ($\bar{d}$)	400 mm	400 mm	325 mm

By applying the formulae it is possible to determine the physical properties of these fertilisers.

Table 8: Example of calculation for the physical properties of fertiliser A.

Sieving Test	Weight	Percentage	Cumulative Percentage
< 1,00 mm	0,1 g	0,1 / 244,2 = 0,04%	0,04%
1,00 to 2,50 mm	1,2 g	1,2 / 244,2 = 0,49%	0,04 + 0,49 = 0,53%
2,50 to 2,80 mm	5,3 g	5,3 / 244,2 = 2,17%	2,17 + 0,53 = 2,70%
2,80 to 3,15 mm	36,7 g	36,7 / 244,2 = 15,03%	5,03 + 2,70% = 17,73%
3,15 to 3,55 mm	115,2 g	115,2 / 244,2 = 47,17%	47,17 + 17,73 = 64,91%
3,55 to 4,00 mm	67,7 g	27,72 / 244,2 = 27,72%	27,72 / 244,2 = 27,72%
4,00 to 5,00 mm	15,9 g	15,9 / 244,2 = 6,51%	6,51 + 92,63 = 99,14%
> 5,00 mm	2,1 g	2,1 / 244,2 = 0,86%	0,86 + 99,14 = 100,00%
TOTAL	**244,2g**		

With the sieving test data, it is possible to determine the mean particle size (d_{50}) and the Granulometric Spread Index (GSI). The d_{16} is just under 3,15 mm, the d_{50} is between 3,15 mm and 3,55 mm and the d_{84} is between 3,55 mm and 4,00 mm. The use of the equation (see paragraph 3.2.1) leads to the following results:

$$d_{16} = 2{,}80 + \frac{16 - 2{,}70}{17{,}73 - 2{,}70}(3{,}15 - 2{,}80) = 3{,}11 \text{ mm}$$

$$d_{50} = 3{,}15 + \frac{50 - 17{,}73}{64{,}91 - 17{,}73}(3{,}55 - 3{,}15) = 3{,}42 \text{ mm}$$

$$d_{84} = 3{,}55 + \frac{84 - 64{,}91}{92{,}63 - 64{,}91}(4{,}00 - 3{,}55) = 3{,}86 \text{ mm}$$

$$GSI = \frac{3{,}86 - 3{,}11}{2 \times 3{,}42} \times 100 = 10{,}96$$

The graphic representation of the cumulative percentage confirms the calculated values (Figure 19).

Figure 19: Example of granulometric curve and graphic determination of d_{16}, d_{50} and d_{84}.

The same method of calculation is used for the other fertilisers (Table 9).

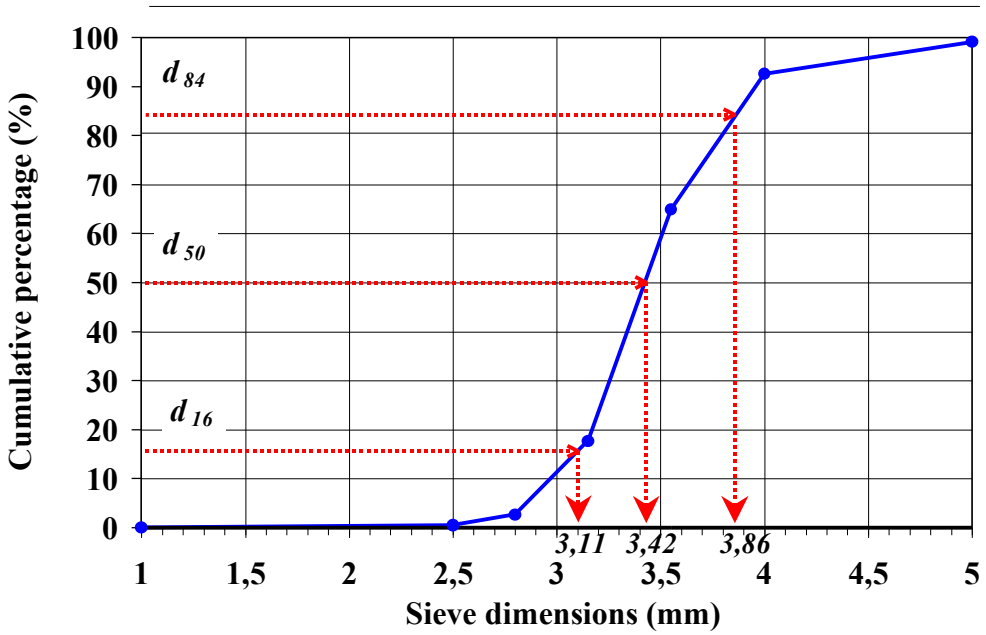


Table 9: Physical properties of fertilisers A, B and C.

	Fertiliser		
	A	**B**	**C**
d_{16}	3,11 mm	2,85 mm	2,34 mm
d_{50}	3,42 mm	3,27 mm	3,04 mm
d_{84}	3,86 mm	3,79 mm	3,83 mm
GSI	10,96	14,42	24,55
Loose bulk density (ρ)	1000 kg/m³	750 kg/m³	950 kg/m³
Angle of repose (α)	32,6°	32,6°	38,7

Comparing the fertilisers, the following comments may be made. The three products have a d_{50} in accordance with the recommendations (Table 2). Fertiliser A has a very low GSI meaning that all particles are of similar size. At the other extreme fertiliser C has a high GSI because of its high content of small particles. Fertiliser B has a low bulk density and fertiliser C has a high angle of repose because of its more angular particles.

9. FORMULATION

The calculation of blend formulations is not a difficult process but it is an essential requirement for producing good quality blends. The process very often is performed by a computer and combined with weigher or metering controls.

Records should be kept of each formulation and this can be simplified by the use of a standard format in the computer.

For accurate formulation the actual raw material analyses should be used but standard analyses are helpful as a first approximation.

Example :

Product required: 20 10 11

Raw materials available:	**N**	**P_2O_5**	**K_2O**
Calcium Ammonium Nitrate (27% Nitrogen)	27	0	0
Di ammonium Phosphate (18% Nitrogen 46% P_2O_5)	18	46	0
Potassium Chloride (60% K_2O)	0	0	60
Filler	0	0	0

1. To obtain 11% K_2O in the final product requires 18,5% Potassium Chloride.
2. To obtain 10% P_2O_5 in the final product requires 22% Di-ammonium Phosphate and this will provide 3,9% Nitrogen.
3. The remaining 16,1% Nitrogen in the final product will require 59,5% Calcium Ammonium Nitrate.
4. Total raw materials required adds up to 100% of the total space and thus no filler is required.

Formulation per tonne of product is thus:

- 595 kg of Calcium Ammonium Nitrate
- 220 kg of Di-ammonium Phosphate
- 185 kg of Potassium Chloride

NOTE:

Formulations containing less than 5% of any ingredient should be avoided as there will be difficulties in mixing this small amount evenly throughout the mixture.

10. REFERENCES

Copies of the listed Standards may be obtained from the National Standards Organisation (e.g. AFNOR in France, DIN in Germany and BSI in the UK).

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