



Association of German Agricultural Analytic and Research Institutes

## Terms of Participation

### VDLUFA EU-Fertiliser Ring Test – EU Q10 / 2018

By organising the EU-Fertiliser Ring Test, VDLUFA offers a quality assurance program concerning the analysis of nutrients and pollutants in fertilisers. It particularly addresses accredited laboratories which have been notified by the EU-Commission for fertiliser analysis, but this interlaboratory test is also open to any laboratory worldwide. VDLUFA membership is not necessary to participate in the VDLUFA EU-Fertiliser Ring Test! An important aim of the test is to offer fertiliser laboratories a platform for testing and documentation of their respective analytical quality.

The EU-Fertiliser Ring Test is carried out by the VDLUFA section III “Fertiliser Analysis” in co-operation with the head office of VDLUFA.

The main objective of the test is the continuous review of the reproducibility of the test results of participating laboratories involved in the official fertiliser monitoring. This includes the monitoring of legal tolerances.

#### **Materials to be analysed: Potassium Chloride 60 (60er Kali®) Organic NPK Fertiliser (LawnFertiliser)**

Both materials are taken from a commercially available fertiliser batch proposed for direct use. They have been homogenised, divided and filled into (PE) plastic bottles. The homogeneity has been verified by Thüringer Landesanstalt für Landwirtschaft (TLL), Naumburger Straße 98, 07743 Jena, Germany.

#### **Schedule**

- 08-01-2018: Start of registration
- 02-02-2018: Registration deadline
- 09-02-2018: Dispatch of fertiliser samples and invoices
- 16-02-2018: Dispatch of files for reporting data
- 16-03-2018: Submission deadline for the analysis data
- 02-07-2018: Dispatch of test reports and certificates of participation

#### **Participation fees**

The participation fee for the “Fertiliser Ring Test” amounts to:

- Euro 500.00 (non-members of VDLUFA e. V.)
- Euro 250.00 (members of VDLUFA e. V.)

**plus** the applicable value-added tax (VAT) **and** forwarding expenses. The participation fee includes a test

sample of each fertilizer of approximately **450 g** for Potassium Chloride 60 (60er Kali®) and **280 g** for the Organic NPK fertiliser (LawnFertiliser). The forwarding or selling of the test samples to third parties is not permitted.

## Execution Instructions

### Sample preparation

Sample preparation for Potassium Chloride 60 (60er Kali®) has to be executed according to EN 1482-2:2007: Fertiliser and liming materials - Sampling and sample preparation - Part 2: Sample preparation.

Sample preparation for Organic NPK fertiliser (LawnFertiliser) has to be executed according to an ISO or national method, e.g. VDLUFA (II.2) 2.

### Sample analysis

The participating laboratories should analyse the fertiliser in accordance with various official or standardised methods (Table 1 and Table 2).

Table 1: Parameters to be determined in Potassium Chloride 60 (60er Kali®) and methods to be applied

No	Analyte	Method for extraction	Method for determination	Unit	Comments
1a	K-water-EN	EN 15477	EN 15477	mass %	reported as K <sub>2</sub> O
1b	K-water-others	national or ISO methods, e.g. VDLUFA (II.1) 5.1.1  in-house methods	national or ISO methods, e.g. VDLUFA (II.1) 4.2.4 (ICP-OES)  in-house methods	mass %	reported as K <sub>2</sub> O
2a	Cl-water-EN	EN 16195	EN 16195	mass %	reported as Cl
2b	Cl-water-others	national or ISO methods, e.g. VDLUFA (II.1) 9.8.1  in-house methods	national or ISO methods, e.g. VDLUFA (II.1) 9.8.2 (pot. titration)  in-house methods	mass %	reported as Cl

VDLUFA (II.1): Verband Deutscher Landwirtschaftlicher Untersuchungs- und Forschungsanstalten (VDLUFA) (Ed.), 1995-2014: Handbuch der Landwirtschaftlichen Versuchs- und Untersuchungsmethodik, Vol. II.1, Die Untersuchung von Düngemitteln (Fertiliser analysis), VDLUFA-Verlag, Darmstadt.

