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PHYSICAL PROPERTIES OF FERTILIZERS AND SPREADING: STANDARDIZATION, REGULATIONS AND MANUFACTURE

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RESUME

Cette communication s'efforcera de mettre l'accent sur les développements récents et les relations entre les propriétés physiques des engrais et leur épandage. C'est une vue intérieure de ce qui se produit dans :

- *L'organisme européen de normalisation CEN, où l'on élabore les normes d'essai du matériel d'épandage et les normes de mesure des propriétés physiques.*
- *La Commission européenne, DG III, où les réglementations sont examinées y compris les discussions sur le bulk blending et l'homogénéité.*
- *L'industrie et son Association européenne EFMA qui a beaucoup oeuvré sur les propriétés physiques et l'épandage des engrais et leurs implications sur les procédés de fabrication.*

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1. INTRODUCTION

« The physical form in which a fertilizer is produced is of considerable importance, both agronomically and in regard to satisfactory handling, transport, storage, and finally application to the field. Most of the problems encountered with fertilizers probably are those resulting from deficiencies in physical properties; frequent problems include caking, dustiness, poor flowability, segregation, and excessive hygroscopicity ».

« Physical properties of fertilizers, unlike the chemical composition, normally are not governed in commerce by laws. Physical condition usually is a matter only of private agreement between purchaser and supplier. As a result, there are few « official » methods for measurement and evaluation of physical properties ».

Those excerpts come from the IFDC « Fertilizer manual » edited back in 1979. They are still perfectly valid even though quite a good deal of work has been done in the mean time to narrow down on some specific areas.

Practically no regulations existed in Europe, either at the European Union level or at the individual Member States level, regarding physical properties of fertilizers or their spreading with the exception of some phosphate fertilizers for which a minimum fineness was required to ensure sufficient availability.

The situation however has changed in the last few years on two counts. The first one was for safety reasons and concerns specifications of physical properties of straight ammonium nitrate having a nitrogen content above 28%. The second change is indirectly related to spreading since European regulations now limit the amount of nitrogen that can be spread per hectare, irrespective of its source.

Introduction of bulk blends in Europe and the partial demise of ISO in the field of standardization of physical properties has led CEN (Comité Européen de Normalisation) to pick up the theme and follow up on TVA and ISO work. Furthermore, European Commission regulators have been requested by a Member State to regulate bulk blends with the underlying principle that the product should not segregate during handling and spreading.

So, where do we stand now and what could be the foreseeable future?

2. STANDARDIZATION

A. Technical Committee 260 "Fertilizers and liming materials"

As far as physical properties are concerned, many have been considered and some can now be measured by standardized methods. However, work priorities had to be chosen in order to achieve a reasonable output while not straining available manpower and laboratory facilities. The following has been done or is underway:

- Angle of repose

Since this item is quite important in certain calculations of storage capacity, the corresponding ISO standard has been transposed to a European Norm (EN). However there was much discussion about the qualification of the title i.e. should it be "Static angle of repose" or "Dynamic angle of repose". At the same time, the IMDG Code « Tilting box » test was considered but finally not accepted.

- Particle size

The European Norm (EN 1235 Test Sieving) has been elaborated from the corresponding ISO standard. The interesting feature however is the definition of median particle diameter D50 which was introduced to facilitate comparisons between fertilizers of different origin or manufacture. In a nutshell this definition says that D50 is the theoretical opening of a screen which would let pass 50% by weight of the sample under testing. It can be easily obtained by calculation from the individual screen results obtained by the sieving test.

Another item, not in the standard now but under discussion, is the so-called « granulometric spread ». Using the same reasoning as above for D50, we can define any fraction which passes through a theoretical sieve opening i.e. D90 or D10 and the granulometric spread would then be defined as the difference between the two, expressed in millimeters. The importance of such a figure, once again obtained by simple calculation from sieving results, is evident; it means that the smaller the number, the nearer the size of 80% of the granules is to the median particle diameter. In other words, for a current granular fertilizer having a D50 of 3.3 mm and a granulometric spread of about 1.5 mm, this means that 80% of the product would be between 2.6 and 4 mm. In practice, no meaningful segregation would occur with such a fertilizer. The discussion as to the most convenient definition is still underway since there are differing opinions as to the choice of the small theoretical opening, mainly due to the fact that on the one hand, D10 is considered by some to be too lax and on the other, some would like to link, mathematically, by a constant, the granulometric spread to the median particle diameter. Such a relationship would be advantageous with respect to segregation since it could probably cover all fertilizers ranging from powders to forestry type granules.

