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Fertilizers — Comparison of the CEN/TC 260/WG 7 ring test results with tolerances given in the Regulation (EC) Nr 2003/2003 Annex II and conclusions

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Engrais - Comparaison des résultats des essais interlaboratoires menés par le CEN/TC 260/WG 7 avec les tolérances données dans le règlement (CE) n°2003/2003 Annexe II et conclusions

Düngemittel - Vergleich der Ringversuchsergebnisse der CEN/TC 260/WG 7 mit den in der Verordnung (EG) Nr. 2003/2003 Anhang II angegebenen Toleranzen und Schlussfolgerungen

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Foreword

This document (CEN/TS 16490:2013) has been prepared by Technical Committee CEN/TC 260 “Fertilizers and liming materials”, the secretariat of which is held by DIN.

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Introduction

Following a request from the European Commission (Mandate M/418), an evaluation was done of the existing tolerances as per Regulation (EC) No. 2003/2003. Input for this evaluation was derived from the precision data obtained via the several ring tests that have been made according to Mandate M/335.

As to exclude eventual interference from effects due to inhomogeneity of physically blended grades, no blends have been included in the ring tests; test samples have been limited and chosen as to be representing some main product grades sold within the EU.

The statistical evaluation has been done in line with ISO 5725-2 and whenever deemed necessary for appropriate evaluation and interpretation of the test results, some extra statistical evaluation was made on the test data.

As to judge to what extent problems arise in case of enforcement controls, some field data have been analysed as to verify to what extent actual conflicts exist in between test results from official controls and applied tolerances.

The evaluation revealed no real need for adjustments to tolerances as they relate to analytical variability. However, it should be emphasised that the tolerances given in Regulation 2003/2003 relate not only to analytical variability but to the total variability including allowances for sampling error and product variability.

Finally if the European Commission sees the necessity of further method improvements, then one could consider a project entitled to develop an alternative method. Only following a full evaluation including ring testing, a final judgment can be made if the newly developed method could be a candidate for replacement of the existing one(s) assuming better accuracy data.

1 Scope

In Regulation (EC) No. 2003/2003 [2] tolerance limits are mentioned for nutrient contents in mineral fertilizers (Annex II of Regulation (EC) No. 2003/2003) as well as prescribed methods for control purposes (Annex IV of Regulation (EC) No. 2003/2003).

Prior to the work done by CEN/TC 260 following Mandate M/335, no statistical data were available for the official analytical methods to be applied. Due to the standardization work done for this mandate, statistical data have been generated as ring testing was a major topic in this mandate.

This Technical Specification describes to what extent the presently applied tolerances are in line with the obtained precision data from the analytical methods studied.

The purpose of this document is to give feedback on the applied tolerances within Regulation (EC) No. 2003/2003 based on the method evaluation done as an outcome of the work executed by CEN/TC 260/WG 7 according to Mandate M/335. This evaluation of the tolerances was part of Mandate M/418.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 5725-1:1994, *Accuracy (trueness and precision) of measurement methods and results — Part 1: General principles and definitions*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1
tolerance (T)
variation including manufacturing, raw materials, sampling and analytical methods

3.2
repeatability limit (r)
the value less than or equal to which the absolute difference between two test results obtained under repeatability conditions may be expected to be with a probability of 95 %

[SOURCE: ISO 5725-1:1994]

Note 1 to entry: In other words, r is the minimum difference between two results in order to be statistically different, under repeatability conditions, at a 95 % probability level.

3.3
reproducibility limit (R)
the value less than or equal to which the absolute difference between two test results obtained under reproducibility conditions may be expected to be with a probability of 95 %

[SOURCE: ISO 5725-1:1994]

Note 1 to entry: In other words, R is the minimum difference between two results in order to be statistically different, under reproducibility conditions, at a 95 % probability level.

4 Statistical method validation

4.1 General

European laboratories were involved, including as well private, industry, official as commercial laboratories.

ISO 5725-2 was applied as standard for ring testing and statistical evaluation of its results.

In general, legislative tolerances are given to accommodate variability in raw materials, manufacturing, sampling and analytical routines.

4.2 Approach taken by CEN/TC 260/WG 7

4.2.1 Product selection

The European market is characterised by a broad portfolio of different types of fertilizer grades, Hence there was a need for selection of a limited number of products to be tested for the ring-test(s). Aim during the selection process was to get fertilizer samples tested reflecting some main grades sold in the EEC.

4.2.2 Samples and their preparation

In order to be able to evaluate the precision data of the method as such, samples have been taken to the utmost extent as a spot (not agglomerated) sample out of a bulk production. This was done on purpose as to reduce the possible variation originating from raw materials, manufacturing and sampling procedure. Furthermore, all samples have been ground before distribution to the participating labs. Only solid, homogeneous samples and liquid samples have been used; no blends. The aim was to work with a limited number of samples, representing the main fertilizer grades applied.

4.2.3 Laboratories involved

The participation of the laboratories was on a voluntary basis, enabling as well private and industry, official and commercial laboratories to contribute. As far as the lab proficiency concerns, the ring test set-up did not require a familiarisation step for each of the individual participating laboratories nor for the methods evaluated. The chosen approach does not imply any assurance on the degree of experience and routine of the labs involved with the method under investigation and reflects the day-to-day situation as is.

4.2.4 Data evaluation

As already stated, ISO 5725-2 has been applied as the standard for the statistical data evaluation. However, for some methods a more in depth approach has been made, with the support of an expert statistician, due to the fact that the reproducibility parameters seemed to be in conflict with the existing tolerances. Based on this refined statistical analysis, it has been the intention to come up with adequate tolerances.

As stated in [1], R should be ≤ 0,7 T. In case this condition is not fulfilled, one should from a theoretical perspective considering the following alternative options:

— improve the method of analysis;

— propose an alternative method with better performance;

— increase the tolerances.

4.3 Statistical data: results versus tolerances

The ring test data and findings are given in the informative Annex A.

4.4 Refined statistical analysis

Despite the method refinements to the determination of water soluble sulfur and water soluble calcium, reproducibility figures could not be improved substantially. Therefore, a more in-depth statistical analysis has been performed by an expert statistician. The main principles are described below; the detailed information can be consulted in Annex B.

In a first step, after applying the classical criteria for removing outliers of a population of data (see ISO 5725-2), it could be concluded that the reproducibility data were in conflict with the existing tolerances.

A substantial number of test results show high R values which implies that the methods only comply with tolerance substantially higher than the existing ones. In order to improve these reproducibility data, data sets outside the 99,9 % confidence interval (after removal of outliers according to Grubb's and Cochran's tests) have been removed. The s_R calculated from the remaining data is used to find a more adequate tolerance value and at the same time comply with the performance of the method.

The same exercise has been performed for the total sulfur content.

Further, the remaining data population has been crosschecked with the official tolerances. However, still conflicts were observed.

In a final step, it has been attempted to derive, based on statistical principles, reasonable tolerances for the concerned nutrients.

4.5 Field data

In order to cross-check the outcome of the statistical evaluation of official methods versus tolerances in place and verify the necessity for eventual adjustment of the existing tolerance limits, a population of obtained anonymised test results have been evaluated. The test results submitted were data derived from official controls (enforcement) and covering data from nutrient analysis on various fertilizer types such as: calcium ammonium nitrate (CAN), CAN+MgO and NPK. In total about 450 analyses were presented, covering analysis of Nitrogen, Sulfur, Magnesium and Phosphorus.

Sampling as well as analyses has been conducted according to EC methods.

These data have only been used to check whether problems arise in case of enforcement controls or not. The enforcement data have not been evaluated statistically on repeatability or reproducibility. Neither have they been used to evaluate the precision or correctness of the data obtained with CEN methods.

Tables 1 to 3 summarise data for fertilizer types that were numerously represented.

Table 1 — Product type: CAN27

Component	Declared value %	Number of analyses	Deviation from declaration %	Out of tolerance
N total	27	30	-0,4 to +0,6	No
N NH_4	13,5	30	-0,4 to +0,5	No

Table 2 — Product type: CAN27 + MgO

Component	Declared value %	Number of analyses	Deviation from declaration %	Out of tolerance
N total	27	20	-0,5 to +0,6	No
N NH_4	13,5	20	-0,6 to +0,5	No
MgO	4	20	-0,04 to +0,4	No
MgO (ws)	1 to 1,8	20	-0,96 to +1,0	Yes

Table 3 — Product type: NPK containing sulfur

Component	Declared value %	Number of analyses	Deviation from declaration %	Out of tolerance
N total	15 to 16	12	-0,2 to +0,8	No
S	2 to 4	9	+0,3 to 2,31	No
S (ws)	1,6 to 3,2	10	-0,3 to +1,4	No
P_2O_5 (citrate)	15	10	-1,3 to +0,7	No

As can be seen from Tables 1 to 3, about 30 samples of CAN have been analysed and no exceeding of the tolerances was detected for the nitrogen content.

