

# Segregation of blended mineral fertilisers in conical piles and in different logistic systems

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The demand for physically mixed compound fertilisers has increased in recent years as a result of individually mixable formulations and lower production costs. For three fertiliser blends, three different components were used in each case. Each blend included different amounts of a 27% calcium ammonium nitrate (round granule or corn), an angular corn NPK complex fertiliser (15% N/15% P<sub>2</sub>O<sub>5</sub>/15% K<sub>2</sub>O/3% S) and a 60% potassium chloride (angular corn). The extent of segregation of the individual fertiliser components was tested in the conical pile formed after the mixing operation, in big bags and in unloaded piles from silo tanker truck and tipper truck respectively. Systematic sampling of the conical pile showed particularly with fertiliser blends 2 and 3 substantial deviations from the target nutrient mix. In the conical pile the target blend proportions were distorted through an increased proportion of K<sub>2</sub>O. The angular K<sub>2</sub>O corns predominated at the top of the conical pile while CAN round corns settled more at the base of the heap. This separation was reduced in the heap unloaded from the tipper truck while the pneumatic unloading from the tanker truck promoted separation. In the big bag, the nutrient distribution could vary significantly.

### Keywords

Mineral compound fertilisers, segregation, tipper truck, tanker truck, big bag

Bulk blends represent an alternative to traditional multi-nutrient fertilisers. The demand for physically mixed compound fertilisers rose in recent years as a result of individually mixable formulations and the lower production costs. In addition to the requirements for optimal adjustment of fertiliser amounts, application machinery and optimised application technique, the companies producing the fertilisers were also increasingly required to offer homogenic fertiliser mixes.

According to the European Fertiliser Directive (Regulation (EC) No. 2002/2003 from Oct. 2003 relating to fertilisers), compound fertilisers comprise at least two primary nutrients mixed chemically or physically. Blended or mixed fertiliser describes a dry mix of several nutrients in their individual granules. Contrary to this, "complex fertiliser" is produced through chemical reaction in solution or solid form among the components creating a homogenous product which in solid form each granule contains all (at least two primary nutrients) components in the declared composition. Specified tolerances (Annex II of the EC Fertiliser Regulation 2003/2003) for mineral compound fertiliser are 1.1% (absolute negative deviation) for the nutrients N,  $P_2O_5$  and  $K_2O$ .

Particularly with physically mixed fertilisers, good homogenisation must be guaranteed so that segregation of individual components during transport and application is prevented. All the more important is a good and optimal adjustment of the mixer in order to prevent physical segregation after the outlet pipe (HEHENBERGER 1993).

Segregation describes the separation of a blended fertiliser into its individual components and is caused by various influences within the logistic chain (European Fertilizer BLENDERS ASSOCIATION 2007, Figure 1). Two groups of influences working together the degree of segregation in mineral fertiliser blends: the material-dependent influences, e.g. range of corn sizes, corn density, corn form, water absorption potential, surface characteristics, etc., and on the other hand, process-related influences within the logistic chain such as mixing, unloading, conveying and distribution (HEHENBERGER 1993).



Figure 1: Influences on segregation of blended mineral fertilisers within the logistics chain

Process-related influences can only cause segregation where material related influences are present and vice versa. Segregation taking place at the beginning of the work chain or during the mixing process can, however, be partially reduced through mixing again during the application process but the original mix cannot be completely restored (HEHENBERGER 1993). Investigations by BALG ET AL. (1979), HEEGE and HELLWEG (1982) MAROUERING (2001), CEN (2014) have shown that the phenomenon of physical segregation occurs after material leaves the mixing plant discharge pipe and while a conical pile is being formed. This is mainly due to the material-related factors (Figure 2).



Figure 2: Segregation of calcium ammonium nitrate (CAN, 27 % N, round corn, blue coloured) and urea (46 % N, round corn, white) (MARQUERING 2001)

Particular importance can be attributed to the material and process related influences with regard to segregation. However, critical for determination and evaluation of the segregation is manner of sampling involved. A partial sampling of a conical pile of mineral fertiliser mix at the cone base can, based on the physical segregation phenomenon, lead to distortion of the nutrient mix in the analysis result.

Within a research project conducted in cooperation with an international mineral fertiliser producer (Borealis L.A.T. GmbH, Linz), three mineral fertiliser blends were tested for the occurrence of physical separation in the conical pile. The influence of three logistic systems (tanker truck, tipper truck and big bag) on the segregation, was also analysed.