- Density

For bulk density, whether granules or powders are considered, this was an easy task since the ISO standards were already available and transposal to EN follows simple rules. As far as apparent granule density is concerned, there was no incentive to have the ISO mercury method transposed to a European standard.

- Dust

With our increasing awareness for human safety and increasingly stringent regulations, this item, once considered as an objectionable nuisance, can not be overlooked. However it is a somewhat subjective matter and the work on a standard for measuring dust is always hampered by the so-called « definition » problem of what should be considered as dust in fertilizers. Nevertheless CEN has undertaken the task of establishing a standardized method for measuring the loss of matter of a sample under defined conditions in a spouting-bed « reactor ». An apparatus has been designed, tested, modified and ring-testing is being carried out. Results are promising.

However we consider that particle size not being taken into consideration since only loss of weight is measured, such a method may not give representative results of any visual impression a customer would get when spreading fertilizer in the field. Therefore another method is now being considered, based on light beam scattering. Only a few preliminary tests have been run so far, which means that a standard method is still quite far away.

- Hardness, crushing strength, impact/abrasion resistance

This one is a sad story. Fertilizer experts are convinced that a standard for the measurement of the granule resistance to breaking is of utmost importance. Three ring tests have been run by the members of CEN Technical Committee 260 Working Group 2 in charge of standardization of physical properties of fertilizers, by applying variations of a method based on the principle of applying force to a single selected granule. The first ring test was a failure with unexplainable and unacceptable discrepancies in test results. Very careful planning and tight procedures were used for the other two extensive tests. The results were slightly better but still unacceptable from the statistical point of view. Finally, and after protracted discussions, it was accepted that nobody had any good idea to explain the discrepancies and the decision was taken to publish the results in a Technical Report for the benefit of the scientific community at large.

The only conclusion that can be drawn from the work that has been done is: whenever crushing resistance figures are published, these should be considered only as indicative and never be used as reference. However, for comparison purposes, intra-laboratory test results on the same type of product, obtained by the same people on the same apparatus under the same conditions, seem valid.

Since crushing strength was a failure, attention turned to other methods designed to evaluate general fertilizer resistance to mechanical forces. Various methods are now being evaluated but it is too early elaborate on the type of methods under investigation or to say if there are any chances for one or several standards.

- Humidity, moisture

This is a difficult subject. Two ISO standards exist; one by a gravimetric measurement before and after drying at 105°C and the other, by drying at reduced pressure. Both have been transposed to EN. However, both have limitations in their scope which render them inappropriate for some fertilizer formulations. On the other hand, one or another form of the Karl-Fischer method is used quite frequently but has not been standardized for fertilizers. Finally there is the problem of absorption or desorption of water on the surface of fertilizer particles which may play a role in the rate of flow, a phenomenon important in fertilizer transfers, mainly in spreading where the flow through a variable size orifice is usually used to regulate the amount of fertilizer to be spread.

The standardization of two Karl-Fischer methods is now under way. The first one is a straightforward one, the second one is slightly more sophisticated in that it requires prior comminution in isopropanol and it is the water extracted by the isopropanol medium that is measured by standard Karl-Fischer procedure.

There seems to be no possibility to estimate surface moisture and it has finally been decided not to try to find a method since the problem appears to be too complex.

The remaining point is what the Tennessee Valley Authority researchers have called « Critical Humidity » which is the determination of the break-point conditions under which a fertilizer is neutral to water absorption and desorption i.e. would neither lose nor gain weight due to water pick-up or loss from the atmosphere. Many methods have been proposed but are deemed somewhat unreliable when it comes to standardization. However it is an important property of a fertilizer with respect to bulk storage, transport, handling, blending and spreading.