For the 20 samples of CAN + MgO that have been analysed no exceeding of tolerances for nitrogen and magnesium was found, except for MgO water soluble where a few deviations were identified.

About 15 samples of S containing NPK showed no exceeding of tolerances, neither for nitrogen, sulfur or phosphorus.

Based on these data, one can conclude that there is no urgent need for adjustments to tolerances, however it is obvious that deviation from declared values are larger for MgO and S compared to those for nitrogen.

5 Conclusions

In the frame of the mandate from the European Commission, and given the working conditions of WG 7, ring tests have been performed using a limited number of samples, representing the main fertilizer grades on the market. However, no physically blended grades have been used in the ring tests. Possible effects due to inhomogeneity of physically blended grades are therefore not reflected in the statistical evaluation of the various methods.

Based on evaluation of precision data there is in general no direct need for adaption of the existing tolerances as they relate to analytical variability (see Introduction). However, some discrepancy between the tolerances and the statistical data of the ring tests has been revealed. It concerns mainly the determination of calcium and magnesium, in particular related to the water soluble part. To a lesser extent also the determination of sulfur and more specifically the water soluble (ws) part, is concerned.

Therefore we do not recommend changing the applied tolerance limits from Regulation (EC) No 2003/2003 to the methods that have been evaluated, with exception of the nutrients mentioned above. The evaluation of a data set of enforcement controls seems to support this conclusion. However, since analytical variability is shown to account for the entire tolerances in Regulation 2003/2003, consideration must be given to increasing the individual tolerances to take account of sampling and product variability.

Regarding the method for determination of water soluble calcium, WG 7 has been exploring several possibilities to improve the accuracy of this method. Nevertheless, no major progress has been achieved in improving especially the reproducibility. Also a bench-mark with other legislative frames did not reveal better and alternative methods, as the water soluble calcium is not incorporated in non-European regulation.

As far as the nutrients water soluble calcium and magnesium, total sulfur and water soluble sulfur are concerned, a more in depth statistical evaluation has been made revealing that there are still conflicts between the analytical results and the official tolerances.

Nonetheless, based on the refined data population and the back calculation of reasonable tolerances, it is recommended to introduce the following adjustments to the officially applied tolerance limits:

— Sulfur (total and water soluble): 1,2 to 1,5;

— Calcium and Magnesium (water soluble): 1,2 to 1,5.

Although the proposal above is an improvement to the existing situation, and given the inherent problems of the method of analysis and certain matrices, the proposed tolerance still does not cover completely the variability originating from sampling, manufacturing and analysis. Nevertheless, considering the cross-check with the field data, the proposed tolerance levels should be capable to take into account the full variability.

The proposed adjustment to the calcium tolerance still reveals possible conflicts for products with high calcium content, i.e. CAN 27.

Method 2.6.2 (EN 15750) for the determination of different forms of nitrogen in fertilizers containing nitrogen only as nitric, ammoniacal and urea nitrogen, such as UAN + S, does not perform well enough. Therefore, two alternative methods have been tested and have been proposed to replace method 2.6.2. It is also recommended to adjust the existing tolerance levels from 0,6 % up to 1,0 %.

However, an extra option could be to consider some further evaluation and development work regarding an alternative method for determination of the concerned nutrients, i.e. water soluble sulfur, calcium and magnesium. The nature of this work would be more in the field of method development at first instance, whereas a next step would be the launch of a ring test to define its precision data. Based on the evaluation of its statistical data, it should be possible to judge whether the alternative method is suitable for integration in the EU regulatory frame.

Annex A
(informative)

Statistical data of the inter-laboratory tests and findings

A.1 General

Tables A.1 to A.32 show the accuracy data compared to tolerances for the methods given in Annex IV of the Regulation (EC) No. 2003/2003,

where

$\bar{x}$ is the mean value (mass fraction);

s_r is the repeatability standard deviation (mass fraction);

RSD_r is the relative repeatability standard deviation;

r is the repeatability limit (mass fraction);

s_R is the reproducibility standard deviation (mass fraction);

RSD_R is the relative reproducibility standard deviation;

R is the reproducibility limit (mass fraction).

A.2 Nitrogen

Table A.1 — Determination of ammoniacal nitrogen (EC method 2.1, EN 15475)

Sample	Sample type	Number of labs	Number of labs retained after elimination of outliers	$\bar{x}$	s_r	RSD_r	r	s_R	RSD_R	R	Current EC Tole-rance
AN 33,5	Solid	22	18	16,67	0,05	0,29	0,13	0,23	1,36	0,63	0,6
CAN 27	Solid	24	24	13,53	0,05	0,36	0,14	0,14	1,02	0,39	0,8
NPK1 (14-8-24+8S)	Solid	24	23	8,38	0,03	0,38	0,09	0,12	1,43	0,34	1,1
NPK2 (16-16-8+4S)	Solid	24	21	10,02	0,03	0,28	0,08	0,16	1,59	0,45	1,1
NP (DAP)	Solid	24	21	17,64	0,04	0,20	0,10	0,23	1,31	0,65	1,1

Table A.2 — Determination of nitric and ammoniacal nitrogen according to Devarda (EC method 2.2.3; EN 15476)

Sample	Sample type	Number of labs	Number of labs retained after elimination of outliers	$\bar{x}$	s_r	RSD_r	r	s_R	RSD_R	R	Current EC Tole-rance
AN 33.5	Solid	21	18	33,45	0,04	0,13	0,12	0,28	0,83	0,77	0,6
CAN 27	Solid	19	16	26,97	0,06	0,23	0,17	0,22	0,80	0,60	0,8
NPK1 (14-8-24+8S)	Solid	23	22	14,14	0,04	0,31	0,12	0,22	1,58	0,63	1,1
NPK2 (16-16-8+4S)	Solid	23	21	16,04	0,04	0,23	0,10	0,21	1,33	0,60	1,1
NP (DAP)	Solid	21	19	17,70	0,05	0,30	0,15	0,18	1,03	0,51	1,1

Table A.3 — Determination of total nitrogen in urea (EC method 2.3.3; EN 15478)

Sample	Sample type	Number of labs	Number of labs retained after elimination of outliers	$\bar{x}$	s_r	RSD_r	r	s_R	RSD_R	R	Current EC Tole-rance
Urea	Solid	19	16	46,26	0,11	0,3	0,24	0,27	0,57	0,74	0,4

Table A.4 — Spectrophotometric determination of biuret in urea (EC method 2.5, EN 15479)

Sample	Sample type	Number of labs	Number of labs retained after elimination of outliers	$\bar{x}$	s_r	RSD_r	r	s_R	RSD_R	R	Current EC Reg.
Urea	Solid	13	11	0,81	0,01	1,7	0,04	0,07	8,9	0,20	Max 1,2
Urea + S	Solid	9	6	0,76	0,02	2,0	0,05	0,14	18,0	0,38	Max 0,9

Table A.5 — Determination of different forms of nitrogen in fertilizers containing nitrogen only as nitric, ammoniacal and urea nitrogen (EC method 2.6.2, EN 15750)

Sample	Sample type	Number of labs	Number of labs retained after elimination of outliers	$\bar{x}$	s_r	RSD_r	r	s_R	RSD_R	R	Current EC Tole-rance
UAN+S	Liquid	11	9	22,76	0,16	0,7	0,45	1,23	5,4	3,45	0,6
UAN+S (repetition)	Liquid	7	7	22,2	0,2	0,9	0,6	1,5	6,9	4,3	0,6

Table A.6 — Determination of different forms of nitrogen in fertilizers containing nitrogen only as nitric, ammoniacal and urea nitrogen (EC method ISO 5315) (new samples)

Sample	Sample type	Number of labs	Number of labs retained after elimination of outliers	$\bar{x}$	s_r	RSD_r	r	s_R	RSD_R	R	Current EC Toler-ance
UAN+S	Liquid	12	12	22,01	0,10	0,5	0,29	0,56	2,5	1,55	0,6

Table A.7 — Determination of different forms of Nitrogen in fertilizers containing nitrogen only as nitric, ammoniacal and Urea nitrogen ("German method using Iron and tin chloride as reducing agent") (new samples)

Sample	Sample type	Number of labs	Number of labs retained after elimination of outliers	$\bar{x}$	s_r	RSD_r	r	s_R	RSD_R	R	Current EC Tole-rance
UAN+S	Liquid	7	7	21,88	0,10	0,4	0,27	0,47	2,2	1,32	0,6

Findings:

EC method 2.1:

— Based on the statistics above, there can be concluded that Method 2.1 performs well, under repeatability as well as reproducibility conditions.

