International Fertilizer Industry Association

IFA Technical Conference

Edmonton, Canada

12-15 September 1988

International Fertilizer Industry Association - Secretariat: 28 rue Marbeuf - 75008 Paris - France
Tel. +33 1 53 93 05 00 - Fax +33 1 53 93 05 45/47 - ifa@fertilizer.org - www.fertilizer.org

THE PHYSICAL QUALITY OF FERTILIZERS

H. Hero, H. Takala
Kemira Oy, Finland

SUMMARY

In the fertilizer trade, the chemical quality of fertilizers is almost a self-evident fact and strictly defined by agreements. On the other hand, the physical quality is less well defined - without either agreements on methods or information on figures.

Both the buyer and the seller have their own views on requirements, for example in respect of free flowing, dust-free, uniform granules.

There are confusions over a simple matter like granulometry.

In practice, every producer uses self-developed or self-applied quality control methods. Results obtained this way are seldom unambiguous or comparable.

The presentation deals with the control and inspection methods regarding physical quality and their efficiency in practice. The most important aspects of physical quality are caking, dust and the granules' handling durability.

The effect of varying raw materials and changing production conditions on the final quality is followed by, for instance, control methods and advanced research.

In order to standardize the methods, intensive co-operation between both the buyers and the sellers as well as between different producers is needed.

1 INTRODUCTION

Fertilizers as a concept encompasses a wide area, including natural fertilizers, compost products, organic fertilizers and so-called chemical fertilizers. Chemical fertilizers as such, are industrially and chemically manufactured fertilizers in liquid, powder, granular or compacted form.

This presentation concentrates on granular field and garden fertilizers. Such fertilizers can be in straight or compound form and may contain secondary and trace nutrients in addition to the major nutrients.

These are entirely chemically produced fertilizers (= compound) either half-dry, dry granulated or with the different granulated components being mechanically bulk blended.

2 QUALITY

For the purposes of this presentation, quality refers to fulfilling the target values set in advance.

For this presentation quality concerns primarily product quality, although product quality alone is only part of the concept of quality. In respect of fertilizers, quality is customer-orientated, that is, the product should meet the customer's expectations and requirements. The customer's opinion of the product is final and decisive.

Quality criteria are measurable values, which have usually been set by company management. In view of production, quality is good when these values are consistantly fulfilled.

3 WHAT KIND OF FERTILIZER IS OF GOOD QUALITY IN RESPECT OF PHYSICAL PROPERTIES?

The customer thinks that good fertilizer:

1. is always of the same quality
2. has a smooth, rounded granular size normally of 3.5 mm
3. is free-flowing
4. is dust-free

For the manufacturer this is possible, but with the use of various raw materials and different processes, great demands are made on the production personnel. Good quality should not be associated with extra costs, but rather it should be recognized that it is poor quality that costs!

4 QUALITY FACTORS AND QUALITY OBJECTIVES

Customer-orientated quality thinking stresses the customer's needs, expectations and requirements. Fertilizers should be both easy to handle and spread. What is most important however, is that they should enable the farmer to achieve the best possible yield.

A most important quality factor is safety: fertilizers should be safe in production, transport, storage and in final use. Both the customer and user should be made aware of the possible risks through sufficient transport and storage directions. (1)

The following points normally fall under the category of physical quality:

1. Caking
2. Dust
3. Granule size distribution
4. Granule strength
5. Abrasion resistance
6. Even spreading
7. Hygroscopicity
8. Bulk volume
9. Granule roundness

In addition, quality development of products concentrates also on:

10. Porosity
11. Smoothness of surface
12. Chemical reactions in granules
    - KCl, K_2SO_4, H_2SO_4 etc.

There are numerous methods for determining these properties and since nearly every company has its own - it is often difficult or even impossible to compare results. Examples of methods: TVA and UKF etc. (2, 3, 4, 5)

5 QUALITY CONTROL

5.1 Organization

Quality control of the physical characteristics of production is best carried out at the manufacturing plant.

Quality control is often divided into two aspects: quality control and quality assurance. To date physical quality has been determined more at the final 'passive' checking stage. With better co-operation within the manufacturing, and with the aid of high technology, quality control should become a more active, on-line function.

In Kemira - Finland, the plant laboratory is entirely responsible for quality control and ever more and more quality control measurements and analyses will be made at the plants on-line.

Physical quality is checked now by taking samples on a 'shift-basis' directly from production lines and on a day-basis from deliveries.

Quality control at the plants is not enough to achieve and maintain good quality. This work requires research and thorough testing. Research and development may take place at the plants, but successful work requires specialist knowledge and equipment. This can best be achieved at a centralized research and development location.

5.2 Daily routines

Physical tests are continuously carried out by the plant laboratory so as to check:

Caking	4	samples/d/plant
Granule strength	4	- " -
Dust	4	- " -
Sieves	4	- " -

This requires 1-2 persons' work/d.

5.3 Control methods of physical quality

5.3.1 Routines

The routine methods developed for the plants should be rapid and easy enough to be employed in daily work. The most common methods involve:

1. Measuring of caking
2. Measuring of dust
3. Measuring of granule size distribution
4. Measuring of granule strength
5. Measuring of roundness
6. Spreading results
7. Thermal decomposition

5.4 Routine methods, advantages and shortcomings

1. Caking tests are performed on 100 g microbags at room temperature. Testing time is 24 h and pressure 100 kPa. This is a relatively reliable laboratory method although it is rather slow. In terms of production, there is need to develop a more rapid method, which will give an indicative result within 1-2 hours.

2. Dust is measured by the fluidized bed method, where all particles less than 0.5 mm are regarded as dust. The method is quick and fairly reliable. However, one sample seldom represents the whole production batch.(6)

3. Sieve analysis. In Kemira, sieves in accordance with the DIN 4188 are used, 20 cm in diameter. The effect of sieving time and shaking ferocity has little impact on the result.

In addition to sieve granules, the result also indicates the average size of granules.

Despite sieving being a relatively simple process, the results between different laboratories are often not alike. In sieving it is important to keep the sieves in good condition - the actual sieving itself has a smaller effect on the results than the condition of the sieves. (Figure 1 and 2)

4. Granule strength is measured as the force (N) which is required to break a granule 3 mm in diameter. 50 parallel determinations are made. The granule strength is of great importance in the caking of fertilizers and other matters concerning handling durability. In Kemira, the minimum value for NPK fertilizers is 50 N.

5. The roundness of granules is measured using an inclined plane which is vibrated in a certain direction. The method is quick, but somewhat difficult to examine as uneven granules tend to remain on a zero-level. The roundness of the granules is a matter of appearance, the significance being merely visual.

5.5 Automatic quality measuring equipment

The manufacturer's need and wish is to get reliable information from production instantaneously and continuously. In fertilizer production this is still difficult but, nevertheless, some points have been automated:

5.5.1. Granule size analyzer

Several automatic granule size analyzers have been developed, the most common being automated sieves.

Using the latest technology, Kemira has developed a granule size analyzer which continuously analyzes granule size distribution, granule roundness and other tendencies both from the process-cycle and the end-product (Figure 3).

5.5.2 Control of coating

Coating can be followed easily from the product line by marking the coatings in a suitable way. However, it is possible to follow only whether coating has been done or not - the smoothness of the coating cannot be so readily seen.

5.5.3. Final temperature

The final temperature of the fertilizer is, especially with NPK fertilizers, very important. Constant control of the temperature is readily achieved by means of an IR meter or PT 100 probe. The final temperature should always be below 40°C, preferably below 35°C. It has been discovered that the quality improves still further if the final temperature is below 10°C.