- Homogeneity, segregation

This item has been on the agenda of the CEN Technical Committee 260 for some time before the status quo was released. The search for a simple method of estimating homogeneity or the degree of segregation proved frustrating even though quite a few, non standardized methods, exist. In a nutshell, most of the methods stumble either on too complex or protracted chemical analysis or on the definition of homogeneity. In fact, this is, in some ways, a "philosophical" multi-faceted question where prior decisions have to be taken, answering questions such as:

- what are we looking for, what is the objective?
  - regulatory control?
  - product comparison?
  - operational requirements in production or raw material procurement?
- do we accept to have an arbitrary method leading to a definition of the subject or,
- do we define the subject and conceive a method for checking?

The Commission DG III working party « Fertilizers » has also had this subject on the discussion table for some time due to a formal request from Spain, asking for a regulation of bulk-blends. It has been studied, discussed, regulatory proposals have been made, but it remained a « Sleeping Beauty » now for at least a year. No doubt however that the « awakening kiss » will come sooner or later.

- Sphericity

Sphericity is an interesting characteristic with respect both to mass motion and to particle flight path shape. Considering the latter, besides higher drag and, consequently, a shorter flight, researchers using high speed photography have confirmed the erratic motion of individual particles along their flight path resulting from the irregular shape if the particle spins on any axis, all resulting in higher dispersion on landing compared to the normal distribution pattern of perfectly spherical elements. Such a dispersion may even be considered as beneficial in some cases. However uneven particles often lead to uneven mass flow through an opening

and there is a definite possibility of bridging and choking. Despite all the interest in this property, no European standardization has been considered yet.

- Rate of Flow: « Flowability »

There is a high incentive to be able to estimate the amount of fertilizer passing through an opening since most of the spreaders now in use in Europe are spinning disc spreaders which use a variable orifice to regulate the flow of fertilizer to the discs. Furthermore, « flowability » being a global characteristic embodying such elements as surface roughness or grain irregularities or even grain stickiness due to superficial absorption of water under high relative humidity conditions, such a method could partly avoid systematic recalibration of a spreader with every fertilizer used under any given set of conditions.

A method and a simple apparatus have been proposed and ring-tested. The apparatus is in fact a funnel, already standardized for fertilizer bulk density measurements. Some difficulties have been encountered during initial ring testing. These were partly attributed to actual funnel construction despite the fact that all the funnels have been built to standard specifications. We have been compelled to calibrate all the funnels with standardized glass beads. In March 1996, we have reached the stage where a final confirmatory ring test has been decided. A standard testing method should therefore be available in 1997.

B. CEN Technical Committee 144 « Agricultural and forestry machinery »

The standardization work of this Technical Committee is self-explanatory from its title and includes all fertilizer spreaders. Of particular interest in our case is a draft proposal in two parts now being finalized by a working group of experts before being forwarded for consideration by the Technical Committee, the title of which is: Full width distributors and broadcasters for solid fertilizer - Environmental preservation - Part 1 Requirements, and, Part 2 Test methods.

Part 1 deals in with the machine itself and its subsystems with respect to environmental considerations. The important point here is that the standard defines maximum allowable deviations for flow rate during emptying and maximum allowable deviations between set flow rate and intended flow rate in % m/m. For the former, this varies between 10% for a flow rate of less than 25 kg/min to 5% for a rate of more than 150 kg/min. The latter is set at 15% for a set flow rate of less than 25 kg/min to 7.5% for a set flow rate over 150 kg/min. Finally the draft says that the maximum coefficient of variation when driving to and fro should not exceed 15%. To note that, at this point, no reference is made to the fertilizer which is being spread.

