



Association of German Agricultural Analytic and Research Institutes

## EU FERTILISER RING TEST Q7/2015: LIQUID MICRONUTRIENT FERTILISER ‘MULTIMICRO® FLUID’

In 2015, the Association of German Agricultural Analytic and Research Institutes (VDLUFA e. V.) carried out an international ring test to determine the major and minor components in a liquid micronutrient fertiliser ‘Multimicro® fluid’. The purpose of this fertiliser ring test was to offer a platform to laboratories in all EU countries for the testing and documentation of their respective analytical quality. 40 laboratories from 16 European countries took part in this ring test with the designation EU Q7/2015.

The analytes to be reported by the participating laboratories had to be determined by various official or standardised methods (see Table 1). The laboratories were asked to prepare the samples for analysis according to Annex IV, Section B, Method 1 (sample preparation) of ‘Regulation (EC) No. 2003/2003 of the European Parliament and of the Council of 13 October 2003 relating to fertilisers’. The results obtained for each individual determination were to be reported in the unit (mass%) given in the table above based on fresh matter (fm).

**Table 1: Analytes to be determined and methods used**

No	Analyte	Method Digestion / Extraction	Method Final determination	Unit	Comments
1	MgO-EU	EU 8.3	EU 8.7 or 8.8	mass %	reported as MgO
2	S-EU	EU 8.3	EU 8.9	mass %	reported as S
3	B-EU	EU 9.2	EU 9.5	mass %	reported as B
4	Cu-EU	EU 9.2	EU 9.7	mass %	reported as Cu
5	Fe-EU	EU 9.2	EU 9.4 or 9.8	mass %	reported as Fe
6	Mn-EU	EU 9.2	EU 9.4 or 9.9	mass %	reported as Mn
7	Mo-EU	EU 9.2	EU 9.10	mass %	reported as Mo
8	Zn-EU	EU 9.2	EU 9.4 or 9.11	mass %	reported as Zn
9	Cl-EU	EU 6.1	EU 6.1	mass %	reported as Cl
1a	MgO-ICP/OES	EU 8.3	e.g. VDLUFA(II.1) 4.2.4	mass %	reported as MgO
2a	S-ICP/OES	EU 8.3	e.g. VDLUFA(II.1) 4.2.4	mass %	reported as S
3a	B-ICP/OES	EU 9.2	e.g. VDLUFA(II.1) 8.10	mass %	reported as B
4a	Cu-ICP/OES	EU 9.2	e.g. VDLUFA(II.1) 8.10	mass %	reported as Cu
5a	Fe-ICP/OES	EU 9.2	e.g. VDLUFA(II.1) 8.10	mass %	reported as Fe
6a	Mn-ICP/OES	EU 9.2	e.g. VDLUFA(II.1) 8.10	mass %	reported as Mn
7a	Mo-ICP/OES	EU 9.2	e.g. VDLUFA(II.1) 8.10	mass %	reported as Mo
8a	Zn-ICP/OES	EU 9.2	e.g. VDLUFA(II.1) 8.10	mass %	reported as Zn
9a	Cl-ICP/OES	EU 6.1	e.g. VDLUFA(II.1) 4.2.4	mass %	reported as Cl

EU (2003): Regulation (EC) No 2003/2003 of the European Parliament and of the Council of 13th October 2003 relating to fertilisers.

VDLUFA (II.1): Verband Deutscher Landwirtschaftlicher Untersuchungs- und Forschungsanstalten (VDLUFA) (Ed.), 1995-2011: Handbuch der Landwirtschaftlichen Versuchs- und Untersuchungsmethodik, Vol. II.1, Die Untersuchung von Düngemitteln (Fertiliser Analysis), VDLUFA-Verlag, Darmstadt.

