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# Transferability of laboratory results on methane yield to full-scale biogas plants

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**Abstract:** The cost-effectiveness of biogas plants is largely determined by the substrate costs, which constitute 40 to 60% of the annual operating costs.in Germany, where energy crops are widely used in anaerobic digesters. When planning and operating digesters, understanding the relationship between the methane yield of different substrates in the lab compared to full-scale operating biogas plants is very important. The determination of the methane yield via batch tests using VDI guideline 4630 is being conducted by approximately 40 different laboratories in Germany.

Our results of methane yields show relatively good agreement between laboratory testing and full-scale biogas plant. When the substrate input are measured well and the residual methane potential of the fermentation residue is measured, we have shown, that laboratory methane yields can be used for planning biogas plants. The biogas yield guidelines compiled by the German agency KTBL based on laboratory values from several well-known biogas laboratories and institutes were shown to be accurate and appropriate for the preliminary economic planning of future biogas plants.

In addition to the laboratory value of the specific methane yield, other parameters also play an important role in the planning of biogas plants (e.g. hydraulic retention time HRT, organic loading rate OLR, substrate mixture), and the accuracy of the measurement technology used (e.g. gas meter, gas quality determination, scales).

Increasingly, laboratories are validating methane yield results through inter-laboratory testing. To ensure the accuracy of the laboratory tests for the determining methane yield, regular participation in these types of inter laboratory tests, along with laboratory internal validation using standard substrates, should become the standard practice to ensure high-quality and replicable results.

**Keywords:** Biogas, laboratory tests, planning of biogas plants, round robin tests

## 1. What are fermentation tests required for?

The economic efficiency of biogas plants is largely determined by the substrate costs. Approx. 40% to 60% of the annual costs of a biogas plant, feeded with energy crops, are attributable to substrate procurement. It is therefore essential to calculate standard figures when planning a biogas plant. When using liquid manure and by-products it is also essential to know the specific methane yield of the substrates used in order to enable optimum planning of a biogas plant. In addition to the data on the specific methane yield, further information on the fermentation substrate used is also required. In particular, the yield per unit area of fresh matter, the dry matter yield and the quality of the fodder have a considerable influence on the amount of methane that can be achieved.

In principle, the content of carbohydrates, fats and proteins correlates directly with the energy content of the substrate used and should actually be sufficient as a basis for calculating the methane yield. There are also a number of methods for this estimation. Starting with the calculation by Buswell (1936), who estimated the biogas yield and the methane and carbon dioxide content in the biogas on the basis of the elementary composition of the substrate, calculations were made to determine the nutrient composition by Baserga (1998) and later Keymer & Schilcher (1999). These values can

only be used as a rough guide for simple planning of full scale biogas plants. As Czepuck et al. (2006) proved in comparison studies, the results deviate by 10 % to 20 % from the measured value in the biogas yield test. Since the plant mass is composed of very different nutrient combinations, further estimation formulas, e.g. according to Amon et al. (2003) and Kaiser (2007), as well as the laboratory determination of the fermentable volatile solid (FVS) value (Weissbach, 2012) lead only to a limited extent to reliable results.