Part 2 deals with the test methods for application of fertilizers at three different application rates i.e. the lowest and highest as given by the machine manufacturer and 400 kg/ha. For the moment, as far as testing for calibration purposes is considered, test fertilizers should be chosen from six different fertilizer groups i.e. prilled (bulk density < 900 kg/m^3 and > 900 kg/m^3), granular (bulk density < 900 kg/m^3 and > 900 kg/m^3), compacted and « crystalline ». The test methods include: contact with obstacles, filling grade control, adjustment of flow rate, reduced working with, flow rate regulation system, coefficient of variation for transversal distribution during variation in flow rate, eveness of transversal distribution, eveness of transversal distribution near the field edge, eveness of distribution when spreading with equipment for top dressing, deviation from intended flow rates for machines with a non stepless regulation system, deviation from calibrated flow rate, eveness of flow rate and rearward throwing length.

Technical Committee 260 experts participated actively in this work from the fertilizer point of view, and to put things frankly, to avoid that a group of machinery experts defines, for its own purposes, test product specifications which would eventually not fit the pattern of products currently available on the European market. Finally it seems that the broad groups defined above will be adhered to without much more stringent specifications. That however does not mean that the instruction handbook which comes with every machine, will not give most of the physical properties of the fertilizers with which the machine has been tested or the complete analysis of the fertilizers with which the machine has been calibrated. To avoid recalibrating his machine, this would lead the farmer to request the same fertilizer from his supplier thus establishing some sort of de-facto product standards, the same way as Microsoft did with the DOS.

3. EUROPEAN REGULATIONS

As stated in the introduction, changes have been brought into European regulations regarding fertilizer physical properties. While the only ones existing now relate to the safety of straight ammonium nitrate, and indirectly to the spreading of nitrogen which is limited to avoid too much nitrate nitrogen in the water table, some new paths are being considered for bulk-blends.

- Ammonium nitrate

Products containing more than 28% nitrogen are currently sold on the European market. To ensure that they remain as safe as possible in storage and during handling while maintaining the possibility of inter-state commerce in Europe, the European Commission has spelled out some specifications for these products. These concern the following points:

- maximum combustible material content of less than 0.4% or 0.2% as carbon whether the nitrogen content is above below or above 31.5% N,
- porosity: fuel oil retention of less than 4% after two thermal cycles between 25 and 50°C,
- pH of more than 4.5 for a 10% water solution,
- granulometry: less than 5% through a 1 mm screen and less than 3% through a 0.5 mm screen,
- content of chlorides expressed as chlorine of less than 0.02%
- heavy metals, no addition, copper limited to 10 mg/kg fertilizer,
- facultative conformity to a specific detonation test.

Although not all of these are physical properties strictly speaking, this is one of the excursions of regulators outside the domain of chemical composition of fertilizers.

Limitation of nitrogen that can be spread per hectare, contained in the so-called « Nitrate Directive », is designed to limit nitrogen seepage to ground water and avoid contamination of drinking water by nitrate. This regulation is not directly related to machine spreading as such, it has nevertheless had an impact since the farmer wishes a spreading of his nutrients as even as possible. Thus the eveness of spreading, although already requested before this regulation, has received more impetus than ever, coupled with more demand on the precision of the quantities actually spread per hectare.

- Bulk-blends

No regulations exist today in Europe. The law says explicitly that a product, for instance NPK, is entitled to bear the « EEC Fertilizer » label if it is a « Product obtained chemically or by mixing without incorporation of fertilizer organic matter of animal or vegetable origin », without any other requirements regarding physical properties. Such a definition includes bulk-blends. For various reasons, mainly because of segregation and uneven spreading of fertilizing elements, Spain has requested that the European Commission takes steps to regulate the very existence of bulk blends, either by outlawing them altogether or, failing that, by writing down stringent requirements to avoid segregation and its consequences.

Discussions in the « Fertilizer » Working Party of the Commission and external studies have lasted for at least eighteen months. During the process several points emerged, three of which are important:

- product denomination; experts agree that if there is to be a regulation regarding bulk-blends, these products will have to bear a specific name for the sake of information of the customer. This in turn legally requires a specific definition of what is a bulk-blend in order to differentiate it from current compound or complex fertilizers having approximately the same nutrient content in every granule. It is practically agreed that the word « mixed ... ». i.e. Mixed NPK Fertilizer will be used. Translational problems will have to be overcome by national adaptations since in French for instance, the wording would have to be: « ... de mélange » to convey the idea of bulk-blending even though it is not the exact translation. A price to pay for our diversity...