## Material and method

Three mineral fertiliser blends (Table 1) were mixed in a stationary fertiliser mixing plant by the company Fuchshuber in Enns (Upper Austria). The plant used the sandwich process with mixing auger (Figure 3) for the procedure. As already mentioned, three components were used in the mix. Calcium ammonium nitrate, NPK complex and muriat of potash being the fertilisers most often applied. Calcium ammonium nitrate and NPK complex fertilisers are often mixed together. The muriat of potash was used because of its different corn form. The aim in using this is to find out how different corn forms influence segregation. The three individual fertilisers were used because they had the same density: calcium ammonium nitrate:1.9–2.0 g/cm<sup>3</sup>, NPK complex: 1.8–2.1 g/cm<sup>3</sup> and potassium chloride 1.9–2.0 g/cm<sup>3</sup>. Therefore, the influence of the density on segregation was minimised.

### Table 1: Composition of the three fertiliser blends

	Composition of the Mixtures	Chemical composition
Blend 1	50% CAN <sup>1)</sup> (round corn, granulated) 50% Complex <sup>2</sup> (round corn, granulated)	21,0% N 7,5% P <sub>2</sub> O <sub>5</sub> 7,5% K <sub>2</sub> O
Blend 2	33% CAN <sup>1)</sup> (round corn, granulated) 33% Complex <sup>2)</sup> (round corn, granulated) 34% MOP <sup>3</sup> (angular, compacted)	13,9 % N 5,0 % P <sub>2</sub> O <sub>5</sub> 25,4 % K <sub>2</sub> O
Blend 3	50% KAS <sup>1)</sup> (round corn, granulated) 50% MOP <sup>3)</sup> (angular, compacted)	13,0 % N 0,0 % P <sub>2</sub> O <sub>5</sub> 30,5 % K <sub>2</sub> O

<sup>1)</sup> CAN = Calcium Ammonium Nitrate 27 % N.

<sup>2)</sup> Complex = Complex fertiliser 15% N/15%  $P_2O5/15\% K_2O/3\% S$ .

 $^{3)}$  MOP = muriate of potash = potassium chloride, 60% K<sub>2</sub>O, origin BPC White Russia.



Figure 3: Stationary fertiliser mixing plant, left hopper filled with NPK complex, middle hopper filled with MOP, right hopper filled with CAN (© J. Pichler)

A conical pile (approx. 25 t) was formed after the mixture left the discharge pipe of the fertiliser mixing plant (Figure 4). The heap was systematically sampled with a grain sampling spear. The conical piles were sampled at three levels (top, centre, bottom) at two depths (30 cm, 80 cm). Per sampling point, 3 samples were in a mixed sample.



Top: 3 mixing samples Centre: 3 mixing samples Bottom: 3 mixing samples

Figure 4: Conical pile under the discharge pipe of the stationary fertiliser mixing plant with sampling scheme (© J. Pichler)

In addition, three commonly used logistic systems (tanker truck, tipper truck, big bag) were analysed. The tanker truck (commercial volume of 30 m<sup>3</sup>) and the tipper truck (25 t payload) were filled directly from the discharge pipe of the stationary fertiliser mixing plant. The big bags were also directly filled via big bag filler (after the fertiliser mixing). The discharge into the storage boxes took place by tipping the tipper truck or by blowing out from the tanker truck. The blended fertilisers were thus transferred into the storage boxes via two handling steps (loading and unloading). However, several handling steps were necessary for filling the big bag: the fertiliser blends were delivered from the mixing plant discharge pipe by wheeled loader into a channel from where the fertiliser was moved by auger into the big bag filler. The conical piles (each 25 t) formed after discharge from the stationary fertiliser mixing plant, the bulk heaps (each 25 t) formed by unloading the tipper and tanker trucks and emptying the big bags (3, each 600 kg), were all sampled using a grain sampling spear. The bulk heap after unloading from the tipper truck was sampled with horizontal spacing of 1.5 m and at 30 cm and 80 cm levels. The heap made from unloading the tanker truck was less high and so samples were taken at 30 cm and 50 cm. The contents of the big bags were sampled from above at depths of 30 cm and 80 cm. The collected samples were then sent for chemical analysis (N,  $P_2O_5$ ,  $K_2O$ ) and sieve analysis (corn size distribution) to the laboratory of Borealis L.A.T. GmbH in Linz. The analytical procedures used comply with the requirements of the EC Fertiliser Regulation 2003/2003 (Table 2).