6. MAIN QUALITY TOPICS

6.1 Caking

Caking is perhaps the worst defect of physical quality.

Caking is almost always measured from the product for sale and the result thus obtained is examined individually. However, the product can be thoroughly dried out, thereby eliminating caking. This can, however, be problematic if the smallest extra amount of water then drastically increases the risk of caking for instance during storage or transportation.

In quality development studies, it is recommended that the increase of caking be measured by letting the product absorb air moisture under normal conditions (Figure 4).

Caking becomes real for the customer when it disturbs the normal application of the fertilizer. From the manufacturer's point of view, as a rule of thumb, caking can become apparent when force is required to break a formed lump in the hand. Another indication may be gained by dropping a bag once from the height of 0.5 m and then testing if the material is then free-flowing.

Caking depends on many factors:

1. Moisture
2. Grade and P_2O_5 water solubility
3. Raw materials
4. Process
5. Temperature
6. Coating
7. Time
8. Pressure

Caking is considered to consist of two phenomena: Crystal bridging and capillary adhesion. In both cases the "bonds" between the granules can be stronger than the granules themselves. (7)

Usually it is a relatively simple matter to prevent N and NP fertilizers from caking. Nitrate-based NPK:s are clearly more difficult as KCl and K_2SO_4 readily react with nitrate causing caking and dust. (8)

The problem increases when the nutrients are changed in the following order, for example:

N P K ratios	
1 : 2 : 1	easy
2 : 1 : 1	
5 : 1 : 1	
3 : 1 : 2	difficult and B-class

6.1.1 Examples of problems and solutions

Reason for caking		Improvement possibility
moisture		changing raw materials/more drying - adding $MgSO_4$ - adding Fe^{3+}
grade		
-	27-6-6	changing raw materials/drying
-	18-7-14	- " - /P_2O_5VL change
raw materials		
-	H_3PO_4	more impurities in acid or rock
-	H_2SO_4	less quantity
-	K_2SO_4	drying/H_3PO_4 quality
-	KCl	dissolving 0 % or 100 %
temperature		as low as possible after cooling high enough in granulation
coating		right materials/amounts
time		not too long a storage not too short a storage

6.2 Dust

Many manufacturers regard dust as almost harmless, however, it is a clear quality defect. Dust causes problems in bag sealing, caking, functioning of the spreader, and in top dressing which leads to plant scorching. Dust can be loose, inert anti-caking agent or loose fertilizer salts. (9)

Cause of dustiness	Improvement
inert powder	coating
abrasion	stronger granules
granule reactions determined by thin layer microscope image	process/raw materials
grade	process

6.3 Appearance of the fertilizer

According to the customer, the products should always be the same with regard to granule size, size distribution, shape and colour. However, the manufacturer produces tens of different grades, which are manufactured from various raw materials from different sources and even using differing processes.

By developing processes and skills it is possible to approach the target, which can, depending on the markets, be for example according to the following granule size distribution:

+ 4 mm	- 10 %
+ 2 - -4 mm	+ 90 %
- 2 mm	- 1 %
Average size	3.0 - 3.2 mm

or

+ 5 mm	- 5 %
+ 3 - -5 mm	+ 90 %
- 3 mm	- 5 %
Average size	∼ 3.6 mm

Customer complaints most usually concern small granules.

6.4 Spreading quality

The quality of fertilizers spreading evenly is one of the most important factors of physical quality for the farmer: The product should flow steadily and uniformally from the machine.

The best type of spreader is the placement spreader whereby fertilizer is fed from the same machine together with seeds. Fertilizer is thus placed directly into the soil between the seed rows. This spreading method sets high quality requirements for the fertilizer.

The most popular mechanical spreading method in the world is to use a centrifugal spreader. However, this makes the necessary examining of the spreading track very difficult. Should the physical quality of the fertilizer change, the spreading pattern would alter radically without the user's knowledge.

Although adjusting the machine's spreading rate may be easy, the width and the centre of the spread is seldom even considered (Figure 5 and 6).

The most important factors in ensuring even spreading are:

1. Grade
2. Granule size distribution
3. Surface of the granule
4. Bulk volume

The condition and maintenance of machines is very important, a point which, however, is beyond the fertilizer manufacturer's control.

6.5 Curing

In cases even of the slightest doubt, quality is checked in Kemira at the plants in respect of curing by taking one sample directly from the production flow, a second sample after two days and a third upon delivery.

As the curing develops, it becomes difficult to take a representative sample of the stored product. However, experience has shown that curing which has taken place at room temperature affords adequate assurance of quality. (Figure 7, 8 and 9)

7 QUALITY CONTROL RESEARCH - A CENTRAL LABORATORY

7.1 Pilot tests

Kemira's Research Centre has pilot plant installations, which can manufacture nearly all fertilizer grades with a capacity of 150 - 300 kg/h. Although the physical properties of the fertilizers thus manufactured are not directly comparable with the plant products, comparison between the products which are manufactured there is reliable.

As an example of the kind of ongoing research, the effect of the following factors on quality have been studied at the pilot plant installations:

1. Process
2. Production conditions
3. Various differing raw materials
4. Coating

7.2 Research and testing methods

7.2.1 Moisture

The presentation deals with the physical properties of fertilizers, nevertheless, moisture analysis is of the utmost importance also.

Several properties of a fertilizer depend on its moisture or, rather, free-water content. Moisture can be analyzed by many different methods, none of which seem absolutely suitable for NPK fertilizers! For the manufacturer, the most important aspect is to maintain a high physical quality, leaving the analyzed moisture results second most. Customers often specify minimal moisture content, even under 0.3% H_2O. However, such a requirement is unnecessary if the quality of the product is high enough. Furthermore, one must take into account the extensive deviation in methods. (Figure 10).

7.2.2 Testing of caking in research

For the sake of comparability, the same methods are used in research as in routine testing at the plants.

However, caking is also studied in more detail, for example by the following methods and dependences:

	Method	Measure
1.	Cylinder method	Strength
2.	Microbag 100 g	Caking per cent
3.	Minibag 4-10 kg	- " -
4.	Bag test 40-50 kg	- " -
5.	Pallet test	- " -

Dependences

1. Pressure
2. Time
3. Moisture
4. Temperature
5. Raw materials
6. P_2O_5 water solubility
7. Process

For examination of the results, experience is very important and ensures that most tests are practicable.

7.2.3 Microscope studies

In the quality development of fertilizers microscope studies are valuable and important methods. Kemira uses a Cambridge scanning electron microscope with which the surfaces and internal structures of granules can been seen quite readily.

In addition to electron microscope studies, optical microscopes are in daily use to study thin layer microscope images and the surfaces of granules.

Thin layer microscope technique, together with SEM, is an indispensable method in developing quality products which are caking resistant and dust-free.

7.2.4 Smoothness of coatings

Nearly all fertilizers have some caking tendency without coating. By means of applying a smooth coating it is possible to attain a high quality finish in most cases. The most usual coating material is oil amine, which is normally used at a ratio of approximately 200 g/t amine + 1 kg oil. For quality's sake, it is important that the coating is spread evenly over the surface of the granule. The evenness of the coating can be studied by marking the coating in such a way that it fluoresces under UV light.

Technically, a smooth coating can be achieved under the following conditions:

1. Smooth granule surface
2. Correct product temperature
3. Correct spraying application
4. Sufficient time in the drum

A good coating on a good granule assures optimum protection and in most cases, even a poorer quality product can be improved.