- product definition; not yet settled as to its precise wording. Even though everybody now seems to agree on what we are talking about, point which was not evident at the beginning, the consensus on the precise wording is not yet on the paper, mainly because agreement has not been reached on the type of official control method to differentiate between compounds and bulk-blends, which has to be simple. In fact the whole problem boils down to the following simple question: how do you determine if a fertilizer is a bulk-blend or not if all the granules have approximately the same external appearance with respect to color, size and shape? On the top of that, some experts contend that there is no need for one single simple official method and that it is up to the Member States to resolve that matter individually!

- product specifications; there is a consensus on the fact that it is highly unlikely for the final product definition to resolve directly the problem of a possible undesirable segregation, and that some product specifications will be needed. The discussions and proposals have centered on product size but no agreement has been hammered out at the time of writing. Very briefly, the proposals for granular products sum up as follows:

a) 90% of the product should be between 2 and 5 mm, and the fractions between 2-3.15 mm and 3.15 and 5 mm should have the same chemical analysis.
b) 90% of the product should be between 2 and 4 mm and have a homegeneous granulometric distribution.
c) Median particle diameter D50 above 2 mm and granulometric spread (D90 - D10) less than 1.8 mm.

The main flaw is that all these proposals address granular fertilizers and not fine-grained or powder forms, nor do they address large size granules used in forestry, which would thus both be implicitly excluded. This does not seem to be acceptable. The solution may lie in a modification of the third proposal c) by relating granulometric spread to median particle diameter by some simple mathematical equation and setting a very low bottom limit on D50.

- finally it was made quite clear that whatever specification would be made applicable to bulk-blends, compound or complex fertilizers would be subject to the same, in order to avoid market discrimination.

4. WORK OF THE EUROPEAN INDUSTRY AND OF EFMA

There is no point in going back to « historical » times when pioneers battled with caking of any fertilizer they manufactured, this is still true, albeit much more sophisticated. Caking problems resolved, even if not entirely to everybody's complete satisfaction, attention turned to spreading. Many may still remember the times of shoe boxes lined up as « collection equipment » to assess the performance of a spreader. Some simple conclusions dating back to those times are still valid and have been improved upon since. Today's environmental pressure combined with farmers' demands, have led to the evolution of the spreading equipment and of the fertilizer. This has not happened in a haphazard way but stems from tedious and costly testing of large quantities of fertilizer in dedicated spreading facilities.

The general trend for fertilizers is dictated by simple mechanics: the farther you want to throw a granule, the more force you need to propel it. This translates in demand for denser, rounder and tougher particles. In practice there is a limit to everything and compromises have to be reached. Such compromises have led to granules getting bigger and bigger.

For spreading machinery the trend is dictated by larger fields, environmental pressures and economics. It all leads to equipment getting larger, more and more sophisticated to obtain the best eveness of spreading attainable at a reasonable, if not always the least, cost.

Today, practically every company has an ongoing testing programme for the spreading of its own fertilizers. However, results from such operations are usually never published in the scientific literature. What appears from time to time is a Ph D thesis and some papers based on the doctoral work which was partly or totally sponsored by an industrial partner but, in those few cases, detailed strategic conclusions are never published.

Some five years ago, the European Fertilizer Manufacturers' Association (EFMA), set up a working group designed to gain better insight into fertilizer spreading. The effort cumulated in an extensive test in Denmark on fifteen different fertilizer formulations, five different disc spreaders working at three levels of output. For each fertilizer three main characteristics were measured prior to spreading: median particle diameter (D50), apparent bulk density and flowability. In a nutshell, the conclusion of the group was that, due to more or less strong correlations found between the above physical properties and the spreading results, it would be helpful for both the spreader manufacturers and the farmers, to be informed about these physical properties and that a way of informing them should be found. A proposal for this information system has been made internally but finally the Steering Committee of EFMA decided to pass the research data and the proposal to CEN TC 260 for further action. This item has been taken up for preliminary discussions in the TC 260 working Group « Physical properties ». Basically, since the above properties can be very easily measured with simple equipment, either a transmission of the results by standardized marking of the bags for instance, or on the spot measurement just before spreading, would lead to a simplification of farmer's work while contributing to more precise spreading. It could also help the spreader manufacturer in establishing his calibration charts on a firmer basis rather than relying on the type of fertilizer and the brand or site of manufacture as has often been done in the past and is still practiced today by some.

