



Association of German Agricultural Analytic and Research Institutes

## EU FERTILISER RING TEST Q5/2013: NPK-FERTILISER 12+12+17 (+2 MgO+8 S)

In 2013, 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 NPK-fertiliser 12+12+17 (+ 2 MgO + 8 S). Purpose of this fertiliser ring test was to offer a platform for testing and documenting the analytical quality of laboratories in all EU countries. 40 laboratories from 15 European countries took part in the ring test with the designation EU Q5/2013.

The analytes to be reported by the participating laboratories had to be determined by various official or standardized 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. For analysing P, K, Mg, S, B, Cu, Mn, Fe and Zn, final determination by inductively coupled plasma optical emission spectrometry (ICP-OES) was accepted as an alternative to official methods. ICP-OES is an official method for these elements in Germany (according to the German fertiliser ordinance), but not in EU Regulation 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	Digestion/Extraction Method	Final Determination Method	Unit	Reported as
1	N-total-VDLUFA	VDLUFA 3.5.2.7	Elemental Analysis	mass% fm	N
2	N-total-EU	EU 2.6	EU 2.6	mass% fm	N
3	N-NO <sub>3</sub> -EU	EU 2.2	EU 2.2	mass% fm	NO <sub>3</sub> -N
4	N-NH <sub>4</sub> -EU	EU 2.2	EU 2.2	mass% fm	NH <sub>4</sub> -N
5	P <sub>2</sub> O <sub>5</sub> -water-EU	EU 3.1.6	EU 3.2	mass% fm	P <sub>2</sub> O <sub>5</sub>
6	P <sub>2</sub> O <sub>5</sub> -nac-EU	EU 3.1.4	EU 3.2	mass% fm	P <sub>2</sub> O <sub>5</sub>
7	P <sub>2</sub> O <sub>5</sub> -tot-EU	EU 3.1.1	EU 3.2	mass% fm	P <sub>2</sub> O <sub>5</sub>
8	K <sub>2</sub> O-water-EU	EU 4.1	EU 4.1	mass% fm	K <sub>2</sub> O
9	MgO-EU	EU 8.1	EU 8.7, EU 8.8	mass% fm	MgO
10	S-water-EU	EU 8.3	EU 8.9	mass% fm	S
11	S-HCl-EU	EU 8.1	EU 8.9	mass% fm	S
12	B-HCl-EU	EU 9.1	EU 9.5	mg/kg fm	B
13	Zn-HCl-EU	EU 9.1	EU 9.11	mg/kg fm	Zn
14	Fe-HCl-EU	EU 9.1	EU 9.8	mg/kg fm	Fe
15	Cd-aqua regia	CEN/TS 16319	CEN/TS 16319	mg/kg fm	Cd

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

fm: fresh matter

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.

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

## Mean, Standard Deviation, HorRat and Tolerance Limits

Method DIN3840Z A45  
 Criterion Zu-Score <= 2

### VDLUFA Fertilizer Ring Test EU Q5/2013

Sample Messurand	Unit	Mean	Reprod.S.D.		Repeat.S.D.		HorRat	Limit of Tolerance		Number of Laboratories	Values
			Abs.	Rel.	Abs.	Rel.		Lower	Upper		
NPK_2 N_TOT_L - N'total\VDLUFA 3.5.2.7 (as N)	mass%	12,122	0,186	1,53 %	0,044	0,36 %	0,6	11,750	12,500	19	67
N_TOT_EU - N'total:EU 2.6 (as N)	mass%	12,082	0,148	1,22 %	0,049	0,40 %	0,4	11,790	12,380	30	107
N03_N - N·N03:EU 2.2 (as NO3-N)	mass%	4,892	0,170	3,48 %	0,048	0,98 %	1,1	4,557	5,238	33	119
NH4_N - N·NH4:EU 2.2 (as NH4-N)	mass%	7,177	0,106	1,47 %	0,029	0,41 %	0,5	6,967	7,390	33	119
P205_W - P205-w ater:EU 3.1.6 (as P2O5)	mass%	9,501	0,305	3,21 %	0,060	0,63 %	1,1	8,901	10,120	37	130
P205_N - P205-nac:EU 3.1.4 (as P2O5)	mass%	12,027	0,236	1,96 %	0,069	0,57 %	0,7	11,560	12,500	35	122
P205_TOT - P205-total:EU 3.1.1 (as P2O5)	mass%	12,187	0,236	1,94 %	0,058	0,48 %	0,7	11,719	12,665	37	129
K2O_W - K2O-w ater:EU 4.1 (as K2O)	mass%	17,057	0,280	1,64 %	0,091	0,53 %	0,6	16,500	17,620	39	137
MGO - MgO:EU 8.1 (as MgO)	mass%	1,989	0,083	4,20 %	0,020	1,01 %	1,2	1,826	2,160	38	131
S_WA - S-w ater:EU 8.3 (as S)	mass%	8,492	0,284	3,35 %	0,061	0,72 %	1,2	7,932	9,071	35	123
S_HCL - S·HCl:EU 8.1 (as S)	mass%	8,727	0,284	3,26 %	0,062	0,71 %	1,1	8,168	9,305	34	117
B_HCL - B·HCl:EU 9.1 (as B)	mg/kg	238,912	57,226	23,95 %	4,111	1,72 %	3,4	135,100	370,700	32	113
ZN_HCL - Zn·HCl:EU 9.1 (as Zn)	mg/kg	110,841	15,825	14,28 %	1,193	1,08 %	1,8	81,140	145,090	33	116
FE_HCL - Fe·HCl:EU 9.1 (as Fe)	mg/kg	2824,558	1533,369	54,29 %	48,613	1,72 %	11,2	592,331	6889,153	33	115
CD - Cd-aqua regia:CEN/TS 16319 (as Cd)	mg/kg dm	4,775	0,509	10,65 %	0,060	1,26 %	0,8	3,807	5,853	32	112

