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To whom it might concern

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2nd July 2024

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Letter of invitation to the KTBL/VDLUFA Proficiency Test Biogas 2024/25

Dear Ladies and Gentlemen,

you herewith receive the letter of invitation to the annually Proficiency Test Biogas performed by Kuratorium für Technik und Bauwesen in der Landwirtschaft e.V. (KTBL) and VDLUFA Qualitätssicherung NIRS GmbH (VDLUFA).

As usual, the scope of the inter-laboratory test can be individually selected by each laboratory from the blocks fermentation test, raw nutrients and/or residual gas potential.

The planned schedule for this year's inter-laboratory campaign is as follows:

2 nd September 2024	Deadline of application
17 th /18 th September 2024	Shipping the samples to the laboratories
20 th January 2025	Analysis period respectively submission of the analysis data
February/March 2025	Submission of inter-laboratory test evaluation results

To the procedure of the inter-laboratory campaign:

The samples of the proficiency test should be handled / analysed as usual in your laboratory in compliance with VDI guideline 4630 or the VDLUFA method "Determination of biogas and methane yield in fermentation tests" for the scope "fermentation test". For the determination of the residual gas potential, the new VDLUFA method "Determination of the residual gas potential from digestate in the laboratory test" (VDLUFA Methods, Volume VII Environmental Analysis, Method 4.1.2) must be applied. In addition, values from the "home method" of the residue gas determination can also be submitted, which must be clearly marked as such.

All laboratory results must be submitted to the VDLUFA Quality Assurance NIRS GmbH for attention of Mr. Tillmann (E-Mail at peter.tillmann@vdlufa-nirs.de), by no later than 20th January 2025, for the evaluation process.

For the proficiency test each laboratory can extent its test individually. Please select by application from the following services.

Scope of analysis	Block	Parameter (Method)	Sample	Sample amount ¹⁾	Price
1	Gas yield	Biogas yield, methane yield, CH ₄ content (VDLUFA 4.1.1, Vol. VII)	<i>Main samples:</i> Maize silage (approx. 30 % DM), separated cattle slurry (solids, 50 % DM), microcrystalline cellulose, mixed feed ²⁾ <i>Additional sample:</i> Cattle slurry	Maize silage, solids separated cattle slurry, and mixed feed approx. 1 kg DM each cellulose 500 g cattle slurry 10 l	EUR 500,-
	VS block	DM, crude ash, VS (VDLUFA Vol. III)	As above	incl.	incl.
	Fermentation acids	Lactic acid, acetic acid, ethanol (other)	Silage from block "gas yield"	incl.	incl.
2	Macro ingredients ³⁾	crude protein, crude starch, others (free)	2 Maize silages (fresh, approx. 30 % DM and dried) ⁴⁾	fresh approx. 300 g DM each dried, ground 100 g	EUR 200,-
		pH-value, NH ₄ -N, ethanol, acids	As above	incl.	incl.
3	Residual gas	Residual gas yield (20/37°C), CH ₄ content (draft VDLUFA method)	Fermentation residue	10 l	EUR 300,-
	Fatty acids	C2-C5 VFA, recovery rate, FOS/TAC (free)	Fermentation residue, maize silage (s. above)	spikes residue 500 ml fatty acid mixture 100 ml	incl.

Additional freight costs

Over-night for non-Germany locations

EUR 150,-

Annotation 1: If you need more material, please let us know. You might be charged the additional costs for dispatching the material.

Annotation 2: This is a homogenized, natural material with a known calorific value.

Annotation 3: The examination of the ingredients (Weender / -van Soest analysis) is offered. The aim of this part of the inter-laboratory test is to check the drying-grinding-analysis chain in the trial.

Annotation 4: dried at 60°C, grinded (1 mm)

At the end of the test the participating laboratories will receive a quality certificate on the "successful" participation. However, the determination of "successful" refers only to the correct determination of the gas yields (scope of analysis: fermentation test) and the analysis of the 4

main samples (maize silage, straw pellets, cellulose, and mixed feed). Neither the parameters "residual gas potential" and "macro ingredients" are considered here, nor the additional sample cattle slurry, which is newly introduced in the "gas yield" block.

The following quality criteria must be observed in order to obtain the certificate:

- All 4 main samples must be analysed.
- For a successful determination of the methane yields, at least 3 of 4 samples need to obtain a z-scores of >-2 and <2 .
- The target methane yield for micro-crystalline cellulose of 368 l_N +/- 30 l_N/kg VS (+/- 8 %) has to be determined.
In addition, a quality seal on the certificate is granted if the methane yield of the laboratory falls within the range methane target +/- 5%. The regular certificate is granted based on the above range methane target +/- 8%.
- The laboratory's result for maize silage is allowed to deviate maximally +/- 10 % from the mean methane yield of all laboratories in the respective proficiency test year.
- The laboratory's internal variation for cellulose is not to exceed 6 %.
For all other samples an internal variation of not more than 5-6 % (for homogeneous material) and 10 % (inhomogeneous material), respectively, is recommended. For the certificate, however, only the internal variation of cellulose is considered.

The questionnaire on the procedure for the analysis of the BMP test, now integrated in the evaluation file, is an elementary component of the inter-laboratory test. Only laboratories that submit a plausibly completed questionnaire are entitled to a certificate. If ambiguities in the evaluation should occur, the laboratory is obliged to provide Mr. Tillmann with information about the detailed procedure on request. Only in this way the overall results of the proficiency test can be improved in the long term.

The certificate will be sent to the appropriate laboratories after the final meeting.

The inter-laboratory evaluation is anonymised - each participating laboratory receives an individual laboratory identification in advance. The evaluation report will be sent electronically to all participants after completion of the inter-laboratory test with all details.

The KTBL Working Group "Inter-laboratory Test" has agreed to publish, if appropriate, targeted results of the inter-laboratory tests (anonymised). By signing up for the Proficiency Test Biogas, you agree that your anonymous results of the lab / data of the inter-laboratory test may be used by the organizers for publication reasons.

Participants of the inter-laboratory test biogas bear the cost of the sample materials, analysis or final meeting (optional) - the indicated prices are plus 19 % VAT. The organization of the inter-laboratory test is free of charge.

The costs for the sample material (the indicated prices are plus 19% VAT) are borne by the participants, the organization of the interlaboratory test is still free of charge for you. The costs incurred by the participants, e.g. for sample analyses or travel costs for the final round-robin meeting, are carried by the respective laboratories themselves.

If you wish to participate at the inter-laboratory campaign Biogas Potential Test, please send your application form until 2nd September 2024 as a binding confirmation with details of the scope of the ring test (fermentation test, macro ingredients, residual gas potential) and required sample volume and the contact details of the competent person and delivery address at u.roth@ktbl.de as well as to m.paterson@ktbl.de.

For further information on KTBL/VDLUFA Proficiency Test Biogas please visit www.ktbl.de/themen/proficiencytest.

We would be happy to confirm your participation and are at your disposal for any further questions.

With best regards,

Handwritten signature of Ursula Roth in black ink.

Ursula Roth and
Managers of KTBL-Workgroup
"Inter-laboratory Test Biogas"

Handwritten signature of Mark Paterson in purple ink.

Mark Paterson