However, some caution has to be exercised in this field since nothing will replace the actual calibration with the product under prevailing ambient conditions just prior to spreading. Furthermore, studies have shown that farmer's knowledge and experience, coupled with good spreader maintenance, are more important for even spreading, than physical properties of fertilizers.

As far as manufacture is concerned, the « writing on the wall » being for bigger and rounder granules with ever smaller granulometric spread, any engineer can imagine what should be done to upgrade his process, starting with the screening operation. This however would have an impact on his recycle ratios and eventually on capacity. Chances are that such secondary impacts could be negative, necessitating other adjustments or even, in the worst case scenario, transforming completely a process if not abandoning it altogether. For instance, there is no easy way of obtaining prills with a D50 of over 3 mm by the traditional prilling tower technology with low viscosity melts like in the case of ammonium nitrate or urea. The solution there would be cold granulation or prill fattening which are both radical departures from the original prilling tower. Finally it is, and will be, up to each manufacturer to decide to what extent he can, or intends to, follow existing trends. These will not all last forever, since there are good agronomic reasons to stop on the path of granule growth for instance.

5. CONCLUSION

Even though recent experience in Europe has shown that the most important « physical » property of any fertilizer is its price, the following quotation from TVA's « Fertilizer manual »:

« Customer acceptance or preference for a particular fertilizer over another of equal plant-food content almost always is based on physical properties of the product » is as true as ever. Based on this fact and in order to avoid conflicting claims, fertilizer manufacturers in Europe agreed to launch a standardization effort through CEN. Such an undertaking has not only the advantage of being able to influence directly the standardization orientations but also the advantage of gaining a foothold in the European Union's regulatory process.

Fertilizer manufacturers have thus exercised their influence on the measurement of fertilizer physical properties by setting priorities and have had their say in the standardization of the fertilizer spreading machines.

Looking on the regulatory side of things, industry is contributing directly to the enlargement of the scope of existing regulations, better market conditions and last but not least, better mutual understanding.

Standardization is a lengthy process which can prove to be frustrating; as a case to the point is the fact that we now have no hope of being able to measure correctly the resistance of a fertilizer to mechanical forces on particulate basis, contrary to commonly accepted prior wisdom. Some other « bad » surprises may still be in store.

Setbacks however, should not obscure the important fact that good progress has been made, (see Table in Appendix) and manufacturing, standardization and regulations tend to work more in cooperation rather than hold to « ivory tower » isolation.

Appendix

TABLE

Standardization of physical properties of solid fertilizers

Method of measurement	CEN Work Item	Work Item status	EN Standard	ISO Standard	EU/DGIII Regulation
Angle of repose	260004	finished	12047	8398	-
Density (bulk loose)	260005	finished	1236	3944	-
Density (bulk tapped)	260006	finished	1237	5311	-
Density (particle apparent)	260010	deleted	-	-	-
Dust	260007	draft TR	-	-	-
Flowability	260028	draft EN	-	-	-
Hardness (resistance to crushing)	260008	finished	TR12333	-	-
Homogeneity	260011	suspended	-	-	-
Moisture content (reduced pressure)	260014	finished	12049	8189	-
Moisture content (heating 105°C)	260015	finished	12048	8190	-
Moisture content (Karl Fischer)	260038	draft EN	-	-	-
Sampling (simplified)	260016	finished	1482	-	76/116/EC
Sieving (particle size & D50)	260003	finished	1235	« 8397 »	-
Spreading (machine specs & testing)	144040	draft EN	-	-	-
Fertilizer Ammonium Nitrate (N > 28%)	-	-	-	-	80/876/EEC
Bulk-Blends	260011	suspended	-	-	in preparation