— Considering the statistical data, the definition of the reproducibility limit, as well as the tolerances mentioned in Annex II of EC Regulations 2003/2003 it can be concluded that, despite the good results, difficulties occur for ammonium nitrate with more than 32 % N. The value of R already exceeds the allowed tolerance of 0,6 % for ammonium nitrate with more than 32 % N (Annex II of Regulation (EC) No. 2003/2003), whereas this tolerance should cover variations in manufacturing, sampling and analysis.

EC method 2.2.3:

— Based on the statistics above, there can be concluded that Method 2.2.3 performs well, under repeatability as well as reproducibility conditions.

— Considering the statistical data, the definition of the reproducibility limit, as well as the tolerances mentioned in Annex II of Regulation (EC) No. 2003/2003 it can be concluded that, despite the good results, difficulties occur for ammonium nitrate with more than 32 % N. The value of R already exceeds the allowed tolerance of 0,6 % for ammonium nitrate with more than 32 % N (Annex II of Regulation (EC) No. 2003/2003), whereas this tolerance should cover variations in manufacturing, sampling and analysis as well. Also for ammonium nitrate with less than 32 % (CAN sample) the allowed tolerance of 0,8 % is almost fully consumed by the method of analysis.

EC method 2.3.3:

— Based on the statistics above, there can be concluded that Method 2.3.3 performs well, under repeatability as well as reproducibility conditions.

— Considering the statistical data, the definition of the reproducibility limit, as well as the tolerances mentioned in Annex II of EC Regulations 2003/2003 it can be concluded that, despite of the good results, difficulties occur for urea. The value of R already exceeds the allowed tolerance of 0,4 % (Annex II of Regulation (EC) No. 2003/2003), whereas this tolerance should cover variations in manufacturing, sampling and analysis.

EC method 2.5:

— Based on the statistics above, there can be concluded that Method 2.5 performs well under repeatability conditions but moderately well under reproducibility conditions, especially considering the maximum allowed biuret content of 1,2 %.

EC method 2.6.2 and variants:

— Based on the statistics above, there can be concluded that Method 2.6.2 does not perform well, in particular under reproducibility conditions.

— Two alternative methods, based on ISO 5315 [6], and a German method using iron and tin chloride [7], have been ring tested and perform better. Therefore, it is recommended to replace method 2.6.2 with both alternative methods.

— Considering the statistical data of both the alternative methods, the definition of the reproducibility limit, as well as the tolerances mentioned in Annex II of EC Regulations 2003/2003 it can be concluded that, despite the good results, difficulties occur. The value of R already exceeds the allowed tolerance of 0,6 % (Annex II of Regulation (EC) No. 2003/2003), whereas this tolerance should cover variations in manufacturing, sampling and analysis.

A.3 Phosphate

Table A.8 — Determination of phosphorus extracted in mineral acids (EC method 3.2 after extraction with EC method 3.1.1, EN 15956)

Sample	Sample type	Number of labs	Number of labs retained after elimination of outliers	$\bar{x}$	s_r	RSD_r	r	s_R	RSD_R	R	Current EC Tolerance
NP (DAP)	Solid	17	16	46,51	0,12	0,27	0,35	0,42	0,90	1,17	1,1
NPK (16-16-8+4S)	Solid	20	20	16,13	0,08	0,50	0,21	0,15	0,90	0,41	1,1
TSP	Solid	18	18	49,35	0,17	0,30	0,48	0,49	1,00	1,38	Not relevant

Table A.9 — Determination of phosphorus extracted in neutral ammonium citrate (EC method 3.2 after extraction with EC method 3.1.4, EN 15957)

Sample	Sample type	Number of labs	Number of labs retained after elimination of outliers	$\bar{x}$	s_r	RSD_r	r	s_R	RSD_R	R	Current EC Tolerance
NP (DAP)	Solid	17	14	46,22	0,09	0,19	0,24	0,38	0,83	1,07	(*)[a]
NPK (16-16-8+4S)	Solid	20	20	16,08	0,06	0,40	0,18	0,15	0,90	0,41	(*)[a]
TSP	Solid	18	14	48,34	0,12	0,24	0,33	0,39	0,81	1,09	0,8

[a] The tolerance allowed in respect of the declared solubilities of phosphorus pentoxide is one-tenth of the overall content of the nutrient concerned with a maximum of 2 % by mass, provided that the overall content of that nutrient remains within the tolerances specified in Annex II of EC Reg. 2003/2003.

Table A.10 — Determination of phosphorus extracted in water (EC method 3.2 after extraction with EC method 3.1.6, EN 15958)

Sample	Sample type	Number of labs	Number of labs retained after elimination of outliers	$\bar{x}$	s_r	RSD_r	r	s_R	RSD_R	R	Current EC Tolerance
NP (DAP)	Solid	17	16	42,72	0,11	0,26	0,31	0,84	1,96	2,34	(*)[a]
NPK (16-16-8+4S)	Solid	20	20	14,22	0,06	0,40	0,18	0,14	1,00	0,38	(*)[a]
TSP	Solid	18	17	46,56	0,15	0,30	0,41	0,73	1,60	2,04	1,3

[a] The tolerance allowed in respect of the declared solubilities of phosphorus pentoxide is one-tenth of the overall content of the nutrient concerned with a maximum of 2 % by mass, provided that the overall content of that nutrient remains within the tolerances specified in Annex II of EC Reg. 2003/2003.

Findings

EC method 3.1.1

— Based on the statistics above, there can be concluded that Method 3.2 after extraction with Method 3.1.1 performs well, under repeatability as well as reproducibility conditions.

— Considering the statistical data of both the alternative methods, the definition of the reproducibility limit, as well as the tolerances mentioned in Annex II of EC Regulations 2003/2003 it can be concluded that, despite the good results, difficulties occur. The value of R already exceeds the allowed tolerance of 1,1 % (Annex II of Regulation (EC) No. 2003/2003), whereas this tolerance should cover variations in manufacturing, sampling and analysis.

EC method 3.1.4

— Based on the statistics above, there can be concluded that Method 3.2 after extraction with Method 3.1.4 performs well, under repeatability as well as reproducibility conditions.

— Considering the statistical data of both the alternative methods, the definition of the reproducibility limit, as well as the tolerances mentioned in Annex II of EC Regulations 2003/2003 it can be concluded that, despite the good results, difficulties occur. The value of R already exceeds the allowed tolerance of 1,1 % (Annex II of Regulation (EC) No. 2003/2003), whereas this tolerance should cover variations in manufacturing, sampling and analysis.

EC method 3.1.6

— Based on the statistics above, there can be concluded that Method 3.2 after extraction with Method 3.1.6 performs well, under repeatability as well as reproducibility conditions.

— Considering the statistical data of both the alternative methods, the definition of the reproducibility limit, as well as the tolerances mentioned in Annex II of EC Regulations 2003/2003 it can be concluded that, despite the good results, difficulties occur. The value of R already exceeds the allowed tolerance of 1,1 % (Annex II of Regulation (EC) No. 2003/2003), whereas this tolerance should cover variations in manufacturing, sampling and analysis.

A.4 Potassium

Table A.11 — Determination of the water-soluble potassium content (EC method 4.1, EN 15477)

Sample	Sample type	Number of labs	Number of labs retained after elimination of outliers	$\bar{x}$	s_r	RSD_r	r	s_R	RSD_R	R	Current EC Tole-rance
NPK1 (14-8-24+8S)	Solid	20	19	24,62	0,15	0,6	0,43	0,28	1,8	0,80	1,1
NPK2 (16-16-8+4S)	Solid	18	17	8,18	0,04	0,5	0,12	0,14	1,7	0,40	1,1

Findings:

EC method 4.1

— Based on the statistics above, there can be concluded that Method 4.1 performs well, under repeatability as well as reproducibility conditions.

— Considering the statistical data, the definition of the reproducibility limit, as well as the tolerances mentioned in Annex II of EC Regulations 2003/2003 for potassic fertilizers it can be concluded that, despite the good results, difficulties might occur for certain products, especially since the total tolerances (for both or all three nutrients respectively) equal 1,5 % for binary (NP, PK, NK) and 1,9 % for ternary (NPK) fertilizers.

A.5 Sulfur

Table A.12 — Determination of total sulfur in the form of sulfates (EC methods 8.1 (EN 15960) and 8.9 (EN 15749))

Sample	Sample type	Number of labs	Number of labs retained after elimination of outliers	$\bar{x}$	sr	RSD_r	r	s_R	RSD_R	R	Current EC Tole-rance
NPK (14-8-14+8S)	Solid	19	16	7,14	0,06	0,9	0,18	0,19	2,6	0,52	(*)[a]
NPK (16-16-8+4S)	Solid	19	19	10,22	0,14	1,4	0,39	0,3	2,9	0,84	(*)[a]
NPK (16-16-8+4S) repetition	Solid	10	10	10,25	0,13	1,3	0,36	0,16	1,6	0,45	(*)[a]
NS (AN based)	Solid	18	17	15,66	0,11	0,7	0,31	0,28	1,8	0,78	(*)[a]

[a] The tolerances allowed in respect of the declared sulfur content shall be a quarter of the declared content up to a maximum of 0,9 % in absolute terms for SO_3, i.e. 0,36 for S.