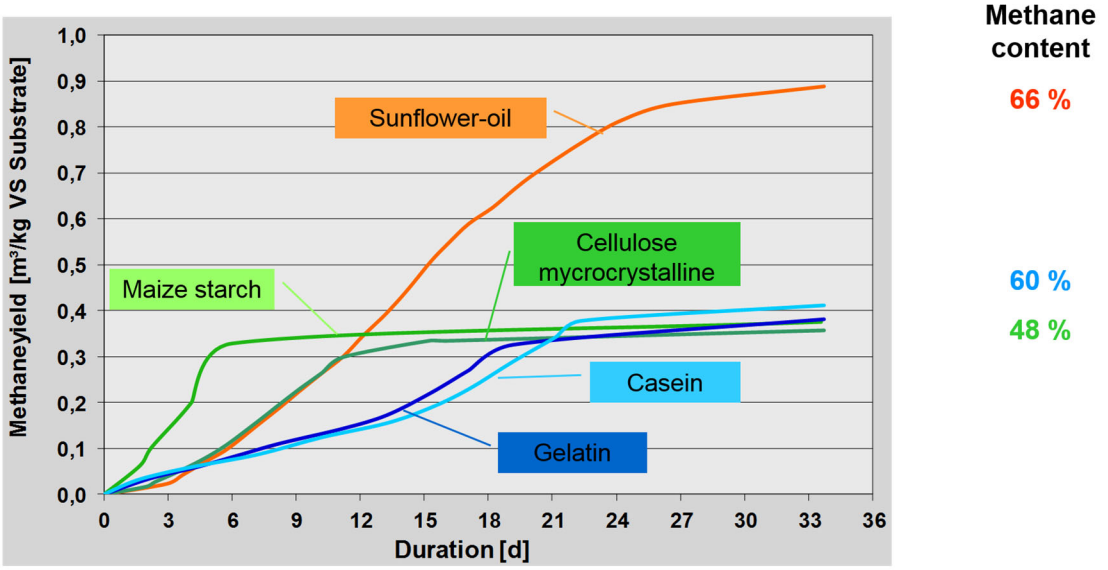


Figure 1. Methane yields and formation kinetics of different nutrient fractions (according to Czepuck et al., 2006)

Above all, the different composition of the carbohydrate fraction and its extremely different digestibility have a clear effect on the degradability, the degradation kinetics and finally the achievable gas yield. While starch can be completely converted into biogas in about 5 days as the plant's energy store, the conversion of plant supporting tissues such as hemicellulose and cellulose is significantly slower. Especially if a high proportion of lignocellulose complexes is present, this has a negative effect on the methane yield of the entire biomass and its degradability. Therefore a fermentation test is the most exact and

recommended method for the exact determination of the methane yield of plant mass (Czepuck et al., 2006).

**2. Benchmark on the methane yield of common substrates**

For the use of common substrates, KTBL (Advisory board for technology and construction in agriculture e.V.) now has numerous gas carrying guideline values, which have been compiled with the help of a large number of individual results from several well-known laboratories and research institutions (KTBL, 2011). As a rule, these reference values are extremely helpful and very useful for the design and planning of biogas plants.

However, it must be noted that depending on the location, variety, vegetation course of the year, harvest time and storage/silification, significant deviations from the standard values are possible. For this reason, laboratory analysis in the biogas laboratory is useful in many cases for determining the methane yield.

**3. Guidelines for carrying out fermentation trials**

Many guidelines are available to get a higher reproducibility of BMP. Beside European standards (Holliger et al., 2016), VDI 4630 is the most common guideline used in Germany. The guideline 4630 (VDI, 2016) describes both, the procedure for carrying out batch and continues tests. The KTBL working group "Interlaboratory Tests" developed together with the VDLUFA, the VDLUFA method guideline for "Determination of the biogas and methane yield in fermentation tests" (VDLUFA, 2011), which somewhat simplifies the most important criteria for carrying out fermentation tests. The aim of this method is to improve the quality assurance of laboratory tests on biogas yield.

The regulations stipulate that a minimum number of test conditions must be satisfied:

- Use of suitable, gas-tight and tempered "small fermenters"
- Use of a suitable inoculum (either specially bred or fermentable material from practice biogas fermenters; starving the inoculum before starting)

- Mixing ratio of inoculum and fermentation substrate (volitale solid (VS)-related) in a ratio of at least 2:1 to find sufficient buffer capacity in the fermentation batch
- Inoculum ferment in parallel as a zero sample
- At least one standard substrate is also fermented, e.g. microcrystalline cellulose or internal laboratory standards
- Fermentation temperature usually 37°C ± 1°C
- Measure biogas formation as often as possible and determine the methane content with each gas extraction
- At least 25 days fermentation time. Termination criterion for the test batch if less than 0.5 % of the gas quantity formed to date is produced on at least 3 consecutive days
- Reference of the methane yield to the VS input in the test substrate
- If silages or substrates with volatile components (fermentation acids, alcohols) are used, the value of the VS content must be corrected (Mukengele et al., 2006)
- Standardization of gas production (0°C, 1013 hPa); consideration of water vapor

In the evaluation, the inoculum's own production of biogas/methane is deducted from the total production in order to determine only the biogas/methane yield of the sample tested. The amount of biogas/methane produced over the experimental time is shown as a sum curve.