	Blend 1	Blend 2	Blend 3	Number of samples
Cone (systematic sampling)	15 <sup>1)</sup>	15	15	45
Bulk blend after unloading from the tipper truck	5 <sup>2)</sup>	5	5	15
Bulk blend after unloading from the tanker truck	11 <sup>3)</sup>	9	8	28
Big bag	6 <sup>4)</sup>	6	6	18
	KAS	Complex	MOP	
Samples of fertilisers -	2	2	2	6
			Total sample	112

Table 2: Number of analysed fertiliser blends

<sup>1)</sup> 30 cm layer depth: 3 levels (cones top, cone centre, material cones base) x 3 composite samples

80 cm layer depth: 2 levels (cones top, cone centre) x 3 composite samples

 $^{2)}$  30 cm layer depth: 3 levels (cones top, cone centre, material cones base) x 1 composite sample

80 cm layer depth: 2 levels (cones top, cone centre) x 1 composite sample

<sup>3</sup>) Different number of samples due to the different size of the conical pile. In addition, the maximum layer depth had to be reduced

from 80 cm to 50 cm.

<sup>4)</sup> In layer depths of 30 cm and 80 cm, 3 composite samples were taken from above in each case.

Data analysis was carried out using Excel and the statistical software SPSS 21. Hereby, a descriptive statistic as well as variance analysis (ANOVA) structural test procedure were applied. After ANOVA, the multiple group comparison according to the Student-Newman-Keuls test (SNK test), and a T-Test were carried out with two groups. The investigated dependent variables are nutrient content (% N, % P<sub>2</sub>O<sub>5</sub>, % K<sub>2</sub>O) and corn size distribution. The investigated factor is the mineral mix fertilisers with three gradations: blend 1, blend 2 and blend 3. The statistical analysis was carried out separately for the conical pile formed after unloading from the discharge pipe of the mixing plant, for the unloaded loose heaps from the tipper and tanker trucks, or from the big bag. The conical pile from the discharge pipe can be seen as the reference sample for all the other trial variants, thereby symbolising the truck filling process.

# Results and discussion Segregation in the conical pile

In Figures 5 to 10 are shown the nutrient composition and particle (corn) size distribution of the three mineral compound fertilisers in the conical pile (30 cm layer depth). Blend 1 (50% CAN, round corn and 50% Complex, round corn) showed, as expected, no statistically significant segregation of nutrients with deviations from the target nutrient blend the lowest in the trial. At a layer depth of 30 cm no significant differences within the mix, as well as in the targeted nutrient presence, (Figure 5) could be determined.



Figure 5: Mean nutrient composition with standard deviation of the mixture 1 in the cone in 30 cm layer of depth. The different letters indicate significant differences (Student-Newman-Keuls test,  $\alpha$  =0.05) between levels top, centre and bottom

Also, the corn size distribution within this mix in the conical pile peak (top), conical pile middle (centre) and conical pile base (bottom) showed, with only one exception, no significant difference (Figure 6).



Figure 6: Average corn size composition with standard deviation of the mixture 1 in the cone in 30 cm layer of depth. The different letters indicate significant differences (Student-Newman-Keuls test,  $\alpha$  =0.05) between levels top, centre and bottom

Blend 2 (comprising 33% CAN, 33% Complex, 34% MOP), shows a physical segregation in the conical pile (Figure 7 and 8). The round corn component (CAN, Complex) and the angular corn MOP segregated, and this effect was shown by the potassium and nitrogen distribution within the conical pile. At the top of the conical pile significantly more potassium accumulated while at the bottom of the conical pile nitrogen accumulated. The corn size distribution showed no significant differences between the levels top, centre and bottom (Figure 8).



Figure 7: Mean nutrient composition with standard deviation of the blend 2 in the conical pile in the 30 cm depth layer. The different letters indicate significant differences (Student-Newman-Keuls test,  $\alpha$  = 0.05) between levels top, centre and bottom



Figure 8: Average corn size composition with standard deviation of the blend 2 in the conical pile at 30 cm layer depth. The different letters indicate significant differences (Student-Newman-Keuls test,  $\alpha$  = 0.05) between levels top, centre and bottom

Blend 3 (50% CAN, round corn, 50% MOP, compact angular corn) shows the largest segregation (Figure 9 and 10). In particular at the top of the conical pile with  $K_2O$  content (40.8%) a much higher content than expected was determined compared with the target content of 30.0%. The expected nitrogen target content of 13.5% could also not be reached. The analysed nitrogen content at the top of the pile was 8.4% and at the bottom 16.3% with  $K_2O$  content in the bottom 22.9%.