Table A.13 — Determination of total sulfur in various forms (EC methods 8.2 (EN 15925) and 8.9 (EN 15749))

Sample	Sample type	Number of labs	Number of labs retained after elimination of outliers	$\bar{x}$	s_r	RSD_r	r	s_R	RSD_R	R	Current EC Tole-rance
Urea+S	Solid	13	11	13,85	0,15	1,1	0,42	0,4	2,9	1,13	(*)[a]
Urea+S repetition	Solid	7	7	14,08	0,11	0,8	0,32	0,62	4,4	1,74	(*)[a]

[a] The tolerances allowed in respect of the declared sulfur content shall be a quarter of the declared content up to a maximum of 0,9 % in absolute terms for SO_3, i.e. 0,36 for S.

Table A.14 — Determination of water-soluble sulfur in the form of sulfates (EC methods 8.3 (EN 15961) and 8.9 (EN 15749))

Sample	Sample type	Number of labs	Number of labs retained after elimination of outliers	$\bar{x}$	s_r	RSD_r	r	s_R	RSD_R	R	Current EC Tole-rance
NPK (14-8-14+8S)	Solid	19	18	6,89	0,10	1,5	0,29	0,28	4,0	0,77	(*)[a]
NPK (16-16-8+4S)	Solid	19	19	9,83	0,13	1,3	0,37	0,36	3,7	1,01	(*)[a]
NS (AN based)	Solid	18	18	14,32	0,18	1,3	0,5	1,32	9,2	3,69	(*)[a]
NS (AN based) repetition	Solid	10	10	14,1	0,2	1,7	0,7	1,6	11,11	4,4	(*)[a]

[a] The tolerances allowed in respect of the declared sulfur content shall be a quarter of the declared content up to a maximum of 0,9 % in absolute terms for SO_3, i.e. 0,36 for S.

Table A.15 — Determination of water-soluble sulfur in various forms (EC methods 8.4 (EN 15926) and 8.9 (EN 15749))

Sample	Sample type	Number of labs	Number of labs retained after elimination of outliers	$\bar{x}$	s_r	RSD_r	r	s_R	RSD_R	R	Current EC Tole-rance
UAN+S	Liquid	15	14	16,35	0,19	1,1	0,52	1,65	10,1	4,63	(*)[a]
UAN+S repetition	Liquid	8	6	17,43	0,09	0,5	0,24	0,09	0,5	0,27	(*)[a]

[a] The tolerances allowed in respect of the declared sulfur content shall be a quarter of the declared content up to a maximum of 0,9 % in absolute terms for SO_3, i.e. 0,36 for S.

Table A.16 — Determination of total sulfur in the form of sulfates, using ICP

Sample	Sample type	Number of labs	Number of labs retained after elimination of outliers	$\bar{x}$	s_r	RSD_r	r	s_R	RSD_R	R	Current EC Tole-rance
NPK (16-16-8+4S)	Solid	16	16	10,09	0,12	1,2	0,34	0,58	5,8	1,63	(*)[a]
NS (AN based)	Solid	16	16	15,05	0,25	1,7	0,71	1,13	7,5	3,17	(*)[a]
UAN+S	Liquid	12	10	13,55	0,15	1,1	0,42	0,91	6,7	2,56	(*)[a]

[a] The tolerances allowed in respect of the declared sulfur content shall be a quarter of the declared content up to a maximum of 0,9 % in absolute terms for SO_3, i.e. 0,36 for S.

Table A.17 — Determination of total sulfur in the form of sulfates, using IC

Sample	Sample type	Number of labs	Number of labs retained after elimination of outliers	$\bar{x}$	s_r	RSD_r	r	s_R	RSD_R	R	Current EC Tole-rance
NPK (16-16-8+4S)	Solid	8	6	10,25	0,06	0,6	0,18	0,4	3,9	1,13	(*)[a]
NS (AN based)	Solid	8	8	14,8	0,3	1,9	0,8	1,2	8,3	3,5	(*)[a]
UAN+S	Liquid	7	6	13,9	0,2	1,4	0,6	1,9	13,6	5,3	(*)[a]

[a] The tolerances allowed in respect of the declared sulfur content shall be a quarter of the declared content up to a maximum of 0,9 % in absolute terms for SO_3, i.e. 0,36 for S.

Findings:

— Based on the statistics above, there can be concluded that the methods for determination of sulfur give poor results, in particular under reproducibility conditions.

— Two alternative instrumental methods, using ICP and IC, have been ring tested and perform equally well.

— Considering the statistical data of both the alternative methods, the definition of the reproducibility limit, as well as the tolerances mentioned in Annex II of EC Regulations 2003/2003 it can be concluded that in most cases difficulties occur. The value of R already exceeds the allowed tolerance of maximum 0,9 % (Annex II of Regulation (EC) No. 2003/2003), whereas this tolerance should cover variations in manufacturing, sampling and analysis.

A.6 Chloride

Table A.18 — Determination of chloride in the absence of organic material (EC method 6.1, EN 16195)

Sample	Sample type	Number of labs	Number of labs retained after elimination of outliers	$\bar{x}$	s_r	RSD_r	R	s_R	RSD_R	R	Current EC Tolerance
Patent kali	Solid	14	11	2,78	0,024	0,9	0,07	0,10	3,6	0,28	0,2
NPK (12-11-18+4+8)	Solid	13	13	0,48	0,027	6,0	0,08	0,11	23,0	0,31	0,2

Findings:

— Based on the statistics above, there can be concluded that EC method 6.1 performs well, under repeatability as well as reproducibility conditions, at least for higher chloride levels.

— Considering the statistical data, the definition of the reproducibility limit, as well as the tolerances mentioned in Annex II of EC Regulations 2003/2003 it can be concluded that, despite the results, difficulties might occur for certain products, especially those with low chloride contents (< 2 %).

A.7 Secondary nutrients

A.7.1 Manganimetric determination of extracted calcium following precipitation in the form of oxalate (EC method 8.6, EN 16196)

Table A.19 — EC methods 8.1 (EN 15960) and 8.1 (EN 16196) – Ring test 2007 and repetition 2009

Sample	Sample type	Number of labs	Number of labs retained after elimination of outliers	$\bar{x}$	s_r	RSD_r	R	s_R	RSD_R	R	Current EC Tolerance
CAN	Solid	14	12	6,39	0,07	1,1	0,19	0,40	6,3	1,13	(*)[a]
NPK1:(23-4-13+7SO_3)	Solid	11	11	0,51	0,03	6,0	0,08	0,15	30,0	0,43	(*)[a]
NPK2:(12-11-18+2+8)	Solid	13	13	3,49	0,06	1,6	0,15	0,43	12,3	1,20	(*)[a]
CAN repetition	Solid	12	12	6,20	0,09	1,4	0,25	0,43	6,9	1,2	(*)[a]
NPK:(12-12-17S+2) repetition	Solid	12	10	5,80	0,05	0,8	0,13	0,26	4,5	0,74	(*)[a]

[a] The tolerances allowed in respect of the declared calcium content shall be a quarter of the declared content up to a maximum of 0,9 % in absolute terms for CaO, i.e. 0,64 for Ca.

Table A.20 — EC methods 8.1 (EN 15960) and ICP – Ring test 2009

Sample	Sample type	Number of labs	Number of labs retained after elimination of outliers	$\bar{x}$	s_r	RSD_r	R	s_R	RSD_R	R	Current EC Tolerance
CAN	Solid	17	15	6,34	0,08	1,2	0,21	0,33	5,3	0,93	(*)[a]
NPK: (12-12-17S+2)	Solid	17	16	5,78	0,09	1,6	0,25	0,33	5,6	0,91	(*)[a]

[a] The tolerances allowed in respect of the declared calcium content shall be a quarter of the declared content up to a maximum of 0,9 % in absolute terms for CaO, i.e. 0,64 for Ca.