**4. Interlaboratory comparisons to ensure test quality**

In Germany, at least 40 laboratories now offer tests to determine the methane yield of substrate samples. These laboratories are made up of very experienced and also less experienced laboratories, which also involve the risk of errors creeping in due to the complex and multi-stage test procedures. In previous publications, the methane yields differ sometimes significantly from each other.

For this reason, a KTBL working group was established to carry out interlaboratory comparisons for biogas laboratories in cooperation with VDLUFA-NIRS GmbH and with the financial support of the BMELV. So far eleven round robin tests have taken place. The number of participating laboratories has been in the range of 20 to 33 laboratories. By evaluating the test results and a comprehensive description of the method as well as a detailed error analysis, the participants were able to identify and eliminate errors in their own procedures. All participating laboratories were required to comply with the VDI Guideline 4630 and the VDLUFA method regulation. Microcrystalline cellulose were used as the standard fermentation substrate for each pass. Beside that other fermentation substrates was chose. This substrates should cover the usual range of substrate variations from practice. Identical sample material was sent to all laboratories in all rounds. The samples were crushed wheat grain, dried maize, dried grass, maize silage, grass silage and rape press cake. When fresh silages were shipped, the effects of sample storage and sample homogenization on the final result were also possible to investigate.

It was noticeable, in the first run in 2006 the results for cellulose showed a relatively wide dispersion, although it was a standardized and very homogeneous test substrate. The comparative coefficient of variation of the methane yield between the laboratories was 19.5 % and 8.4 % in the last eleven test. When comparing the test setup and the results, it became clear that the differences were not due to the type and size of the respective test facilities. Rather, the accuracy of methane measuring instruments, their regular calibration, the mathematical evaluation under consideration of the reference variables for standard conditions and the consideration of water vapour correction in the event of deviations played a much more recognisable role.

**5. Influence of the mode of operation - transfer from batch to continuous operation**

With the exception of a few systems, biogas plants are not operated as a batch in practice. Instead of this it operate as continuous system with addition of fresh fermentation substrate several times a day. As a result, the process steps for anaerobic decomposition

of organic matter run parallel and simultaneously. While the batch approach shows a clear change in the composition of the biogas during the test sequence, the continuous approach hardly allows a difference in gas quality to be measured over the course of the day, if the same substrate is feeding continuesly. In addition, with fully mixed fermenters, but also with the so-called "plug flow fermenters", in close correlation with the hydraulic retention time of the substrate in the fermenter as well as with the fermenter geometry and the feeding frequency, certain parts of fresh or only partially degraded substrate always leave the fermenter before the hydraulic retention time has elapsed. This occurs even before their entire methane yield potential has been exhausted, which thus has an effect on the methane yield achieved.

Batch systems, on the other hand, wait until the fermentation process has almost completely decayed. For this reason, deviations are to be expected when batch results are transferred to continuous operation. Since today's biogas plants in Germany are equipped with very long hydraulic retention times, partly also due to legal requirements, and partly operated in cascade arrangement, the methane yield potential can possibly be fully exploited. Methane potentials could then be at the same level as methane yields determined by batch experiments. By determining the residual gas potential as in the overflow of biogas fermenters, it is relatively easy to check to what extent the fermentation substrate used is utilized or whether there is still an energy potential. Investigations on various practical biogas plants and sampling of fermenter cascades have shown that the residual gas potential correlates with the hydraulic retention time and the cascades (Oechsner et al., 2006). As a rule, only a very low residual gas potential (< 5 % when determining the residual gas potential at mesophilic temperature) was measurable for retention times of the fermenter (possibly including secondary fermenter) over 100 days. The results of the two federal measurement programs (FNR, 2005; FNR, 2009) confirm these statements. Intensively examinations at 25 biogas plants with different feedstuff and different digester cascades showed, that the transformability of laboratory results to practical plants are possible (Ruile, et al. 2015). In case of comparison of batch