7.3 Secondary coating

Sometimes after bulk storage, the quality of a product may require that it be retreated in order that it may be fit for delivery. Thus the quality can be significantly improved if the product is coated for the second time. Secondary coating can be carried out by re-cycling the material through the plant or by arranging a so-called secondary coating drum. In order for it to be successful, the coating must be carried out thoroughly.

8 FUTURE

The future competition for markets will be still tougher. Many companies have already made large investments in quality development - as a result the customers come to expect better and better fertilizers.

Nearly all of the testing methods of physical quality used by the various manufacturers today are still customized and the results are only seldom comparable with those of other manufacturers.

This is a waste of resources and often reflects a misunderstanding of the competitive situation since the testing methods are seldom secret. In future, it would be wise to co-operate, compare testing methods, results obtained by them and ultimately agree upon common methods. The benefits of such co-operation would also be past on to the customer.

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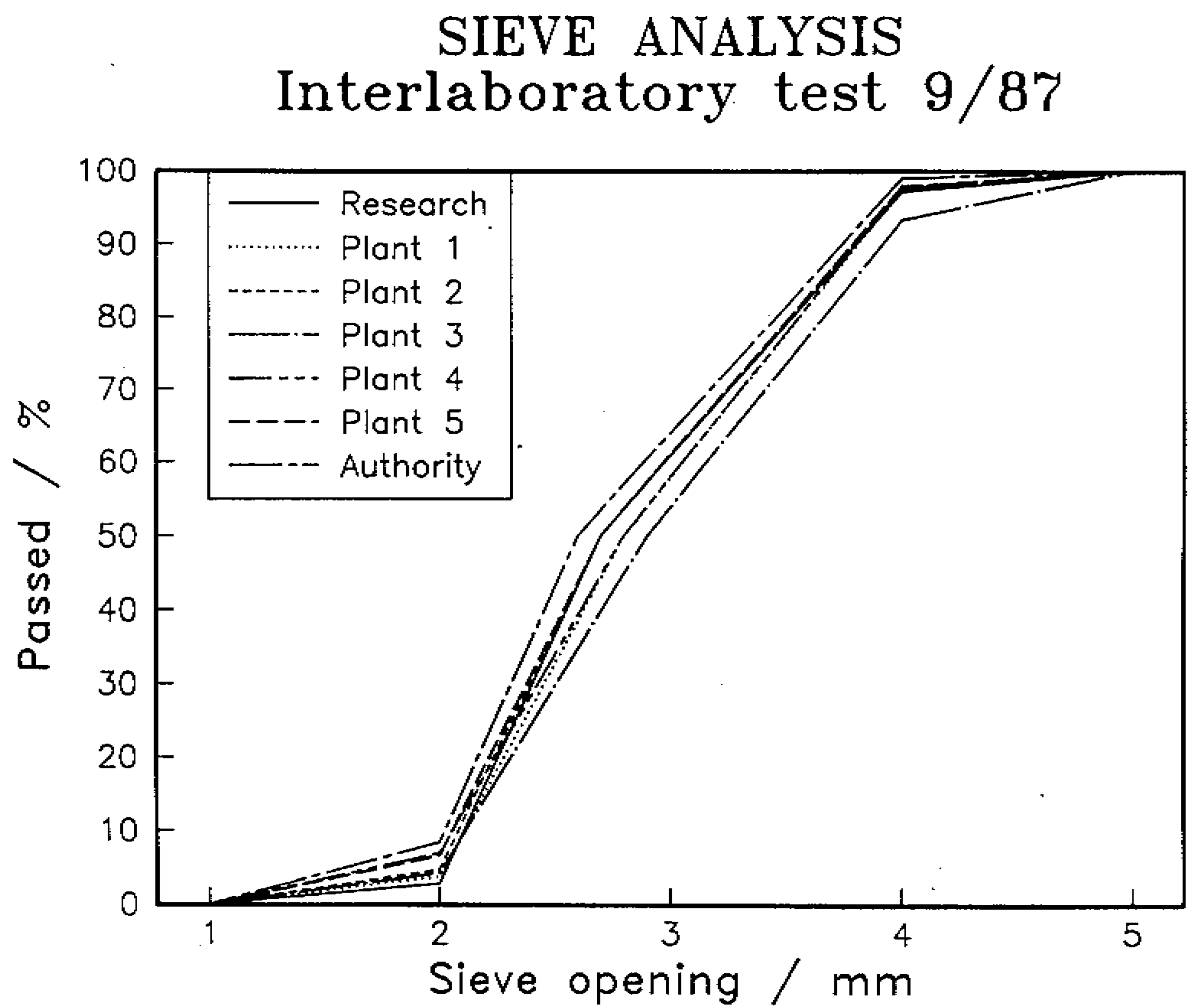
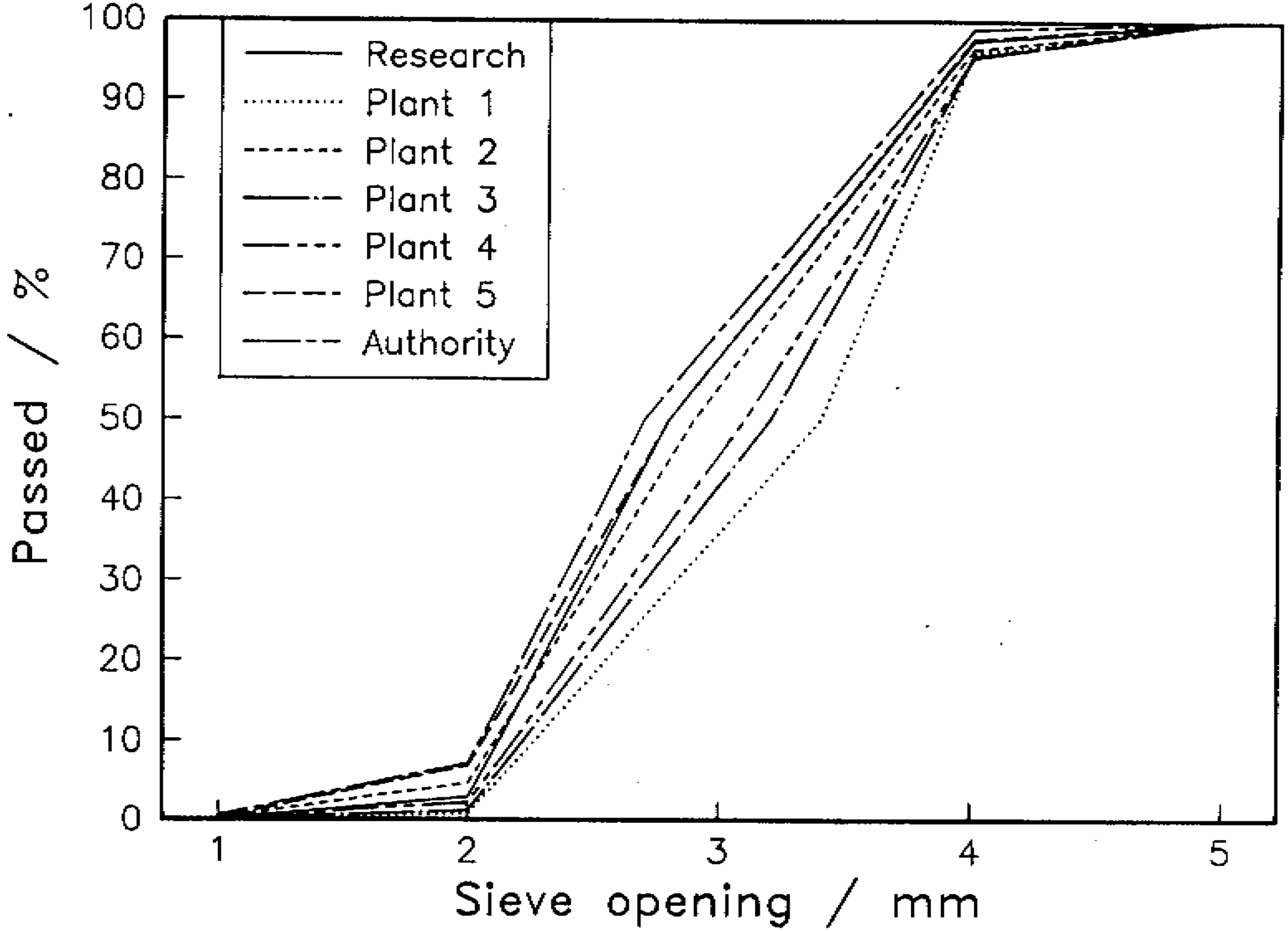
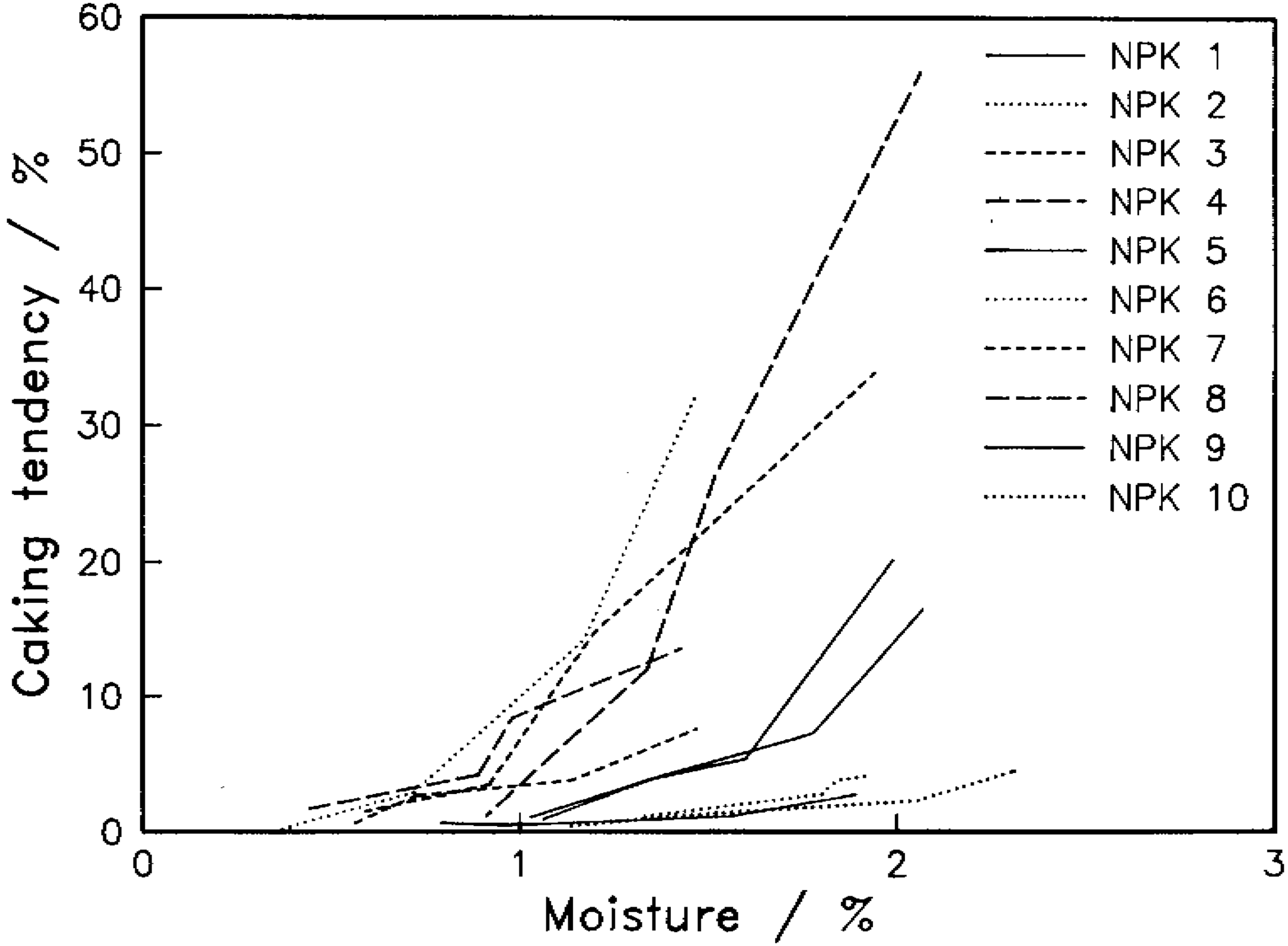