Table A.21 — EC methods 8.3 (EN 15961) and 8.6 (EN 16196) – Ring test 2007 and repetition 2009

Sample	Sample type	Number of labs	Number of labs retained after elimination of outliers	$\bar{x}$	s_r	RSD_r	R	s_R	RSD_R	R	Current EC Tole-rance
CAN	Solid	14	14	2,49	0,14	5,0	0,38	0,66	27,0	1,85	(*)[a]
NPK1: (23-4-13+7SO_3)	Solid	10	10	0,36	0,02	6,0	0,07	0,11	31,0	0,31	(*)[a]
NPK2:(12-11-18+2+8)	Solid	12	12	0,53	0,03	7,0	0,10	0,18	33,0	0,49	(*)[a]
CAN repetition	Solid	12	10	2,61	0,07	3,0	0,19	1,45	55,0	4,05	(*)[a]
NPK: (12-12-17S+2) repetition	Solid	12	12	3,52	0,10	3,0	0,27	1,40	40,0	3,93	(*)[a]

[a] The tolerances allowed in respect of the declared calcium content shall be a quarter of the declared content up to a maximum of 0,9 % in absolute terms for CaO, i.e. 0,64 for Ca.

Table A.22 — EC methods 8.3 (EN 15961) and ICP – Ring test 2009

Sample	Sample type	Number of labs	Number of labs retained after elimination of outliers	$\bar{x}$	s_r	RSD_r	R	s_R	RSD_R	R	Current EC Tole-rance
CAN	Solid	16	15	2,45	0,08	3,0	0,24	1,38	56,0	3,85	(*)[a]
NPK: 12-12-17S+2	solid	16	16	3,34	0,07	2,2	0,21	1,43	42,7	4,00	(*)[a]

[a] The tolerances allowed in respect of the declared calcium content shall be a quarter of the declared content up to a maximum of 0,9 % in absolute terms for CaO, i.e. 0,64 for Ca.

Findings:

— Based on the statistics above, there can be concluded that the methods for determination of calcium give poor results, in particular under reproducibility conditions.

— Especially in case of water soluble calcium, the relative standard deviation under reproducibility conditions is unacceptably high from a statistical point of view.

— An alternative instrumental method, using ICP, has been ring tested and performs equally well.

— Considering the statistical data of both the alternative methods, the definition of the reproducibility limit, as well as the tolerances mentioned in Annex II of EC Regulations 2003/2003 it can be concluded that in most cases difficulties occur. The value of R already exceeds the allowed tolerance of maximum 0,9 % (Annex II of Regulation (EC) No. 2003/2003), whereas this tolerance should cover variations in manufacturing, sampling and analysis.

— Additional testing, especially for the water soluble calcium, has been performed (i.e. heating rate, aliquot, digestion temperature, digestion time, reflux conditions) without improved statistical data.

— If from an enforcement point of view the statistics are insufficiently in line with the tolerance limits, improvement of the method is needed and further work has to be done.

A.7.2 Determination of magnesium by atomic absorption spectrometry (EC method 8.7, EN 16197)

Table A.23 — EC methods 8.1 (EN 15960) and 8.7 (EN 16197) – Ring test 2007 and repetition 2009

Sample	Sample type	Number of labs	Number of labs retained after elimination of outliers	$\bar{x}$	s_r	RSD_r	r	s_R	RSD_R	R	Current EC Tole-rance
CAN	Solid	13	13	4,75	0,05	1,1	0,15	0,29	6,2	0,82	(*)[a]
KALI ROH	Solid	14	12	6,28	0,09	1,4	0,25	0,29	4,6	0,80	(*)[a]
NPK2: 12-11-18+2+8	Solid	14	12	3,38	0,05	1,4	0,13	0,27	7,9	0,74	(*)[a]
CAN repetition	Solid	10	8	4,38	0,04	0,9	0,11	0,59	13,5	1,65	(*)[a]
NPK: 12-12-17S+2 repetition	Solid	10	10	1,88	0,03	1,5	0,08	0,12	6,2	0,32	(*)[a]

[a] The tolerances allowed in respect of the declared magnesium content shall be a quarter of the declared content up to a maximum of 0,9 % in absolute terms for MgO, i.e. 0,55 for Mg.

Table A.24 — EC method 8.1 (EN 15960) and ICP – Ring test 2009

Sample	Sample type	Number of labs	Number of labs retained after elimination of outliers	$\bar{x}$	s_r	RSD_r	r	s_R	RSD_R	R	Current EC Tole-rance
CAN	Solid	17	16	4,61	0,05	1,0	0,13	0,17	3,7	0,48	(*)[a]
NPK: 12-12-17S+2	Solid	17	17	1,88	0,02	1,1	0,06	0,09	5,0	0,27	(*)[a]

[a] The tolerances allowed in respect of the declared magnesium content shall be a quarter of the declared content up to a maximum of 0,9 % in absolute terms for MgO, i.e. 0,55 for Mg.

Table A.25 — EC methods 8.3 (EN 15961) and 8.7 (EN 16197) – Ring test 2007 and repetition 2009

Sample	Sample type	Number of labs	Number of labs retained after elimination of outliers	$\bar{x}$	s_r	RSD_r	r	s_R	RSD_R	R	Current EC Tole-rance
CAN	Solid	13	12	1,83	0,09	5,0	0,25	0,42	23,0	1,18	(*)[a]
KALI ROH	Solid	14	14	6,05	0,07	1,2	0,20	0,30	4,9	0,83	(*)[a]
NPK2: 12-11-18+2+8	Solid	14	14	3,04	0,05	1,6	0,13	0,22	7,2	0,62	(*)[a]
CAN repetition	Solid	9	8	2,38	0,11	4,0	0,30	1,15	48,0	3,23	(*)[a]
NPK: 12-12-17S+2 repetition	Solid	9	9	1,58	0,03	1,8	0,08	0,18	11,6	0,52	(*)[a]

[a] The tolerances allowed in respect of the declared magnesium content shall be a quarter of the declared content up to a maximum of 0,9 % in absolute terms for MgO, i.e. 0,55 for Mg.

Table A.26 — EC method 8.3 (EN 15961) and ICP – Ring test 2009

Sample	Sample type	Number of labs	Number of labs retained after elimination of outliers	$\bar{x}$	s_r	RSD_r	r	s_R	RSD_R	R	Current EC Tole-rance
CAN	Solid	16	15	1,89	0,07	4,0	0,19	0,90	48,0	2,53	(*)[a]
NPK: 12-12-17S+2	Solid	16	15	1,59	0,03	2,0	0,09	0,22	13,8	0,61	(*)[a]

[a] The tolerances allowed in respect of the declared magnesium content shall be a quarter of the declared content up to a maximum of 0,9 % in absolute terms for MgO, i.e. 0,55 for Mg.

Findings:

— Based on the above-mentioned statistics it can be concluded that the methods for determination of magnesium give poor results, in particular under reproducibility conditions.

— An alternative instrumental method, using ICP, has also been ring tested.

— Considering the statistical data of both the alternative methods, the definition of the reproducibility limit, as well as the tolerances mentioned in Annex II of Regulation (EC) No. 2003/2003 it can be concluded that in most cases difficulties occur. The value of R already exceeds the allowed tolerance of maximum 0,9 % (Annex II of Regulation (EC) No. 2003/2003), whereas this tolerance is meant to cover variations in manufacturing, sampling and analysis.

A.7.3 Determination of magnesium by complexometry (EC method 8.8, EN 16198)

Table A.27 — EC methods 8.1 (EN 15960) and 8.8 (EN 16198) – Ring test 2007 and repetition 2009

Sample	Sample type	Number of labs	Number of labs retained after elimination of outliers	$\bar{x}$	s_r	RSD_r	r	s_R	RSD_R	R	Current EC Tole-rance
CAN	Solid	12	10	4,80	0,05	1,0	0,13	0,18	3,8	0,51	(*)[a]
KALI ROH	Solid	12	11	6,28	0,09	1,4	0,25	0,21	3,3	0,58	(*)[a]
NPK2:12-11-18+2+8	Solid	7	6	3,46	0,09	3,0	0,26	0,18	5,0	0,51	(*)[a]
CAN Repetition	Solid	10	9	4,61	0,06	1,4	0,18	0,20	4,3	0,55	(*)[a]
NPK: 12-12-17S+2 repetition	Solid	9	9	1,90	0,05	3,0	0,15	0,20	11,0	0,57	(*)[a]

[a] The tolerances allowed in respect of the declared magnesium content shall be a quarter of the declared content up to a maximum of 0,9 % in absolute terms for MgO, i.e. 0,55 for Mg.

Table A.28 — EC methods 8.3 (EN 15961) and 8.8 (EN 16198) – Ring test 2007 and repetition 2009

Sample	Sample type	Number of labs	Number of labs retained after elimination of outliers	$\bar{x}$	s_r	RSD_r	r	s_R	RSD_R	R	Current EC Tole-rance
CAN	Solid	9	9	2,29	0,08	3,0	0,21	0,51	22,0	1,43	(*)[a]
KALI ROH	Solid	12	10	6,22	0,03	0,5	0,09	0,19	3,0	0,52	(*)[a]
NPK2:12-11-18+2+8	Solid	7	7	2,10	0,13	6,0	0,37	1,15	55,0	3,22	(*)[a]
CAN repetition	Solid	10	9	1,83	0,05	3,0	0,14	0,42	23,0	1,19	(*)[a]
NPK: 12-12-17S+2 repetition	Solid	9	8	1,55	0,03	1,8	0,08	0,27	17,3	0,75	(*)[a]
[a] The tolerances allowed in respect of the declared magnesium content shall be a quarter of the declared content up to a maximum of 0,9 % in absolute terms for MgO, i.e. 0,55 for Mg.											