For analysing Mg, S, B, Cu, Fe, Mn, Mo, Zn and Cl, final determination by ICP-OES (inductively coupled plasma optical emission spectrometry) was accepted as an alternative method to EU-official methods. For these elements determination by ICP-OES is an official method in the 'German National Fertiliser Regulation' (DüMV, 2012), but not in EU Regulation No. 2003/2003.

The statistical evaluation was performed with 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 cases where a sufficient number of results had been reported. We used the validated software package ProLab for all statistical calculations.

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 Fertiliser Ring Test EU Q7/2015

Sample	Measurand	Unit	Mean	Reprod.S.D.		Repeat.S.D.		HorRat	Limit of Tolerance		Number of Laboratories	Values
				Abs.	Rel.	Abs.	Rel.		Lower	Upper		
·MULTI	MgO_EU · MgO-EU 8.3 (as MgO)	mass%	3,546	0,168	4,75 %	0,018	0,50 %	1,4	3,209	3,900	16	57
	S_EU · S-EU 8.3 (as S)	mass%	5,451	0,072	1,33 %	0,025	0,46 %	0,4	5,304	5,600	15	55
	B_EU · B-EU 9.2 (as B)	mass%	0,295	0,015	5,25 %	0,004	1,50 %	1,1	0,264	0,327	12	47
	CU_EU · Cu-EU 9.2 (as Cu)	mass%	0,508	0,016	3,06 %	0,002	0,43 %	0,7	0,477	0,541	14	52
	FE_EU · Fe-EU 9.2 (as Fe)	mass%	1,082	0,051	4,72 %	0,010	0,92 %	1,2	0,980	1,189	14	51
	MN_EU · Mn-EU 9.2 (as Mn)	mass%	1,490	0,040	2,69 %	0,012	0,80 %	0,7	1,409	1,574	14	51
	MO_EU · Mo-EU 9.2 (as Mo)	mass%	0,011	0,001	11,37 %	0,000	1,27 %	1,4	0,009	0,014	5	19
	ZN_EU · Zn-EU 9.2 (as Zn)	mass%	1,117	0,035	3,15 %	0,008	0,72 %	0,8	1,046	1,190	14	51
	CL_EU · Cl-EU 6.1 (as Cl)	mass%	0,037	0,029	78,26 %	0,001	3,68 %	11,9	0,005	0,117	10	40
	MGO_ICP · MgO:ICP/OES VDLUFA (as MgO)	mass%	3,495	0,117	3,34 %	0,030	0,85 %	1,0	3,259	3,739	29	114
	S_ICP · S:ICP/OES VDLUFA (as S)	mass%	5,384	0,134	2,49 %	0,036	0,66 %	0,8	5,112	5,662	29	111
	B_ICP · B:ICP/OES VDLUFA (as B)	mass%	0,300	0,012	4,13 %	0,003	0,98 %	0,9	0,275	0,326	30	116
	CU_ICP · Cu:ICP/OES VDLUFA (as Cu)	mass%	0,514	0,015	2,97 %	0,004	0,85 %	0,7	0,483	0,546	30	117
	FE_ICP · Fe:ICP/OES VDLUFA (as Fe)	mass%	1,069	0,043	4,06 %	0,010	0,95 %	1,0	0,982	1,160	30	117
	MN_ICP · Mn:ICP/OES VDLUFA (as Mn)	mass%	1,501	0,058	3,88 %	0,014	0,93 %	1,0	1,383	1,622	30	117
	MO_ICP · Mo:ICP/OES VDLUFA (as Mo)	mass%	0,011	0,001	6,11 %	0,000	0,78 %	0,8	0,010	0,013	27	105
	ZN_ICP · Zn:ICP/OES VDLUFA (as Zn)	mass%	1,119	0,037	3,30 %	0,011	0,97 %	0,8	1,044	1,196	30	117
	CL_ICP · Cl:ICP/OES VDLUFA (as Cl)	mass%	0,051	0,009	18,48 %	0,002	4,44 %	3,0	0,033	0,073	8	30



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