to full-scale biogas plant, it seems like the influence of low data quality on the full-scale biogas plant results in a bigger mistake than the measurement error based on batch tests.
In an extensive trial at the University of Hohenheim, various fermentation substrates were investigated in a combination of batch tests and continuous trials in the biogas laboratory. For continuous trails the residual gas potential of the fermenter overflow was measured in batch test. In these tests, maize silage, ground wheat seed and a mixture of both substrates were fermented. The energy plants were gently dried and milled at 60°C to inhibited effects on pretreatment. The energy plants were then fermented in digesters. The substrates were supplemented with liquid manure (17 % VS content) to enable stable operating conditions. Liquid manure was also fermented alone as a control variant. In continuous operation, 15 horizontal fermenters each with a useful fermenter volume (FV) of 17 l were used with two organic loading rates (2.5 and 4.0 kg VS $m^{-3}$ FV $d^{-1}$). The hydraulic retention time in the continuous fermenters was 35 days at a fermentation temperature of 37 ± 2°C. For each variation two repetitions were applied and the experiments lasted over 123 days, (= more than 3 retention times). In addition to the amount of biogas and methane, the volatile fatty acid content and the FOS/TAC value were regularly monitored.
The fermenter operation was very stable in all investigated variants. Only the maize silage variant with a organic loading rate of 4 kg VS/$m^3_{FV}$d showed an increase in the fatty acids (HAC) to a maximum of 6,500 mg/l and a slight drop in the pH value to 7.2 from the 67th day of the experiment onwards. All other variants were stable (pH values 7.4 to 7.6) (see Mukengele, 2017).
The discharge (fermentation residue) of the continuous running fermenters was collected towards the end of the experiment (105th, 108th and 115th days) in order to determine the residual methane potential in the Hohenheim biogas yield test (HBT) at a fermentation temperature of 37 °C for 35 days. In addition, the methane yield of the fermentation substrates used as fodder was determined as standard in the HBT at 37 °C and a retention time of 35 days. This resulted in relatively high methane yields between 0.377 and 0.399

Nm³ methane per kg VS. Values between 4.7 and 5.3 kWh/kg VS were measured during the bomb calorimetric determination of the calorific value.

Fig. 2 shows a balance of the various experimental approaches. It can be seen, that with a high volume load (OLR 4) in continuous running digesters, especially with maize silage, less methane yield (81.0 % of the HBT potential) can be achieved than with a low loading rate (89.5 % with OLR 2.5). This also tends to be the case for cereals, but due to the very good degradability of ground cereal grain this is of little significance (89.7 % and 91.2 %).