Findings:

— Based on the statistics above, there can be concluded that the methods for determination of magnesium give poor results, in particular under reproducibility conditions.

— Considering the statistical data of both the alternative methods, the definition of the reproducibility limit, as well as the tolerances mentioned in Annex II of Regulation (EC) No. 2003/2003 it can be concluded that in most cases difficulties occur. The value of R already exceeds the allowed tolerance of maximum 0,9 % (Annex II of Regulation (EC) No. 2003/2003), whereas this tolerance should cover variations in manufacturing, sampling and analysis.

A.7.4 Determination of extracted sodium flame-emission spectrometry (EC method 8.10, EN 16199)

Table A.29 — EC methods 8.1 (EN 15960) and 8.10 (EN 16199) – Ring test 2007 and repetition 2009

Sample	Sample type	Number of labs	Number of labs retained after elimination of outliers	$\bar{x}$	s_r	RSD_r	r	s_R	RSD_R	R	Current EC Tole-rance
KALI ROH	Solid	11	8	30,2	0,3	1,0	0,9	1,6	5,2	4,4	(*)[a]
NPK 1: 20+0+8+Na	Solid	9	7	0,56	0,03	5,0	0,08	0,1	17,0	0,3	(*)[a]
NPK: 12-12-17S+2 repetition	Solid	5	4	5,46	0,08	1,0	0,22	0,08	1,0	0,22	(*)[a]
[a] The tolerances allowed in respect of the declared sodium content shall be a quarter of the declared content up to a maximum of 0,9 % in absolute terms for Na_2O, i.e. 0,67 for Na.											

Table A.30 — EC method 8.1 (EN 15960) and ICP – Ring test 2009

Sample	Sample type	Number of labs	Number of labs retained after elimination of outliers	$\bar{x}$	s_r	RSD_r	r	s_R	RSD_R	R	Current EC Tole-rance
NPK: 12-12-17S+2	Solid	14	12	5,32	0,05	1,0	0,15	0,32	6,0	0,90	(*)[a]

[a] The tolerances allowed in respect of the declared sodium content shall be a quarter of the declared content up to a maximum of 0,9 % in absolute terms for Na_2O, i.e. 0,67 for Na.

Table A.31 — EC methods 8.3 (EN 15961) and 8.10 (EN 16199) – Ring test 2007 and repetition 2009

Sample	Sample type	Number of labs	Number of labs retained after elimination of outliers	$\bar{x}$	s_r	RSD_r	r	s_R	RSD_R	R	Current EC Tole-rance
KALI ROH	Solid	10	7	29,8	0,16	0,5	0,44	0,5	1,7	1,4	(*)[a]
NPK1: 20+0+8+Na	Solid	9	8	0,52	0,01	3,0	0,04	0,04	8,0	0,12	(*)[a]
NPK: 12-12-17S+2 repetition	Solid	5	5	5,73	0,14	2,0	0,39	0,66	12,0	1,85	(*)[a]

[a] The tolerances allowed in respect of the declared sodium content shall be a quarter of the declared content up to a maximum of 0,9 % in absolute terms for Na_2O, i.e. 0,67 for Na.

Table A.32 — EC method 8.3 (EN 15961) and ICP – Ring test 2009

Sample	Sample type	Number of labs	Number of labs retained after elimination of outliers	$\bar{x}$	s_r	RSD_r	r	s_R	RSD_R	R	Current EC Tole-rance
NPK: 12-12-17S+2	Solid	13	12	5,36	0,07	1,3	0,20	0,47	8,7	1,31	(*)[a]

[a] The tolerances allowed in respect of the declared sodium content shall be a quarter of the declared content up to a maximum of 0,9 % in absolute terms for Na_2O, i.e. 0,67 for Na.

Findings:

— Based on the statistics above, there can be concluded that the methods for determination of sodium give poor results, in particular under reproducibility conditions.

— An alternative instrumental method, using ICP [7], has also been ring tested.

— Considering the statistical data of both the alternative methods, the definition of the reproducibility limit, as well as the tolerances mentioned in Annex II of Regulation (EC) No. 2003/2003 it can be concluded that in most cases difficulties occur. The value of R already exceeds the allowed tolerance of maximum 0,9 % (Annex II of Regulation (EC) No. 2003/2003), whereas this tolerance should cover variations in manufacturing, sampling and analysis.

Annex B
(informative)

Refined statistical evaluation

B.1 General

This annex provides detailed information on the statistical evaluation of the data from the ring tests in secondary nutrients. Explanation on statistical data analysis is given in 4.2 and 4.4.

B.2 Meaning of symbols

r is the repeatability limit;

R is the reproducibility limit;

s_r is the repeatability standard deviation;

s_R is the reproducibility standard deviation;

RSD_r is the relative repeatability standard deviation;

RSD_R is the relative reproducibility standard deviation;

s_L^2 is the estimate of the between –laboratory variance;

s_W^2 is the estimate of the within –laboratory variance;

s_r^2 is the arithmetic mean of s_w^2 and is the estimate of the repeatability variance; the arithmetic mean is taken over all those laboratories taking part in the accuracy experiment. Analysis of raw data includes all values. If outliers are found, those are omitted in the calculation of the arithmetic mean.

s_R^2 is the estimate of the reproducibility variance given by Formula B.1.

$$s_R^2 = s_L^2 + s_r^2 \tag{B.1}$$

The relation between reproducibility limit R, reproducibility standard deviation s_R and tolerance T is given by Formula B.2.

$$R = 2{,}8 \cdot s_R \leq 0{,}7 \cdot T \tag{B.2}$$

The relation between reproducibility standard deviation s_R, mean, $\bar{x}$, relative reproducibility standard deviation RSD_R and tolerance T is given by Formula (B.3).

$$s_R \cdot 4 = \bar{x} \cdot \frac{RSD_R}{100} \cdot 4 \leq T \tag{B.3}$$

B.3 Tolerances for SO_3

Table B.1 — Results based on raw data

Analysis	$\bar{x}$ Raw Data %	Number of labs used in calculation n	RSD_R %	RSD "Normal" (Horwitz) %	Minimum Tolerance Raw Data %	Minimum Tolerance "Normal" RSD %	EC Tolerance %	Max RSD to comply with EC tolerance
NPK1_8.9&8.1	7,16	19	3,42	3	0,98	0,86	0,90	3,1
NPK2_8.9&8.1	10,21	19	2,93	3	1,19	1,22	0,90	2,2
NS_8.9&8.1	15,65	18	2,17	3	1,36	1,88	0,90	1,4
NPK1_8.9&8.3	6,83	19	5,97	3	1,63	0,82	0,90	3,3
NPK2_8.9&8.3	9,83	19	3,56	3	1,40	1,18	0,90	2,3
NSA_8.9&8.3	14,41	18	9,25	3	5,33	1,73	0,90	1,6

Table B.2 — Results based on ISO 5725-2

Analysis	$\bar{x}$ ISO 5725-2 %	Number of labs used in calculation n	RSD_R %	RSD "Normal" (Horwitz) %	Minimum Tolerance ISO 5725-2 %	Minimum Tolerance "Normal" RSD %	EC Tolerance %	Max RSD to comply with EC tolerance
NPK1_8.9&8.1	7,14	16	2,52	3	0,72	0,86	0,90	3,1
NPK2_8.9&8.1	10,21	19	2,93	3	1,19	1,22	0,90	2,2
NS_8.9&8.1	15,69	17	1,90	3	1,19	1,88	0,90	1,4
NPK1_8.9&8.3	6,91	18	3,97	3	1,10	0,83	0,90	3,3
NPK2_8.9&8.3	9,83	19	3,56	3	1,40	1,18	0,90	2,3
NSA_8.9&8.3	14,41	18	9,25	3	5,33	1,73	0,90	1,6