SIEVE ANALYSIS
Interlaboratory test 9/87
Research
Plant 1
Plant 2
Plant 3
Plant 4
Plant 5
Authority
Passed / %
100
90
80
70
60
50
40
30
20
10
0
1
2
3
4
5
Sieve opening / mm

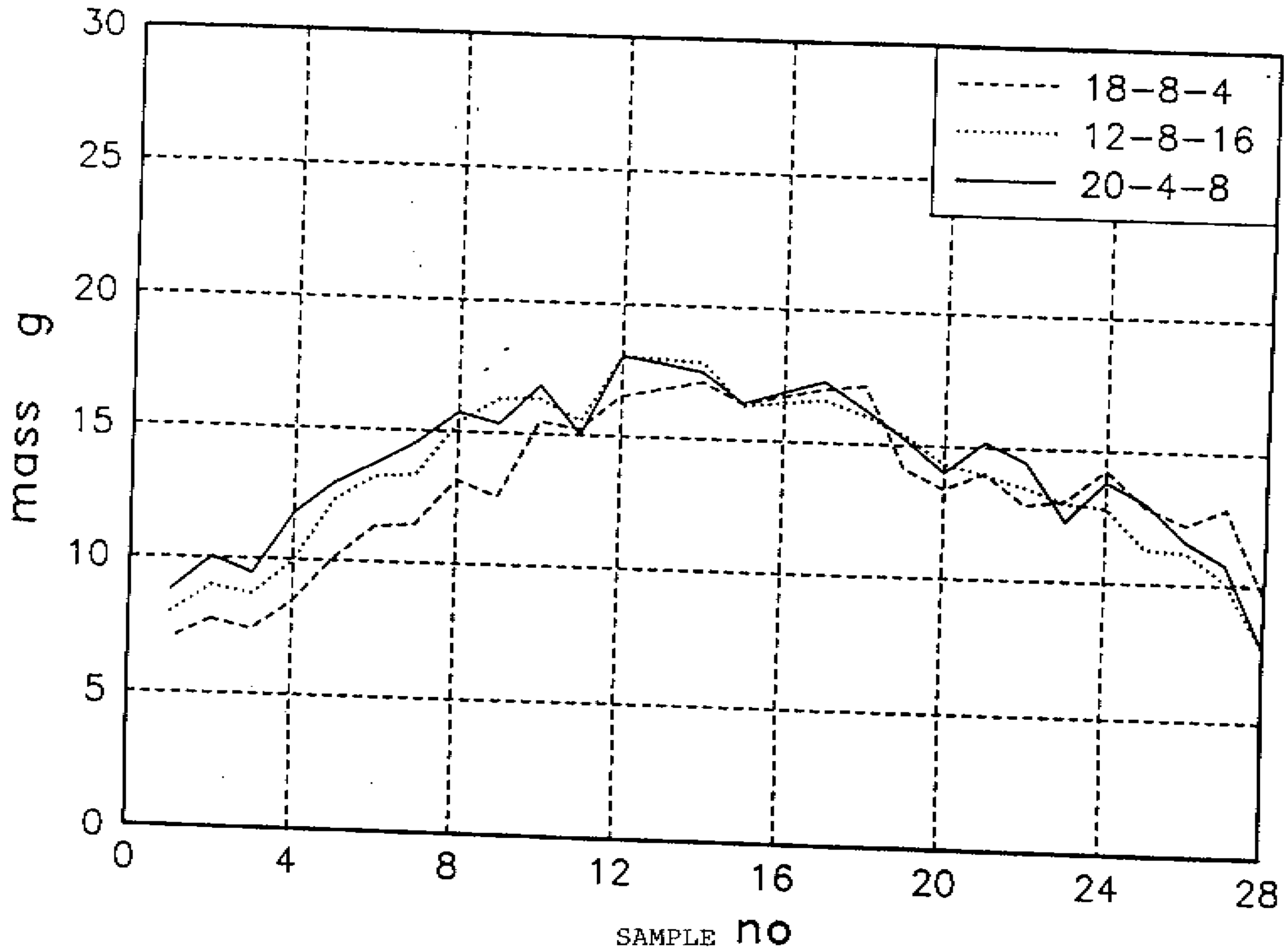
SIEVE ANALYSIS
Interlaboratory test 10/87