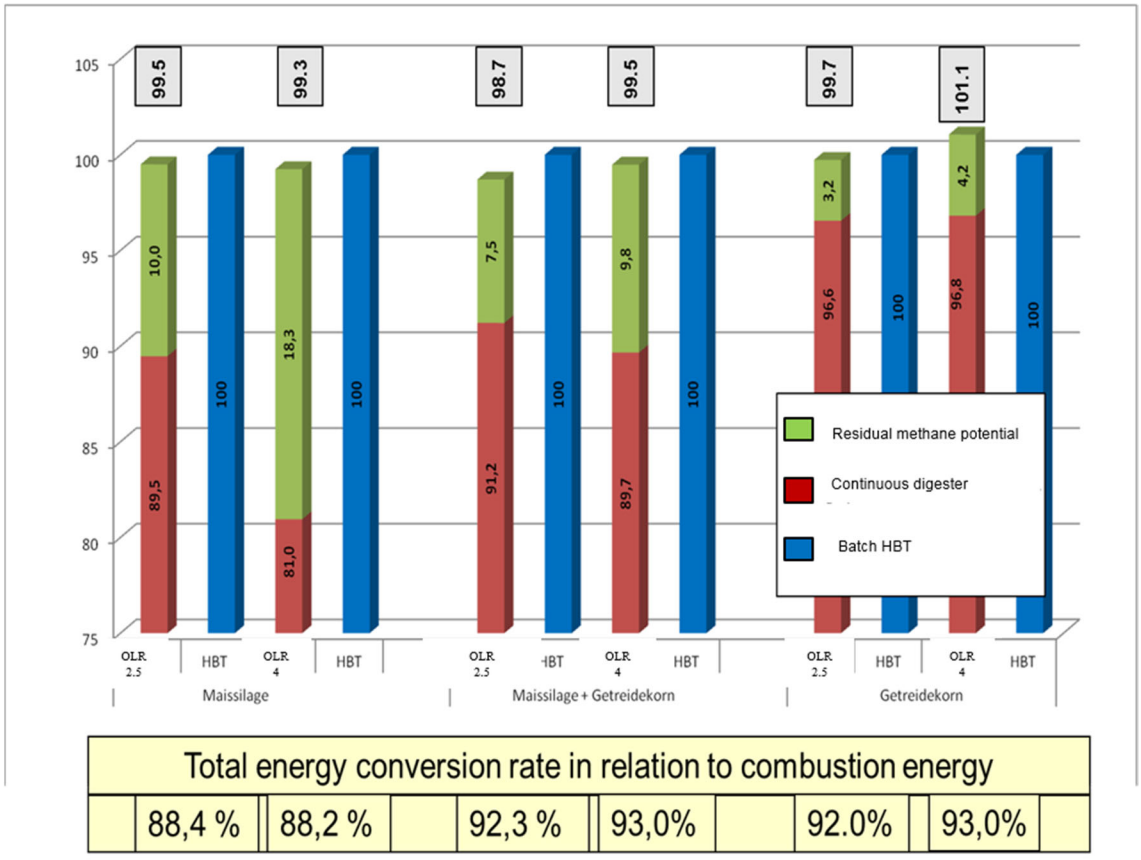


Figure 2. Methane yields from a continuous test at different OLR with subsequent determination of the residual methane potential in comparison to the methane yields from batch tests using the HBT method – comparative presentation

Resiudal gas potential and yields of the continuous tests results in near to the same methane potential like measured in batch test.

In total, values between 98.7 and 101.1 % result. This proves that the results from batch tests can be transferred in high precision to continuous tests. However, it is essential to ensure that part of the methane potential is flushed out of the fermenter via the fermentation residue. This is particularly noticeable with short retention times, as in the example shown (35 days). When recording the residual gas potential, the missing methane quantity can be detected. This is not to be expected when transferring the results to practice biogas plants with residence times of more than 100 days and series connection of several fermenters.

**6. Transferability of the batch results to practical plants**

Possible causes for deviations in the gas yields between laboratory and practice are the different fermentation conditions, e.g. the loading rate or the in practice "unpredictable" influences on the biological processes. An overview of selected process differences between batch test set-ups and biogas plants is shown in Table 1 below.

In the design of biogas plants, the ratio of fermenter size and volume load to the targeted CHP output plays an important role. In plant management, the daily supply of substrates through the substrates used and their gas yields is decisive. Uncertainties arising from the performance of fermentation tests and their evaluation and transferability to practical plants thus have a major impact on all the factors mentioned for biogas production (KTBL, 2011).

Possible deviations from the guideline values can result from the substrates, as they are required in large quantities for the biogas plant and their substrate properties can vary depending on the variety, harvest time and year of cultivation. For example, the substrate also changes its composition (DM/VS content, content of fermentation acids, pH value, ...) in the bunker silos over the storage period. The determination of the substrate input quantity is difficult despite existing weighing equipment, because the scales often do not have the required accuracy, bridging between the mixing vessel and the input screws occurs and because the individual substrates are often not recorded separately and exactly,

specially, if mixed silage as substrate used. The amount of liquid manure and other liquids (rainwater, silo leachate) is often not measured, too.