Table B.3 — Results based on refined data

Analysis	$\bar{x}$ Refined Data %	Number of labs used in calculation n	RSD_R %	RSD "Normal" (Horwitz) %	Minimum Tolerance Refined Data %	Minimum Tolerance "Normal" RSD %	EC Tolerance %	Max RSD to comply with EC tolerance
NPK1_8.9&8.1	7,16	12	1,39	3	0,40	0,86	0,90	3,1
NPK2_8.9&8.1	10,23	14	2,09	3	0,85	1,23	0,90	2,2
NS_8.9&8.1	15,67	14	1,36	3	0,85	1,88	0,90	1,4
NPK1_8.9&8.3	6,90	15	3,11	3	0,86	0,83	0,90	3,3
NPK2_8.9&8.3	9,86	13	1,64	3	0,65	1,18	0,90	2,3
NSA_8.9&8.3	14,81	11	3,93	3	2,33	1,78	0,90	1,5

B.4 Tolerances for CaO, MgO. Na_2O and Cl

Table B.4 — Results based on raw data

Analysis	$\bar{x}$ Raw Data	Number of labs used in calculation	RSD_R	RSD "Normal" (Horwitz)	Minimum Tolerance Raw Data	Minimum Tolerance "Normal" RSD	EC Tolerance	Max RSD to comply with EC tolerance
	%	n	%	%	%	%	%	
Chloride (6.1)	0,91	16	23,41	4	0,85	0,14	0,23	6,3
total CaO 8.6 liquid	0,13	10	12,85	5,5	0,07	0,03	0,03	6,3
total CaO ICP liquid	0,14	14	15,77	5,5	0,09	0,03	0,04	6,3
total CaO 8.6 NPK	5,84	12	4,62	3	1,08	0,70	0,90	3,9
total CaO ICP NPK	5,80	17	5,64	3	1,31	0,70	0,90	3,9
WS CaO 8.6 NPK	3,52	12	39,89	3,5	5,62	0,49	0,88	6,3
WS CaO ICP NPK	3,39	16	42,66	3,5	5,78	0,47	0,85	6,3
total CaO 8.6 CAN	6,20	12	6,91	3	1,72	0,74	0,90	3,6
total CaO ICP CAN	6,34	17	6,01	3	1,53	0,76	0,90	3,5
WS CaO 8.6 CAN	2,92	12	53,51	3,5	6,24	0,41	0,73	6,3
WS CaO ICP CAN	2,70	16	58,19	3,5	6,27	0,38	0,67	6,3
total MgO 8.7 NPK	1,88	10	6,18	3,5	0,46	0,26	0,47	6,3
total MgO 8.8 NPK	1,90	9	10,70	3,5	0,81	0,27	0,48	6,3
total MgO ICP NPK	1,88	17	5,13	3,5	0,39	0,26	0,47	6,3
WS MgO 8.7 NPK	1,58	9	11,68	4	0,74	0,25	0,40	6,3
WS MgO 8.8 NPK	1,43	9	30,42	4	1,74	0,23	0,36	6,3
WS MgO ICP NPK	1,59	16	13,35	4	0,85	0,26	0,40	6,3
total MgO 8.7 CAN	4,33	10	13,01	3,5	2,25	0,61	0,90	5,2
total MgO 8.8 CAN	4,43	10	13,41	3,5	2,38	0,62	0,90	5,1
total MgO ICP CAN	4,60	17	3,79	3,5	0,70	0,64	0,90	4,9
WS MgO 8.7 CAN	2,29	9	48,63	3,5	4,45	0,32	0,57	6,3
WS MgO 8.8 CAN	2,11	10	45,96	3,5	3,87	0,29	0,53	6,3
WS MgO ICP CAN	2,08	16	51,80	3,5	4,30	0,29	0,52	6,3
total Na_2O 8.10 NPK	5,71	5	9,86	3	2,25	0,69	0,90	3,9
total Na_2O ICP NPK	5,83	14	27,15	3	6,34	0,70	0,90	3,9
WS Na_2O 8.10 NPK	5,73	5	11,57	3	2,65	0,69	0,90	3,9
WS Na_2O ICP NPK	5,02	13	25,14	3	5,05	0,60	0,90	4,5

Table B.5 — Results based on ISO 5725-2

Analysis	$\bar{x}$ ISO 5725-2 %	Number of labs used in calculation n	RSD_R %	RSD "Normal" (Horwitz) %	Minimum Tolerance ISO 5725-2 %	Minimum Tolerance "Normal" RSD %	EC Tolerance %	Max RSD to comply with EC tolerance
Chloride (6.1)	0,95	15	11,84	4	0,45	0,15	0,24	6,3
total CaO 8.6 liquid	0,13	10	12,85	5,5	0,07	0,03	0,03	6,3
total CaO ICP liquid	0,14	14	15,77	5,5	0,09	0,03	0,04	6,3
total CaO 8.6 NPK	5,80	10	4,53	3	1,05	0,70	0,90	3,9
total CaO ICP NPK	5,80	17	5,64	3	1,31	0,70	0,90	3,9
WS CaO 8.6 NPK	3,52	12	39,89	3,5	5,62	0,49	0,88	6,3
WS CaO ICP NPK	3,39	16	42,66	3,5	5,78	0,47	0,85	6,3
total CaO 8.6 CAN	6,20	12	6,91	3	1,72	0,74	0,90	3,6
total CaO ICP CAN	6,34	15	5,27	3	1,33	0,76	0,90	3,6
WS CaO 8.6 CAN	2,68	11	51,98	3,5	5,57	0,38	0,67	6,3
WS CaO ICP CAN	2,50	15	56,41	3,5	5,65	0,35	0,63	6,3
total MgO 8.7 NPK	1,88	10	6,18	3,5	0,46	0,26	0,47	6,3
total MgO 8.8 NPK	1,90	9	10,70	3,5	0,81	0,27	0,48	6,3
total MgO ICP NPK	1,88	17	5,13	3,5	0,39	0,26	0,47	6,3
WS MgO 8.7 NPK	1,58	9	11,68	4	0,74	0,25	0,40	6,3
WS MgO 8.8 NPK	1,55	8	17,35	4	1,07	0,25	0,39	6,3
WS MgO ICP NPK	1,59	15	13,76	4	0,88	0,25	0,40	6,3
total MgO 8.7 CAN	4,57	7	4,96	3,5	0,91	0,64	0,90	4,9
total MgO 8.8 CAN	4,61	9	4,27	3,5	0,79	0,65	0,90	4,9
total MgO ICP CAN	4,60	16	3,82	3,5	0,70	0,64	0,90	4,9
WS MgO 8.7 CAN	2,29	9	48,63	3,5	4,45	0,32	0,57	6,3
WS MgO 8.8 CAN	1,83	9	23,26	3,5	1,70	0,26	0,46	6,3
WS MgO ICP CAN	1,75	14	35,75	3,5	2,50	0,24	0,44	6,3
total Na_2O 8.10 NPK	5,46	4	1,11	3	0,24	0,66	0,90	4,1
total Na_2O ICP NPK	5,31	12	6,11	3	1,30	0,64	0,90	4,2
WS Na_2O 8.10 NPK	5,73	5	11,57	3	2,65	0,69	0,90	3,9
WS Na_2O ICP NPK	5,35	12	8,67	3	1,86	0,64	0,90	4,2

Table B.6 — Results based on refined data

Analysis	$\bar{x}$ Refined Data %	Number of labs used in calculation n	RSD_R %	RSD "Normal" (Horwitz) %	Minimum Tolerance Refined Data %	Minimum Tolerance "Normal" RSD %	EC Tolerance %	Max RSD to comply with EC tolerance
Chloride (6.1)	0,96	10	6,53	4	0,25	0,15	0,24	6,3
total CaO 8.6 liquid	0,14	9	8,11	5,5	0,05	0,03	0,03	6,3
total CaO ICP liquid	0,15	11	7,26	5,5	0,04	0,03	0,04	6,3
total CaO 8.6 NPK	5,79	8	2,93	3	0,68	0,69	0,90	3,9
total CaO ICP NPK	5,78	12	2,36	3	0,55	0,69	0,90	3,9
WS CaO 8.6 NPK	2,76	9	10,18	3,5	1,12	0,39	0,69	6,3
WS CaO ICP NPK	2,80	10	12,76	3,5	1,43	0,39	0,70	6,3
total CaO 8.6 CAN	6,20	10	4,10	3	1,02	0,74	0,90	3,6
total CaO ICP CAN	6,32	14	2,93	3	0,74	0,76	0,90	3,6
WS CaO 8.6 CAN	2,34	9	36,04	3,5	3,37	0,33	0,58	6,3
WS CaO ICP CAN	2,02	13	31,12	3,5	2,52	0,28	0,51	6,3
total MgO 8.7 NPK	1,86	9	5,43	3,5	0,40	0,26	0,46	6,3
total MgO 8.8 NPK	1,85	8	8,36	3,5	0,62	0,26	0,46	6,3
total MgO ICP NPK	1,90	12	2,80	3,5	0,21	0,27	0,48	6,3
WS MgO 8.7 NPK	1,54	8	8,88	4	0,55	0,25	0,38	6,3
WS MgO 8.8 NPK	1,55	8	17,35	4	1,07	0,25	0,39	6,3
WS MgO ICP NPK	1,62	9	7,36	4	0,48	0,26	0,40	6,3
total MgO 8.7 CAN	4,57	7	4,96	3,5	0,91	0,64	0,90	6,3
total MgO 8.8 CAN	4,56	8	3,33	3,5	0,61	0,64	0,90	4,9
total MgO ICP CAN	4,63	12	1,97	3,5	0,36	0,65	0,90	4,9
WS MgO 8.7 CAN	2,00	8	36,50	3,5	2,92	0,28	0,50	6,3
WS MgO 8.8 CAN	1,83	9	23,26	3,5	1,70	0,26	0,46	6,3
WS MgO ICP CAN	1,65	12	18,46	3,5	1,22	0,23	0,41	6,3
total Na_2O 8.10 NPK	5,46	4	1,11	3	0,24	0,66	0,90	4,1
total Na_2O ICP NPK	5,30	10	4,58	3	0,97	0,64	0,90	4,2
WS Na_2O 8.10 NPK	5,73	5	11,57	3	2,65	0,69	0,90	3,9
WS Na_2O ICP NPK	5,25	11	5,59	3	1,17	0,63	0,90	4,3