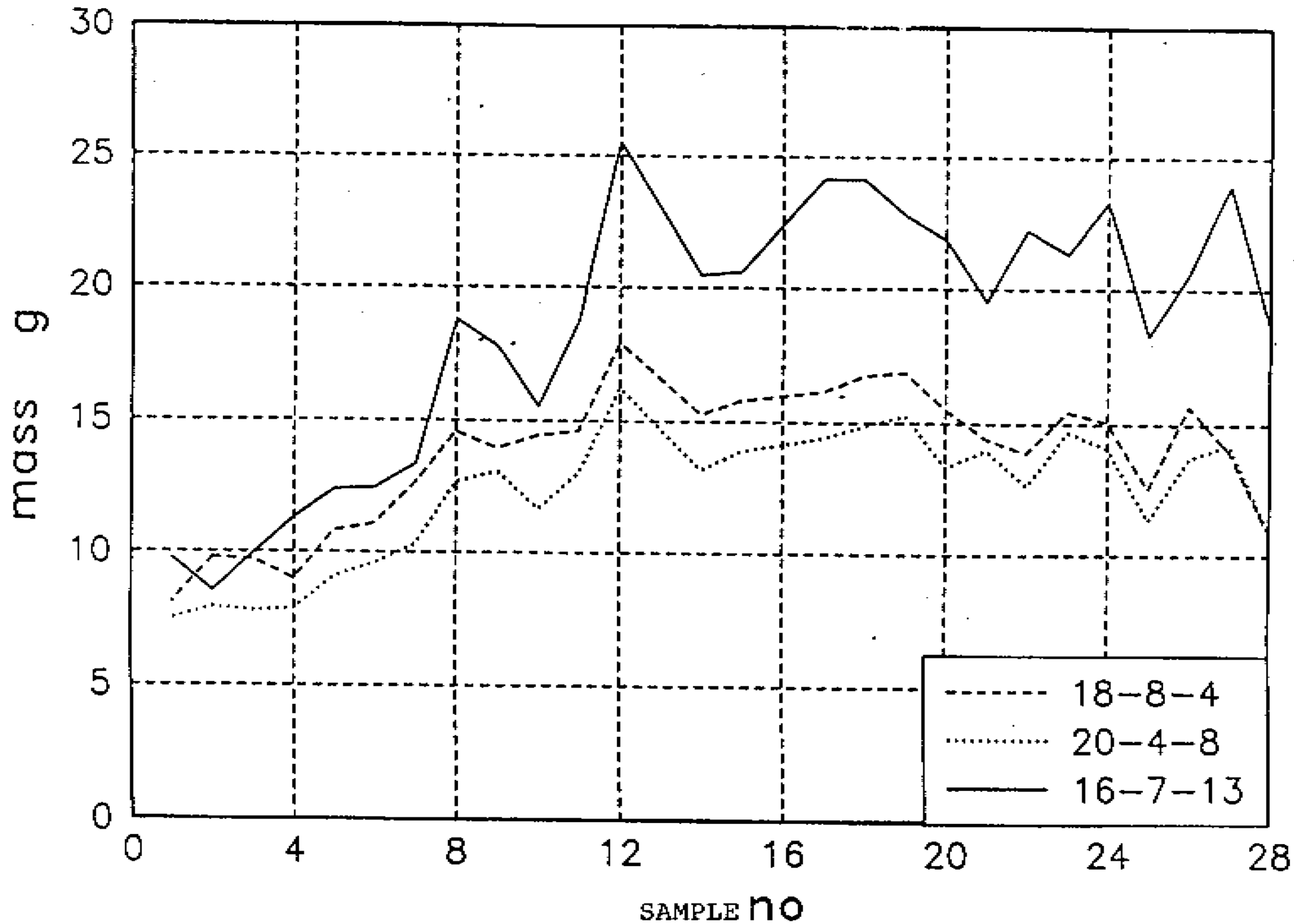
CAKING TENDENCY

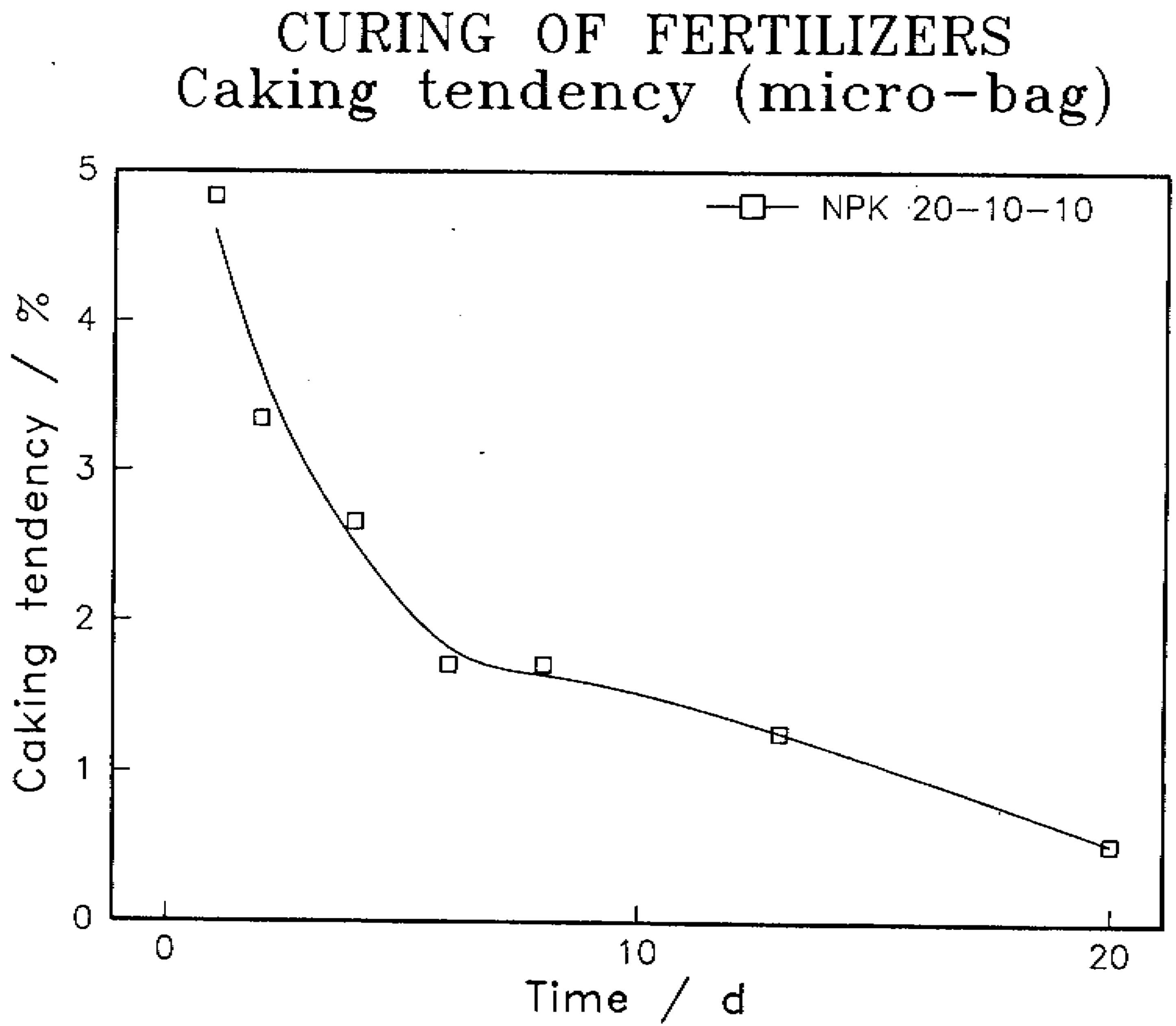


SPREADING PATTERN, NORMAL GRANULES



SPREADING PATTERN, BIG GRANULES




CURING OF FERTILIZERS
Caking tendency (micro-bag)
NPK 20-10-10
Caking tendency / %
Time / d
0
1
2
3
4
5
0
10
20