Table 2: Parameters to be determined in Organic NPK fertiliser (LawnFertiliser) and methods to be applied

No	Analyte	Method for extraction	Method for determination	Unit	Comments
1a	N-total-EN	EN 13654-1	EN 13654-1	mass %	reported as N
1b	N-total-others	national or ISO methods  in-house methods	national or ISO methods, e.g. VDLUFA (II.2) 3.1.1 (elemental analysis)  in-house methods	mass %	reported as N
2	NH <sub>4</sub> -N	national or ISO methods, e.g. VDLUFA (II.2) 3.7.1.1  in-house methods	national or ISO methods, e.g. VDLUFA (II.2) 3.7.1.1  in-house methods	mass %	reported as NH <sub>4</sub> -N

No	Analyte	Method for extraction	Method for determination	Unit	Comments
3	P <sub>2</sub> O <sub>5</sub> -total	EN 13650  national or ISO methods, e.g. VDLUFA (II.2) 5.1.1.1  in-house methods	ICP-OES e.g. EN 11885  national or ISO methods, e.g. VDLUFA (II.2) 3.2.2.2 (ICP-OES)  in-house methods	mass %	reported as P <sub>2</sub> O <sub>5</sub>
4	K <sub>2</sub> O-total	EN 13650  national or ISO methods, e.g. VDLUFA (II.2) 5.1.1.1  in-house methods	ICP-OES e.g. EN 11885  national or ISO methods, e.g. VDLUFA (II.2) 3.3.2.2 (ICP-OES)  in-house methods	mass %	reported as K <sub>2</sub> O
5	MgO-total	EN 13650  national or ISO methods, e.g. VDLUFA (II.2) 5.1.1.1  in-house methods	ICP-OES e.g. EN 11885  national or ISO methods, e.g. VDLUFA (II.2) 3.5.2.2 (ICP-OES)  in-house methods	mass %	reported as MgO
6	CaO-total	EN 13650  national or ISO methods, e.g. VDLUFA (II.2) 5.1.1.1  in-house methods	ICP-OES e.g. EN 11885  national or ISO methods, e.g. VDLUFA (II.2) 3.4.2.2 (ICP-OES)  in-house methods	mass %	reported as CaO
7	Na-total	EN 13650  national or ISO methods, e.g. VDLUFA (II.2) 5.1.1.1  in-house methods	ICP-OES e.g. EN 11885  national or ISO methods, e.g. VDLUFA (II.2) 4.6.2.2 (ICP-OES)  in-house methods	mass %	reported as Na
8	SO <sub>3</sub> -total	EN 13650  national or ISO methods, e.g. VDLUFA (II.2) 5.1.1.1  in-house methods	ICP-OES e.g. EN 11885  national or ISO methods, e.g. VDLUFA (II.2) 3.6.2.2 (ICP-OES)  in-house methods	mass %	reported as SO <sub>3</sub>
9	Cu	EN 13650  national or ISO methods, e.g. VDLUFA (II.2) 5.1.1.1  in-house methods	ICP-OES e.g. EN 11885  national or ISO methods, e.g. VDLUFA (II.2) 3.8.1 (ICP-OES)  in-house methods	mass %	reported as Cu
10	Zn	EN 13650  national or ISO methods, e.g. VDLUFA (II.2) 5.1.1.1  in-house methods	ICP-OES e.g. EN 11885  national or ISO methods, e.g. VDLUFA (II.2) 3.8.1 (ICP-OES)  in-house methods	mass %	reported as Zn