Table B.7 — Explanation of column headings

Column heading	Explanation
$\bar{x}$ Raw Data, %	Mean value of the mean value for all labs
$\bar{x}$ ISO 5725-2	Mean value of the mean value for all labs after elimination of outliers according to ISO 5725-2
$\bar{x}$ Refined Data, %	Mean value of the mean value for all labs after refinement according description
RSD_R	Relative standard deviation for the different means taking into consideration both repeatability and reproducibility variance
Number of labs used in calculations, n	Number of labs included in the estimation of variance of means
RSD "Normal" (Horwitz), %	"Normal" attainable precision taking into account both between- and within laboratory components, expressed as relative standard deviation at the concentration level of the analyte under consideration (according to Horwitz et. al.)
Minimum Tolerance Raw Data, %	The smallest tolerance that comply with the estimated RSD_R from raw data, i. e. comply with $s_R \cdot 4 = \bar{x} \cdot \frac{RSD_R}{100} \cdot 4 \leq T$
Minimum Tolerance ISO 5725-2, %	The smallest tolerance that comply with the estimated RSD_R after removal of outliers (Dixon & Massey, Grubb's test and Cochran's test) i. e. comply with $s_R \cdot 4 = \bar{x} \cdot \frac{RSD_R}{100} \cdot 4 \leq T$
Minimum Tolerance Refined Data, %	The smallest tolerance that comply with the estimated RSD_R from refined data, i. e. comply with $s_R \cdot 4 = \bar{x} \cdot \frac{RSD_R}{100} \cdot 4 \leq T$
Minimum Tolerance "Normal" RSD_R Data, %	The smallest tolerance that comply with the "normal" RSD_R as estimated by Horwitz et. al., i. e. comply with $s_R \cdot 4 = \bar{x} \cdot \frac{RSD_R}{100} \cdot 4 \leq T$
EC Tolerance, %	Tolerance put forward by European Commission
Max. RSD_R to comply with EC Tolerance, %	Upper limit of RSD_R that can comply with EC tolerances, i. e. $s_R \cdot 4 = \bar{x} \cdot \frac{RSD_R}{100} \cdot 4 \leq T$

B.5 Refinement of results

— Samples with standard deviation = 0 are omitted in calculations of repeatability.

— Remove results with poor repeatability (outliers from Cochran's test).

— Remove outliers among mean values (Dixon & Massey and Grubb's test).

— Calculate mean of remaining mean values reported by the laboratories.

— Calculate the standard deviation of mean for the remaining laboratories.

— Calculate the mean of the standard deviation for remaining laboratories.

— Calculate "gross" standard deviation: $\sigma R = \sqrt{\sigma_{\mathrm{L}}{}^2 + \sigma_r{}^2}$

— Calculate confidence interval of means: $\bar{x} \pm t_{0,1} \cdot \sigma_R / \sqrt{n}$ (99,9 % probability that the means are within this interval).

— Remove results outside the confidence interval ("outliers").

— Recalculate mean and standard deviations on remaining results.

B.6 Comments

Inspection of the results from the ring test reveals that some data have a big variance.

This big variance imply that to define the result (the mean of the replicates) from a laboratory as an outlier by Grubb's test and the test of Dixon & Massey it must show an extreme value compared to the mean value of the results from the attending laboratories. The associated Tolerance level that can comply with the method is therefore in some cases unacceptable high for use in commercial relations.

It has therefore been done an extra exercise on the data: Based on the standard deviation of the results from the date after removal of outliers with the tests mentioned above, the 99,9 % confidence interval of the remaining means have been calculated. Then the results outside the confidence interval are removed and the standard deviation of results within the 99,9 % confidence interval calculated. These values are the bases for the RSD_R of the "refined data".

For the methods investigated in this ring test, the RSD_R are not far from expected according to Horwitz et al. It can therefore be questioned if the tolerance set is too tight.

Bibliography

[1] *Official analytical methods and legal tolerances for fertilizers in Europe*: IFS Proceedings No 490 (2002)

[2] *Regulation (EC) No 2003/2003 of the European Parliament and of the Council of 13 October 2003 relating to fertilisers*, Official Journal L 304, 21/11/2003 P. 0001-0194 and amendments available from: http://eur-lex.europa.eu/LexUriServ/LexUriServ.do?uri=OJ:L:2003:304:0001:0194:EN:PDF

[3] Box, Hunter & Hunter, *Statistic for Experimenters. Chapt. 5* John Wiley & Sons. Inc. ISBN 0-470-09315-7

[4] W. Horwitz, L.R. Kamps and K.W. Boyer, J.Assoc. Off. Anal. Chem., 63 (1980) 1344

[5] D. L. Massart et.al, *Data Handling in Science and Technology 2, Chemometrics*: a textbook, p. 21-22

[6] German method using Iron and Tin Chloride for method 2.6.6: Methode 3.5.2.3: *Bestimmung von Gesamtstickkstoff – Bei Anwesenheit von Nitratstickstoff, Reduktion des Nitratanteils mit Eisenpulver und Zinn(II)-Chlorid*

[7] *An alternative instrumental method, using ICP* (8.10)

[8] *Determination of Ca, Mg, Na content using ICP-OES*; in-house method

[9] *Methods of Sampling and Analysis,* Annex IV of Regulation (EC) No 2003/2003 of the European Parliament and of the Council of 13 October 2003 relating to fertilisers, Official Journal L 304, 21/11/2003 P. 0073-0191

[10] EN 15475, *Fertilizers — Determination of ammoniacal nitrogen*

[11] EN 15476, *Fertilizers — Determination of nitric and ammoniacal nitrogen according to Devarda*

[12] EN 15477, *Fertilizers — Determination of the water-soluble potassium content*

[13] EN 15478, *Fertilizers — Determination of total nitrogen in urea*

[14] EN 15479, *Fertilizers — Spectrophotometric determination of biuret in urea*

[15] EN 15749, *Fertilizers — Determination of sulfates content using three different methods*

[16] EN 15750, *Fertilizers — Determination of total nitrogen in fertilizers containing nitrogen only as nitric, ammoniacal and urea nitrogen by two different methods*

[17] EN 15925, *Fertilizers — Extraction of total sulfur present in various forms*

[18] EN 15926, *Fertilizers — Extraction of water soluble sulfur where the sulfur is in various forms*

[19] EN 15956, *Fertilizers — Extraction of phosphorus soluble in mineral acids*

[20] EN 15957, *Fertilizers — Extraction of phosphorus which is soluble in neutral ammonium citrate*

[21] EN 15958, *Fertilizers — Extraction of water soluble phosphorus*

[22] EN 15960, *Fertilizers — Extraction of total calcium, total magnesium, total sodium and total sulfur in the forms of sulfates*

[23] EN 15961, *Fertilizers — Extraction of water-soluble calcium, magnesium, sodium and sulfur in the form of sulfates*

[24] EN 16195, *Fertilizers — Determination of chlorides in the absence of organic material*

[25] EN 16196, *Fertilizers — Manganimetric determination of extracted calcium following precipitation in the form of oxalate*

[26] EN 16197, *Fertilizers — Determination of magnesium by atomic absorption spectrometry*

[27] EN 16198, *Fertilizers — Determination of magnesium by complexometry*

[28] EN 16199, *Fertilizers — Determination of the sodium extracted by flame-emission spectrometry*

[29] ISO 5315, *Fertilizers – determination of total nitrogen content – titrimetric method after distillation*

[30] ISO 5725-2, *Accuracy (trueness and precision) of measurement methods and results — Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method*

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