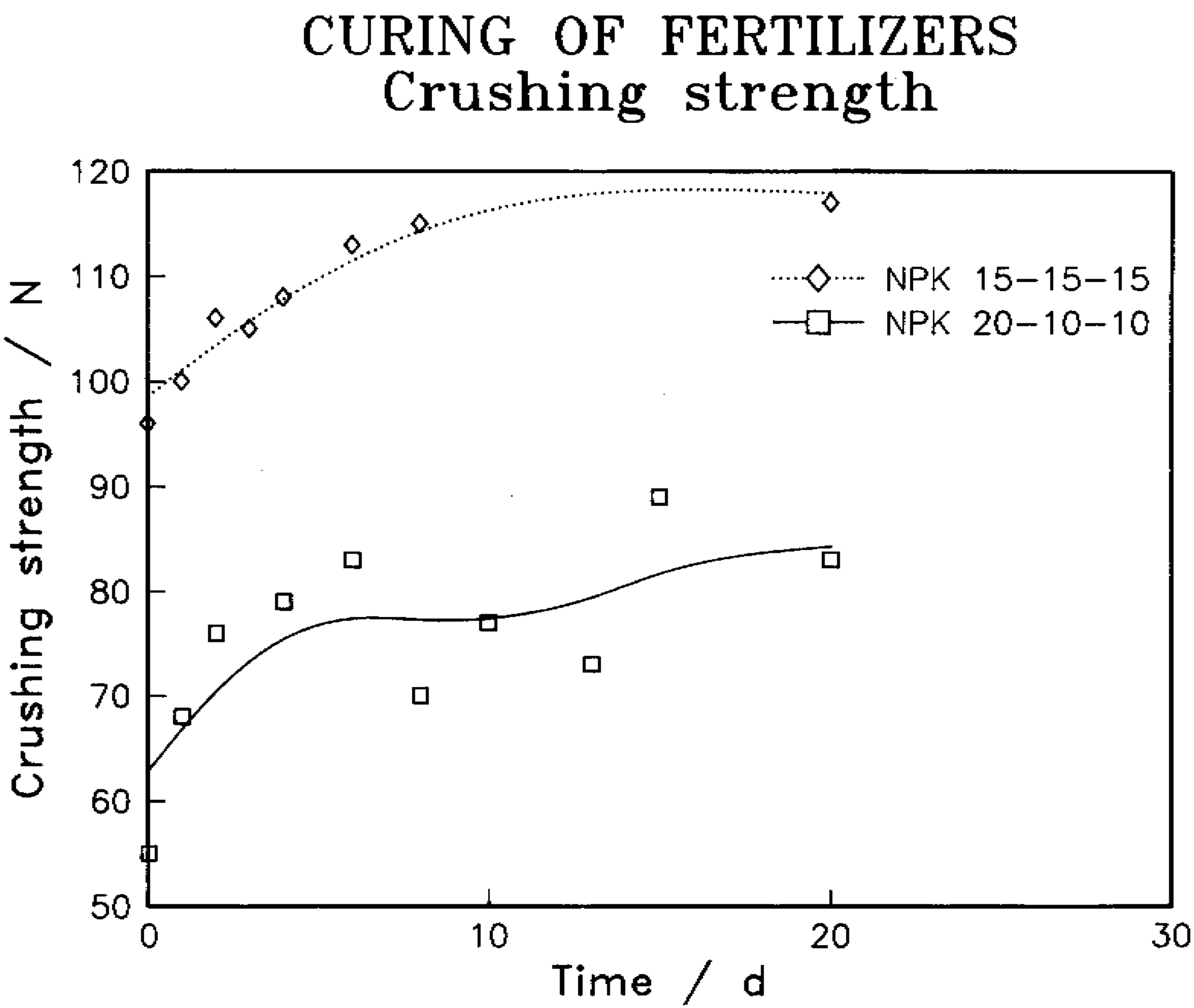
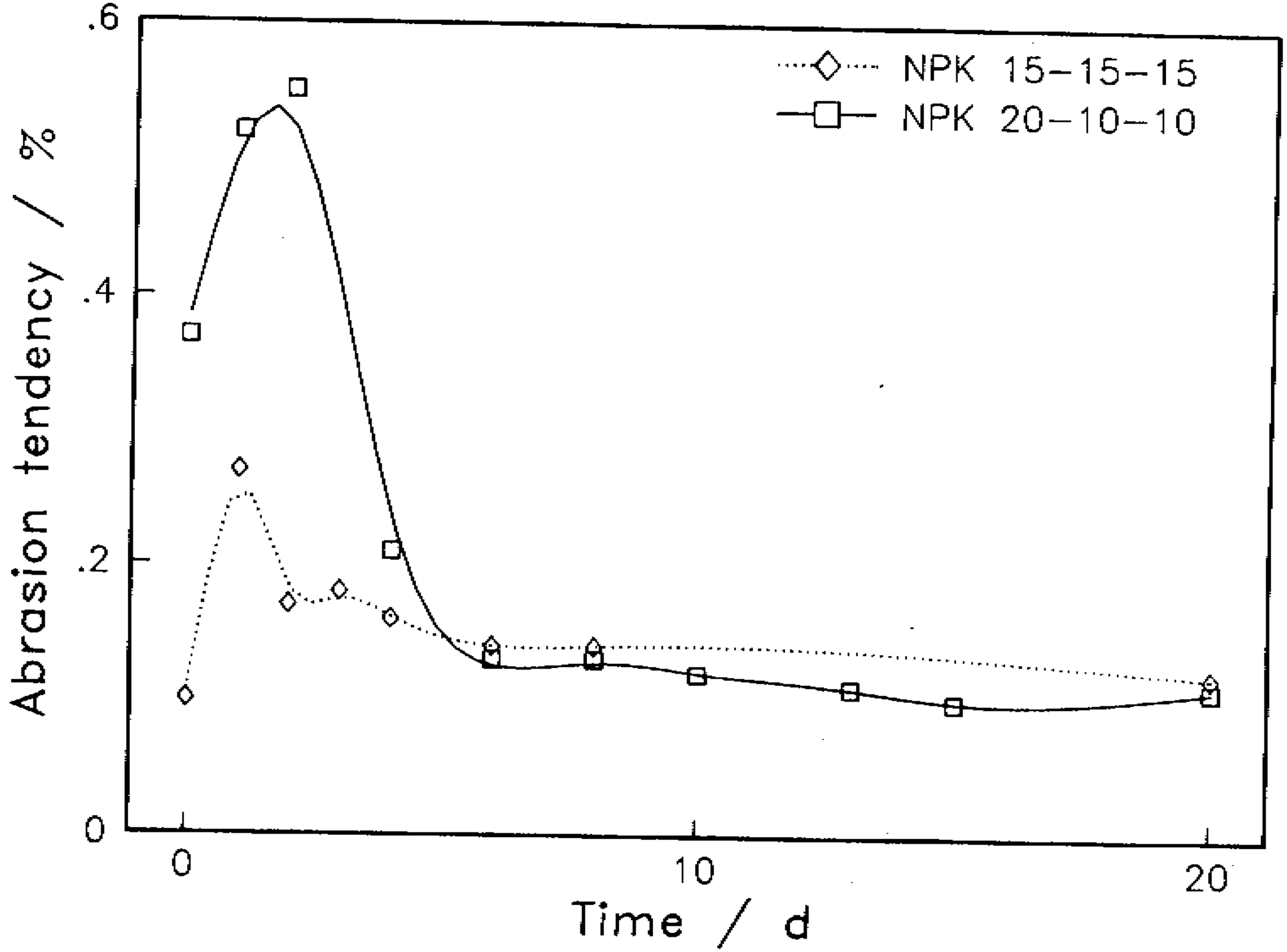
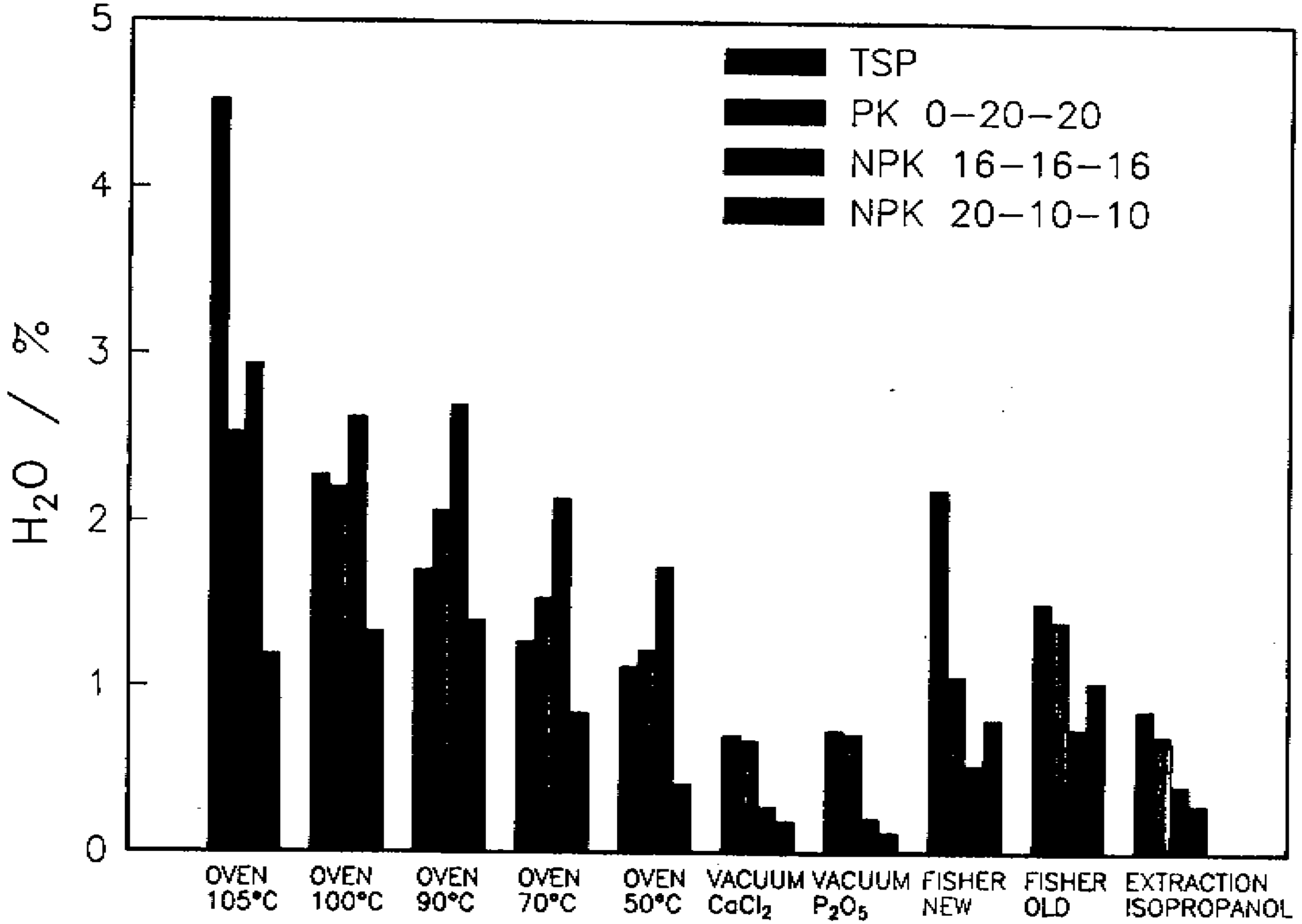

CURING OF FERTILIZERS
Crushing strength
Crushing strength / N
Time / d
NPK 15-15-15
NPK 20-10-10
120
110
100
90
80
70
60
50
0
10
20
30

Figure 9

CURING OF FERTILIZERS