Figure 9: Mean nutrient composition with standard deviation of blend 3 in the conical pile in 30 cm layer depth. The different letters indicate significant differences (Student-Newman-Keuls test,  $\alpha = 0.05$ ) between levels top, centre and bottom



Figure 10: Average corn size composition with a standard deviation of the blend 3 in the conical pile in 30 cm layer depth. The different letters indicate significant differences (Student-Newman-Keuls test,  $\alpha$  = 0.05) between levels top, centre and bottom.

Table 3 shows the effect of physical segregation in the conical pile on the theoretical amount of nutrients in the field. The largest deviation can be determined in mix 3. The amount of applied nitrogen is 37.5% less than the target quantity if the fertiliser mix is taken from the top of the conical pile. On the other hand, the quantity of K<sub>2</sub>O is 36.2% higher. The effects of segregation in the conical pile increases with the share of MOP in the blends 2 and 3.

	N in kg/ha	P₂O₅ in kg∕ha	K₂O in kg∕ha
Target quantity blend 1	120	55	80
Тор	117 (-2.5%)	59 (+7.3%)	85 (+6.3%)
Centre	121 (+0.8%)	52 (-5.5%)	73 (-8.8%)
Bottom	123 (+2.5%)	50 (-9.1%)	71 (-11.3)
Target quantity blend 2	120	55	80
Тор	94 (-21.7%)	51 (-7.3%)	102 (+27.5%)
Centre	107 (-10.8%)	59 (+7.3%)	89 (+10.1%)
Bottom	130 (+8.3%)	62 (+12.7%)	69 (-13.8%)
Target quantity blend 3	120		80
Тор	75 (-37.5%)		109 (+36.3%)
Centre	93 (-22.5%)		97 (+21.3%)
Bottom	145 (+20.8%)		61(-23.8%)

Table 3: Theoretical amount of nutrients in the field in kg/ha<sup>-1</sup> and their percentage deviation from the target quantity if the mineral compound fertilisers are taken from the conical pile (30 cm layer depth).

Fertiliser recommendation for an average expected yield of 5 t/ha winter wheat according to the guidelines for proper fertilisation (BMLFUW 2006)

The potential segregation effects during fertiliser application with a two-disc spreader were not considered in this regard. Numerous studies (MATHES and BRUBACH 1966, Heege and Hellweg 1982, MARQUERING 2001) show that, in mineral fertiliser spreading, transverse distribution accuracy is significantly influenced by corn size distribution and corn form.

## Segregation caused by means of transport

Load transfer processes in logistics can reinforce or reduce the original segregation within the conical pile (Tables 4–6). Blend 1, which already showed a uniform distribution of nutrients in the conical pile showed hardly any segregation tendencies after unloading from the tipper truck, tanker truck and big bag. The blends 2 and 3 showed an interaction between fertiliser blend and the means of transport. The pneumatic discharge of the blends 2 and 3 from the tanker truck into the storage box increased the segregation (Table 4). The mean relative deviation is between 16.4% and 43.2%. This segregation effect is due to the different flight characteristics of the round corns and angular compact corns on the flight distances and thus the spatial distribution of the corns in the bulk heaps.

	N in%	$P_2O_5$ in %	K <sub>2</sub> O in %	Mean relative deviation <sup>1)</sup> in %
Target content blend 1	21.0	7.5	7.5	
30 cm (N = 6)	20.9 (3.0%) <sup>1)</sup>	7.5 (11.6%)	7.3 (12.5%)	9.0
50 cm (N = 5)	20.8 (2.3%)	7.5 (8.5%)	7.3 (8.8%)	6.5
Target content blend 2	13.9	5.0	25.4	
30 cm (N = 7)	15.2 (12.4%)	5.1 (5.8%)	21.8 (30.9%)	16.4
50 cm (N = 2)	15.4 (33.4%)	5.5 (8.2%)	21.3 (87.9%)	43.2
Target content blend 3	13.5		30.0	
30 cm (N = 6)	14.9 (21.7%)		26.1 (52.3%)	37.0
50 cm (N = 2)	13.4 (29.9%)		29.5 (30.1%)	30.0

Table 4: Average nutrient content in % with relative deviation in % of the mineral compound fertilisers 1, 2 and 3 in 30 and 50 cm layer depth, after unloading from the tanker truck.

<sup>1)</sup> Relative deviation = (target value – measured value measured value)/measured valuet × 100.