Table 1. Selected process differences between batch test setups and biogas plants

| Paramater | Batch-test | Continuous feeded full scale biogas plant |
|---|---|---|
| Digester volume | 100 ml – 15 l | > 1000 m³ |
| Operating mode | no exchange of substrate | daily exchange of material, substrate recirculation possible |
| Biological process | process stages run one after the other | process stages run in parallel |
| Organic loading rate | up to 50 g VS/l at the start of batch test | 2-5 g VS/l d |
| Hydraulic retention time | up to 35 days | > 150 days<br>digester cascade possible |
| Substrates | usually single substrate, representative, homogenized | mostly substrate mixture with different composition |
| Measuring method | exact weighing possible, exact determination of biogas quantity and quality | weighing equipment in practice often inaccurate<br>gas meter not calibrated and often inaccurate<br>gas quality often not recorded |

In the fermenter, process-related factors such as the mixture of substrates, their content of nutrients, especially trace nutrients, the biological environment in the fermenter, the retention time and the volume load also have an effect on the methane yield.

As with the implementation of measurement programs, e.g. Federal Measurement Programmes I and II. (FNR, 2005; FNR, 2009), there are in practice considerable problems in determining the biogas yield and especially the biogas quality. The electricity yield is often measured and estimated via the electrical efficiency of the CHP due to a lack of gas meters. The actual proportion of ignition oil and possibly own electricity consumers running through the meters must also be taken into account. In most cases, no

conversion to standard conditions is carried out when providing practical measurement data. This can cause an overestimation of the gas volume by up to 20 % (Ruile, et al, 2015).

**7. Practical data and batch tests**

The University of Hohenheim has a research biogas plant on a practical scale with an output of 350 kWel. This plant is equipped with two separate fermenter lines (800 m³ usable volume each), so that comparative investigations are possible. The plant is also high equipped. The measurement device are also calibrated frequently. It's guranteed a high quality of data. In a study carried out by Mönch-Tegeder (2013), an attempt was made to balance the data at this plant on a practical scale, similar to what had previously been shown during transmission in the laboratory. This biogas plant is intensively monitored and the addition quantity and quality of all input materials is precisely recorded and, as far as technically possible, remains constant over a long period of time. In the experiment, a relatively high proportion of liquid manure (50.0 %) was used (fresh mass). In addition, horse manure with other solid manure (23.6 %), maize silage (10.1 %), grass silage (8.8 %), cereal whole plant silage (4.6 %) and ground cereal grain (2.9 %) were used. The VS share of horse manure was 27.5 %. The substrate quality of all input materials was determined and a fermentation test for methane yield of all substrate was carried out. The observed fermenter is equipped with a mechanical processing technology (Cross flow grinder, MEBA, Nördlingen, Germany). The daily input quantity of fresh mass was 12.1 t/d with a standard deviation of 2.9 %. The organic loading rate was 2.49 kg VS per m³ FV and day, the HRT was 62.4 days.

With the aid of the weighed input quantities, their content of volitale solid and the laboratory values for the methane yield, the expected methane quantity of the fermenter was calculated and compared with the values measured by the gas meter of the fermenter. The results are shown in Fig. 3.