Abrasion tendency



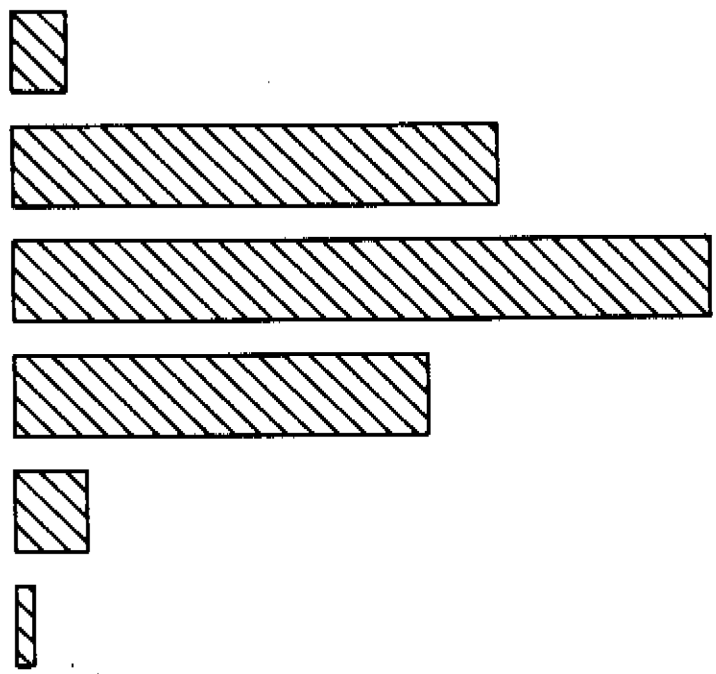
HUMIDITY ANALYSIS OF FERTILIZERS



KEMIRA OY
PARTICLE-SIZE ON LINE ANALYSATOR

SIZE FRACTION	%
<1.0 mm	0
1.0-1.4 mm	0
1.5-1.9 mm	3
2.0-2.4 mm	27
2.5-2.9 mm	39
3.0-3.4 mm	23
3.5-3.9 mm	4
4.0-4.4 mm	1
4.5-4.9 mm	0
>5 mm	0

