EU FERTILISER RING TEST Q6/2014: NP-FERTILISER (12 + 52)

In 2014, 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 NP-fertiliser 12 + 52. Purpose of this fertiliser ring test was to offer a platform for testing and documenting the analytical quality of laboratories in all EU countries.
39 laboratories from 16 European countries took part in the ring test with the designation EU Q6/2014.

The analytes to be reported by the participating laboratories had to be determined by various official or standardised methods (see Table 1).

Table 1: Analytes to be determined and methods used

No	Analyte	Method Digestion / Extractio	Method Final determinatio	Unit	Comments
1	N-total-EU	EU 2.6.2 or 2.1	EU 2.6.2	mass %	reported as N
1a	N-total-VDLUFA	VDLUFA(II.1) 3.5.2.7	VDLUFA(II.1) 3.5.2.7	mass %	reported as N
2	N-NH4-EU	EU 2.1	EU 2.1	mass %	reported as NH4-N
2a	N-NH4-VDLUFA	VDLUFA(II.1) 3.2.1	VDLUFA(II.1) 3.2.1	mass %	reported as NH4-N
3	P2O5-water-EU	EU 3.1.6	EU 3.2	mass %	reported as P2O5
3a	P2O5-water-VDLUFA	VDLUFA(II.1) 4.1.7	VDLUFA(II.1) 4.2.4	mass %	reported as P2O5
4	P2O5-nac-EU	EU 3.1.4	EU 3.2	mass %	reported as P2O5
4a	P2O5-nac-VDLUFA	VDLUFA(II.1) 4.1.4	VDLUFA(II.1) 4.2.4	mass %	reported as P2O5
5	Ca-HCl-EU	EU 8.1	EU 8.6?	mass %	reported as CaO
5a	Ca-HCl-VDLUFA	VDLUFA(II.1) 6.1.1	VDLUFA(II.1) 4.2.4	mass %	reported as CaO
6	S-HCl-EU	EU 8.1	EU 8.9	mass %	reported as S
6a	S-HCl-VDLUFA	VDLUFA(II.1) 6.1.1	VDLUFA(II.1) 4.2.4	mass %	reported as S
7	As-aqua regia	CEN/TS 16317	CEN/TS 16317	mg/kg*)	reported as As
7a	As-aqua regia	VDLUFA(II.1) 9.5.1	VDLUFA(II.1) 9.5.2	mg/kg*)	reported as As
8	U-aqua regia	ISO 11466	DIN EN ISO 17294-2	mg/kg*)	reported as U
8a	U-aqua regia	VDLUFA(VII) 2.2.3.1	VDLUFA(VII) 2.2.3.1	mg/kg*)	reported as U
9	Dry matter	EN 12048	EN 12048	mass %	reported as dm
9a	Dry matter	VDLUFA(II.1) 15.2.1	VDLUFA(II.1) 15.2.1	mass %	reported as dm

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

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.
For any parameter to be reported, at least 4 aliquots of the sample material were to be processed and analysed.
The results obtained for each individual determination were to be reported in the units (mass% or mg/kg) given in the table above based on fresh mass (fm).

For analysing P, Ca and S, final determination by inductively coupled plasma optical emission spectrometry (ICP-OES) was accepted as an alternative to EU-official methods. ICP-OES is an official method for these elements in Germany (according to the German fertiliser ordinance), but up to now not in EU Regulation No. 2003/2003.

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).

Mean, Standard Deviation, HorRat and Tolerance Limits

Method DIN38402 A45

Criterion Zu-Score <= 2

VDLUFA Fertilizer Ring Test EU Q6/2014

Sample	Measurand	Unit	Mean	Reprod.S.D. Abs.	Reprod.S.D. Rel.	Repeat.S.D. Abs.	Repeat.S.D. Rel.	HorRat	Limit of Tolerance Lower	Limit of Tolerance Upper	Number of Laboratories	Number of Values
NP_20	N_TOT_EU · N·total·EU (as N)	mass%	12,044	0,149	1,24 %	0,060	0,50 %	0,4	11,748	12,344	28	108
	N_TOT_L · N·total·VDLUFA (as N)	mass%	12,060	0,175	1,45 %	0,061	0,50 %	0,5	11,712	12,413	15	58
	NH4_N_EU · N·NH4·EU (as NH4·N)	mass%	12,018	0,126	1,05 %	0,048	0,40 %	0,4	11,767	12,272	29	113
	NH4_N_L · N·NH4·VDLUFA (as NH4·N)	mass%	11,974	0,101	0,85 %	0,047	0,39 %	0,3	11,772	12,177	10	40
	P2O5_W_E · P2O5·water·EU (as P2O5)	mass%	49,083	0,968	1,97 %	0,205	0,42 %	0,9	47,166	51,038	32	123
	P2O5_W_L · P2O5·water·VDLUFA (as P2O	mass%	48,822	1,363	2,79 %	0,212	0,43 %	1,3	46,133	51,586	14	56
	P2O5_N_E · P2O5·nac·EU (as P2O5)	mass%	51,912	0,858	1,65 %	0,185	0,36 %	0,7	50,209	53,643	32	124
	P2O5_N_L · P2O5·nac·VDLUFA (as P2O5)	mass%	51,684	0,881	1,71 %	0,298	0,58 %	0,8	49,935	53,462	11	43
	CA_HCL_E · Ca·HCl·EU (as CaO)	mass%	1,992	0,205	10,29 %	0,026	1,29 %	2,9	1,601	2,425	25	96
	CA_HCL_L · Ca·HCl·VDLUFA (as CaO)	mass%	2,068	0,176	8,50 %	0,027	1,31 %	2,4	1,730	2,435	13	52
	S_HCL_EU · S·HCl·EU (as S)	mass%	2,223	0,102	4,60 %	0,035	1,55 %	1,3	2,023	2,432	27	104
	S_HCL_L · S·HCl·VDLUFA (as S)	mass%	2,157	0,099	4,59 %	0,020	0,91 %	1,3	1,964	2,360	15	60
	AS_EU · As·aqua regia·EU (as As)	mg/kg	2,501	1,264	50,53 %	0,095	3,80 %	3,6	0,579	5,830	16	62
	AS_L · As·aqua regia·VDLUFA (as As)	mg/kg	1,728	0,103	5,93 %	0,033	1,89 %	0,4	1,529	1,940	12	48
	U_EU · U·aqua regia·EU (as U)	mg/kg	2,379	0,199	8,36 %	0,050	2,09 %	0,6	1,996	2,795	10	39
	U_L · U·aqua regia·VDLUFA (as U)	mg/kg	2,318	0,284	12,26 %	0,045	1,93 %	0,9	1,781	2,926	10	40
	DRY_EU · Dry matter·EU (as dm)	mass%	98,074	0,812	0,83 %	0,056	0,06 %	-	96,457	99,705	22	86
	DRY_L · Dry matter·VDLUFA (as dm)	mass%	97,856	0,410	0,42 %	0,040	0,04 %	-	97,037	98,678	14	56