



Association of German Agricultural Analytic and Research Institutes

EU FERTILISER RING TEST Q4/2012: POTASSIUM CHLORIDE WITH MAGNESIUM 40 (+6+4+12)

In 2012, the Association of German Analytical and Research Institutes (VDLUFA e. V.) carried out an international fertiliser ring test to determine major and minor nutrients in a Potassium Chloride fertiliser with Magnesium 40 (+6+4+12). Purpose of this fertiliser ring test was to offer a platform for testing and documenting the analytical quality of laboratories in all EU countries. 34 laboratories from 12 European countries took part in the ring test with the designation EU Q4/2012.

The analytes to be reported by the participating laboratories had to be determined by various official or standardised methods (see Table 1). Laboratories were asked to prepare the samples for analysis according to Annex IV, Section B, Method 1 (sample preparation) of the Regulation (EC) No 2003/2003 of the European Parliament and of the Council of 13th October 2003 relating to fertilisers. Four aliquots of the sample material of the ring test EU Q4/2012 were prepared in order to be analysed. The results obtained for each individual determination (n=4) were reported in the units (mass %) given in Table 1, based on fresh mass.

Concerning the analysis of K, Mg, Na, S and Cl, final determination by ICP-OES (inductive coupled plasma optical emission spectrometry) was accepted as an alternative to the official methods. Element determination by means of ICP-OES is an official method of German National Fertiliser Regulation, but not of the Regulation (EC) No 2003/2003. It is reported in the VDLUFA METHODS BOOK, Volume II.1, Fertiliser Analysis (Ed. 1995-2012).

Table 1: Analytes to be determined and methods used

No.	Analyte	Method Digestion/ Extraction	Method Final determination	Unit	Comments
1	Na-water-EU	EU*) 8.3	EU 8.10	mass %	reported as Na ₂ O
2	K-water-EU	EU 4.1	EU 4.1	mass %	reported as K ₂ O
3	Mg-EU	EU 8.3	EU 8.7, EU 8.8	mass %	reported as MgO
4	S-water-EU	EU 8.3	EU 8.9	mass %	reported as SO ₃
5	Cl-water-EU	EU 6.1	EU 6.1	mass %	reported as Cl

*) Regulation (EC) No 2003/2003 of the European Parliament and of the Council of 13th October 2003 relating to Fertilisers.

The statistical evaluation was done by robust methods (DIN 38402 A45, Q-method, HAMPEL estimate). Z_u-scores (tolerance limit $|Z_u| \leq 2,0$) were calculated as a bias estimate using IUPAC guidelines, so that laboratories can evaluate their performance in comparison to other laboratories. HorRat values were calculated for the methods in case a sufficient number of results

had been reported. For all statistical calculations, the validated software package ProLab was used.

Table 2 shows all mean values, comparative standard deviations (absolute + relative), repeated standard deviation, tolerance limits and HorRat values.

Interested laboratories can be supplied with material from the tested fertiliser in order to use it as internal reference material (see order form).

Means, standard deviations and tolerance limits

Statistical method: DIN38402 A45

Criterion: Zu-Score <= 2

Ring Test: VDLUFA Fertilizer Ring Test EU Q4/2012

Sample	Measurand	Unit	Mean	(assigned) Standard deviations		Repeatability s.d.		Tolerance limits		Number of selected Laboratories	Values*
				Absolute	Relative	Absolute	Relative	Lower	Upper		
KORNKALI	K ₂ O · W · K · w ater · EU (as K ₂ O)	mass%	40,323	0,495	1,23 %	0,180	0,45 %	39,340	41,320	34	131
	MgO · EU (as MgO)	mass%	6,060	0,176	2,90 %	0,052	0,86 %	5,713	6,417	34	127
	Na ₂ W · Na · w ater · EU (as Na ₂ O)	mass%	4,996	0,268	5,36 %	0,044	0,89 %	4,474	5,547	34	120
	S ₂ W · S · w ater · EU (as SO ₃)	mass%	13,173	0,478	3,63 %	0,092	0,70 %	12,234	14,146	34	123
	Cl ₂ W · Cl · w ater · EU (as Cl)	mass%	35,780	0,566	1,58 %	0,133	0,37 %	34,656	36,922	34	111

The column 'Values' includes all states (therefore NB as well)