DISTRIBUTION



PRODUCTION SIZE 2.0-4.0 mm

93

SHAPE COEFFICIENT 69 133178 PARTICLES MEASURED

TA/88/2 Physical quality of fertilizers by H. Hero, Kemira Oy, Finland

DISCUSSION (Rapporteur Mr. J.M. BIRKEBAEK, Superfos Fertilizers, Denmark)

Q - Mr. K. GOVINDARAJAN (Southern Petrochemical Industries, India)

1/ Have you experienced any caking of fertilizers with a granule strength of 50 N2 ? What is the minimum allowable granule strength to avoid caking in case of DAP ?

2/ Kindly provide details of granule size analyser for the on-line analysis of granule size distribution.

A - 1/ Caking can be observed even with fertilizers with crushing strength higher than 50 N as other parameters, for example moisture, can have influence on caking tendency. It is our experience though that crushing strength must be higher than 50 N to ensure free flowing properties.

Regarding DAP, the crushing strength must be higher than 25 N. Otherwise problems will arise during handling, but we have no particular experience in production of DAP.

2/ The samples are taken from the recycle lines and we take continuous pictures with TV cameras. By analyzing the pictures, we can calculate the size distribution and the shape coefficient and even the history of the granulation.

Q - Mr. G. KONGSHAUG (Norsk Hydro, Norway).

As a daily routine, you recommend 4 physical test samples per day. Is this frequent sampling routine caused by process variations, and why do you include the caking test in this routine ?

A - To ensure the quality and avoid variations in the product, we have to make a lot of tests, among other reasons because we have very frequent changes of grades. We have sufficient capacity in our laboratories to make this large amount of tests.

Q - Mr. D. BELLIS (SICNG, Greece).

1/ Is Kemira Oy using another method than the UV-lamp for control of the coating during the production, and if so, which method ?

2/ Which material is Kemira Oy using for control of the coating ?

A - 1/ We only use the UV-lamp method for control of the coating in the production.

2/ Anticaking materials used at Kemira are normal commercial products (with or without amine) and talc.

Q - Mr. RAHOUI (SAEPA, Tunisia).

One of the factors which increase the caking tendency is humidity. How do you estimate the impact of respectively free and bound water on the caking tendency ?

A - First, we do not know what is free water and what is bound water in the fertilizer, because we often find it impossible to determine it.

.../.../...

It is well known that impurities do affect the caking tendency, but who knows how the impurities bind water. As an example, I can tell that we have found grades containing 2% of water and being free flowing, whereas in other cases the same grades containing only 0.6% of water (determined by the same method) have been caked.

I think it is a difficult question to everyone in the fertilizer production how to choose the right water content when varying the raw materials.

Q - Mr. ORFANIDIS (Duetag, France).

Can you please identify the fertilizer grades shown on Fig.4 of your presentation (caking tendency related to moisture).

A - Number 1 - 5 are 16-16-16
Number 6 - 10 are 20-10-10

In all cases water soluble P205 was 70-75%.

Q - Mr. Roy HUTCHINS (Texasgulf Inc., USA).

You showed 10 samples of NPK in comparing caking tendency with moisture content, with markedly different caking tendencies. How do you explain the difference in the caking tendencies ?

A - Even though the same grades were set to absorb moisture under the same conditions, we see a marked difference in absorption tendency. One reason for that is difference in porosity. For example, we often find a higher porosity in grades produced in a pugmill than in the same grades produced in a spherodizer.

Q - Mr. A. BENMANSOUR (SAEPA, Tunisia).

Regarding sampling for control of the physical or chemical quality of a fertilizer, please indicate the following:

- automatic or manual sampling
- frequency
- sampling methods.

How has the question of sampling been solved ? Which influence has the sampling on the results of the control ?

A - We have automatic sampling systems in production lines and in bagging stations. The samples are taken every 30 min. and collected, which means that we do not rely on random samples.

Comparison between these collected samples and random samples shows only small variance.

According to the Finnish fertilizer law every bag must have a chemical analysis within certain limits from the declaration of the fertilizer.

Q - Mr.A. HAMDI (ICM-SAEPA, Tunisia)

Can the secondary coating of a product take place at ambiant temperature ? or is it necessary to recondition the product ?

.../.../...

A - We normally do not deliver products which have shown caking tendency. Secondary coating is, however, a good idea, and it should be applied at ambiant temperature as heating and cooling again is an economical obstruction.

Q - Mr.P. ORFANIDIS (Duetag, France).

In relation to my previous question , can you please give some additional information about the processes in producing 16-16-16 with such dramatic caking tendency (process, conditions, raw materials, other physical characteristics).

A - The grade 16-16-16 is quite "full". The physical quality of this grade depends much on the phosphoric acid, i.e. with very pure acid the caking tendency is high.

With different potashes the quality varies a lot.

Normally caking tendency, dust and crushing strength go together. The very fast indicator for caking tencendy is the drop in crushing strength.

<u>Questions put in writing:</u>

Q - Mr.N. GAURON (Norsk Hydro Azote, France).

My question refers to Fig. 7-9 in your paper. It is here shown that the curing of NPK fertilizer should have a positive impact on the physical properties and in particular on the caking tendency.

Have you registered the same positive impact on dusting properties or dust formation?

A - The dusting properties go often together with the caking tendency. Sometimes there is developed some dust during the curing time, but after the final screening, it is very low. When stored in a dry place, the dust formation is about nothing.

Q - Mr. R. HUTCHINS (Texasgulf Inc., USA).

Were the tests relating caking tendency to moisture content with 10 samples of two grades of NPKs run under constant humidity conditions ? What were the test conditions ? Granted that differences in physical properties could affect caking between samples of the same grade. Were there chemical differences as well which could have contributed to the differences within the grades ?

The comparison of H20 content by various methods did not distinguish between "bound" and "free" water. Would not some of the procedures measure both and some only "free" water and some part of the "bound" and all of the "free" water ?

A - The samples were originally from the the delivery (0 level). Then, in the research, the absorbed moisture in constant conditions, i.e. + 25° C and 70% relative humidity. Between the samples the main chemical differences were small, the differences within the grades were more in the physical properties and the granulation methods.

.../.../...

From H_2O analysis, it is really difficult to say what is "free" and what is "bound" water. We prefer the extraction method to give free water results, but anyway the best method to "get back" the added water has been the oven method.

Water is bound in and on the particles by many different ways and I do not see any really reliable method to give absolutely right values.

Q - Dr. R. NITZSCHMANN (BASF, Federal Republic of Germany).

What methods do you use to get a representative sample for automatic size-distribution measurement or automatic analysis ?

How large are these samples ?

A - The sampler is quite normal equipment which takes samples from the falling stream by cutting the stream (from conveyor belt down to another). The size of the sample for the particle size analyzer is about 1.1 kg.