The unloading of blends 2 and 3 from the truck trailer reduces in tendency the segregation effect (Table 5). With the exception of blend 3 at a depth of 80 cm, the mean relative deviation ranges between 2.3 and 5.3%.

	N in %	$P_2O_5$ in %	K <sub>2</sub> 0 in %	Mean relative deviation <sup>1)</sup> in %
Target content blend 1	21.0	7.5	7.5	
30 cm (N = 3)	20.4 (2.8%) <sup>1)</sup>	7.8 (3.8%)	7.9 (5.4%)	4.0
80 cm (N = 2)	20.5 (2.5%)	8.0 (5.6%)	8.2 (7.9%)	5.3
Target content blend 2	13.9	5.0	25.4	
30 cm (N = 3)	13.2 (5.1%)	5.0 (3.3%)	26.5 (4.1%)	4.2
80 cm (N = 2)	13.1 (6.6%)	5.1 (1.9%)	27.1 (6.0%)	4.8
Target content blend 3	13.5		30.0	
30 cm (N = 3)	13.6 (2.6%)		29.7 (2.0%)	2.3
80 cm (N = 2)	11.8 (14.4%)		32.9 (8.8%)	11.6

Table 5: Average nutrient content [%] with relative deviation [%] of the mineral compound fertilisers 1, 2 and 3 after unloading from the tipper trucks in 30 cm and 80 cm layer depth

<sup>1)</sup> Relative deviation = (target value – measured value measured value)/measured value × 100.

After filling of big bags, blends 2 and 3 showed considerable segregation tendencies within the big bags (Table 6). This effect can be attributed to segregation during the filling of the big bags whereby similar segregations can occur as in the formation of a conical pile after mixing in the mixing plant (Figure 4). Further investigations are necessary for clarification of this presumption.

	N in %	$P_2O_5$ in %	K <sub>2</sub> O in %	Mean relative deviation <sup>3)</sup> in %
Target content blend 1	21.0	7.5	7.5	
30 cm <sup>1)</sup> (N = 3)	21.0 (2.4%) <sup>3)</sup>	7.0 (11.6%)	7.0 (13.3%)	9.1
$80 \text{ cm}^{2}$ (N = 3)	20.7 (2.9%)	7.5 (7.7%)	7.6 (10.1%)	6.9
Target content blend 2	13.9	5.0	25.4	
30 cm (N = 3)	15.5 (10.1%)	5.2 (5.7%)	21.2 (19.7%)	11.8
80 cm (N = 3)	14.3 (3.0%)	5.1 (5.3%)	23.8 (6.8%)	5.0
Target content blend 3	13.5		30.0	
30 cm (N = 3)	16.4 (17.3%)		20.9 (44.1%)	30.7
80 cm (N = 3)	14.1 (8.5%)		26.7 (14.6%)	11.5

Table 6: Average nutrient content in % with relative deviation in % of the mineral compound fertilisers 1, 2 and 3 in the big bag at 30 cm and 80 cm layer depth

 $^{1)}$  30 cm or  $^{2)}$  50 cm vertical depth from the top of big bag.

3) Relative deviation = (target value - measured value measured value)/measured value × 100.

## Conclusions

The miscibility of blended fertilisers is affected by chemical properties (e.g. nutrient composition, water absorption capacity, explosiveness) and physical properties (surface, bulk weight, corn size spectrum). Systematic sampling of a conical pile of blended fertilisers shows approximate nutrient distribution in the conical pile and can deviate from results of punctual sampling.

The investigations carried out here show that muriat of potash accumulates centrally within the top of the conical pile. The granulated round CAN corns roll downwards and collect at the bottom of

the conical pile. This segregation can lead with blend 3 (50% share of MOP in the blend) to a lower nitrogen quantity (-37.5%) and higher potassium quantity (+36.3%) in the field. The number of handling procedures, as well as the type of transport used, have differing effects on nutrient segregation. While the transport systems tanker truck and big bags demonstrate a clear segregation effect, using a tipper truck can offer partially less segregation. Due to these segregation effects, fertiliser after blending should not be stored intermediately because multiple restructuring of the conical pile can cause segregation

If different single fertilisers are used that have very different physical characteristics, e.g. corn form – angular or round, it should be considered whether these fertilisers might be better applied individually (rather than as a mix). Alongside adjustments for a possible range of material-specific properties and logistic system effects, inaccuracy of application may be further increased through compromises in machinery adjustment (MAROUERING 2001).

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