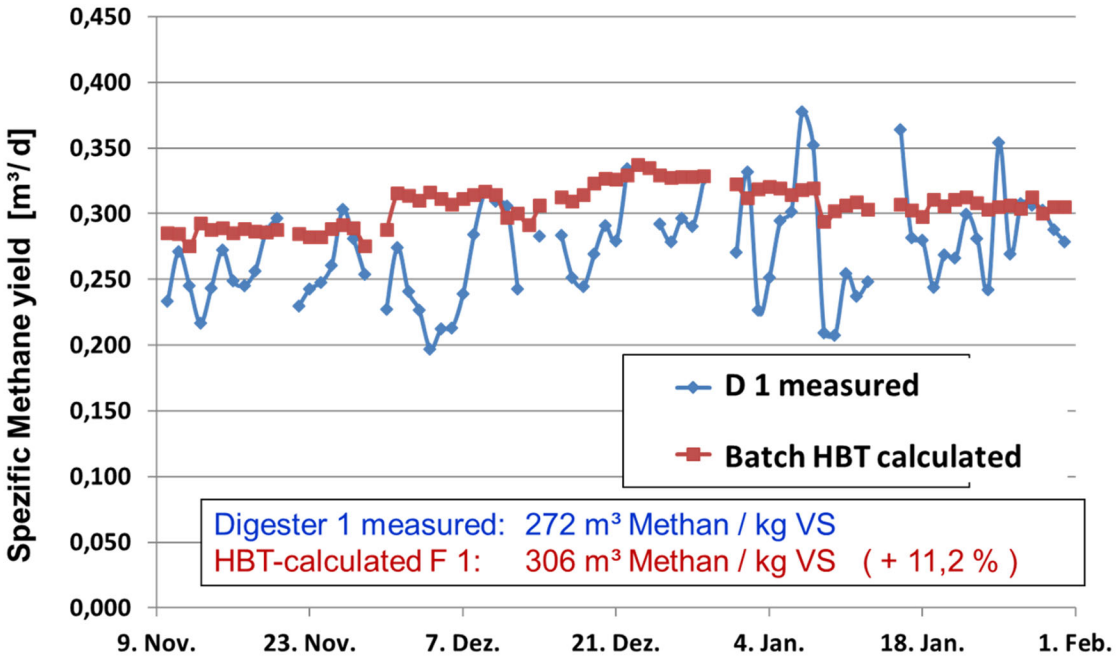


Figure 3: Comparison of the methane production determined by input quantity and HBT batch measurement with the actually measured methane production of the fermenter (Mönch-Tegeder, 2013)

During the 20-days observation phase there are certain fluctuations in the measured values, which are further scattered in the input quantities. This dispersion can be explained by the fact that the daily fluctuations of the VS input have a direct effect on the calculated value for methane formation and are thus clearly visible in the graph, while these fluctuations in the measured values are compensated by the substrate degradation in the fermenter spread over several days. The mean specific methane yield of the values calculated via HBT was on average 305 l $CH_4$/kgVS, that of the measured values only 2 % higher at 311 l $CH_4$/kgVS. This very small deviation between the methane yields determined from laboratory values and the measured values at the practical fermenter confirms that the laboratory values can be used very well for an estimate of the methane yield to be expected and thus for an economic efficiency estimate. This applies if a representative sample was analysed for the methane yield, the VS content of the substrate

input and its exact weight were recorded regularly and at short intervals. As a rule, as this last example has shown, inaccuracies are more likely to occur when determining the mass in practice than when determining the methane yield in the laboratory.

## 8. Conclusions

The determination of the methane yield by batch tests is now widely used in Germany and is carried out by at least 40 laboratories. Their quality is regularly assured by interlaboratory comparisons.

Examples in the laboratory and in practice have shown that there is a relatively good correlation between laboratory and practical values for the methane yield. This also proved that the gas yield guidelines compiled by the KTBL on the basis of laboratory values from several well-known biogas laboratories are important and indispensable for the economic preliminary planning of biogas plants.

In some cases, there are certain deviations from the assumed values at the biogas plant operated later. However, a large number of influencing factors affect the fermentation substrate used and its quality, but are also linked to the process and operating mode of the biogas plant.

The production of biogas is a microbial degradation process involving a large number of microorganisms. Here, certain deviations in the range of 5 to 10 % are always possible. In order to ensure the accuracy of the laboratory tests when determining the methane yield, regular participation in interlaboratory comparisons and constant internal laboratory testing using standard substrates should be a matter of course.

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