No	Analyte	Method for extraction	Method for determination	Unit	Comments
11a	dry matter (d.m.) content		EN 12048	mass %	reported as dry matter (d.m.) content
11b	dry matter (d.m.) content		national or ISO methods, e.g. VDLUFA (II.2) 9.1  in-house methods	mass %	reported as dry matter (d.m.) content
12a	ignition loss-EN		EN 13039	mass %	reported as content in dry matter (d.m.)
12b	ignition loss-others		national or ISO methods, e.g. VDLUFA (II.2) 4.1  in-house methods	mass %	reported as content in dry matter (d.m.)
13a	Corg-EN		EN 15936	mass %	reported as content in dry matter (d.m.)
13b	Corg-others		national or ISO methods, e.g. VDLUFA (II.2) 4.2  in-house methods	mass %	reported as content in dry matter (d.m.)
14	Cr	EN 13650  national or ISO methods, e.g. VDLUFA (II.2) 5.1.1.1  in-house methods	ICP-OES e.g. EN 11885  national or ISO methods, e.g. VDLUFA (II.2) 5.1.2.3.2 (ICP-MS)  in-house methods	mg/kg d.m.	reported as Cr
15	Ni	EN 13650  national or ISO methods, e.g. VDLUFA (II.2) 5.1.1.1  in-house methods	ICP-OES e.g. EN 11885  national or ISO methods, e.g. VDLUFA (II.2) 5.1.2.5.2 (ICP-MS)  in-house methods	mg/kg d.m.	reported as Ni

VDLUFA (II.2): Verband Deutscher Landwirtschaftlicher Untersuchungs- und Forschungsanstalten (VDLUFA) (Ed.), 2000-2014: Handbuch der Landwirtschaftlichen Versuchs- und Untersuchungsmethodik, Vol. II.2, Die Untersuchung von Sekundärrohstoffdüngern, Kultursubstraten und Bodenhilfsstoffen (Analysis of Fertilisers based on Waste Materials), VDLUFA-Verlag, Darmstadt.

Methods are to be reported with the results. **Please give information on the procedure you actually used if more than one procedure is described in a method document of standard for the same analyte.**

### Registration to VDLUFA EU-Fertiliser Ring Test

To register for the current program, please use the attached PDF registration form.

The form should be filled out on the computer, printed and signed. The signed registration form is either to be scanned and send to [info@vdlufa.eu](mailto:info@vdlufa.eu) or to be faxed to +49 6232 136 122.

### Implementation of the Test

Identical fertiliser samples are sent to the laboratories which have registered for the current test cycle.

The participating laboratories are asked to analyse a specified number of chemical parameters using official or standardised methods within the defined timeline.

For each parameter the **results of 4 determinations** have to be reported.

### **Assignment of Laboratory Identification Numbers**

All data are handled anonymously using assigned laboratory identification numbers. Each institution receives a laboratory identification number, which is necessary for reporting the data. The laboratory identification number will be sent to the laboratory together with the samples.

### **Reporting the results**

For reporting analytical results to VDLUFA, a file and an instruction sheet are sent to each participant via email. The file should be edited using the freeware program RingDat4 and according to the given instructions. The analysis data are to be returned as an email attachment.

The results obtained for each individual determination are to be reported in the units (mass % based on fresh matter or mg/kg dry matter (d.m.) given in the table above.

Results are to be reported with 4 significant digits, i.e. either four decimal places if the result is smaller than 1 (example: 0.1234); three decimal places if the result is one-place (example: 1.234); two decimal places if the result is two-place (example: 12.34); one decimal place if the result is three-place (example: 123.4); or no decimal place if the result is four-place or larger (example: 1234 or 12340).

**The data must be submitted on 16 March 2018 at the latest.**

### **Statistics**

The statistical evaluation of the test results, including the calculation of laboratory means, analytical uncertainties and a summarising statistical evaluation, is carried out using the validated software package "ProLab". A robust method is applied (DIN 38402 A45, Q-method, Hampel estimate) and  $Z_u$ -scores (tolerance limit  $|Z_u| = 2.0$ ) are calculated as a bias estimate using IUPAC guidelines so that laboratories may gauge their performance relative to their peers. Horwitz ratios are calculated, if possible.

### **Participation Certificate and Final Test Results**

Each participating laboratory receives a certificate of participation in the VDLUFA EU-Fertiliser Ring Test. Additionally each participant receives a final report of the ring test which includes a statistical evaluation based on the anonymous participant data.