

Requirements for measurement of biochemical methane potential (BMP)

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February 27, 2020

1 DBFZ BMP methods collection

Document number 100. File version 1.1. This document is from the DBFZ BMP methods collection. For more information, visit the site at <https://www.dbfz.de/en/BMP>.

2 Introduction

This document presents the minimal requirements for measurement of biochemical methane potential (also called biomethane potential) (BMP) in batch tests, based on the consensus of more than 40 biogas researchers. The list of requirements is based on Holliger et al. [1], with some modifications of validation

criteria [2] and additional details on calculation standardization. For details and many additional recommendations, see these papers [1, 2].

3 Requirements for BMP measurement

3.1 Analysis of substrate and inoculum

Volatile solids (VS) content of inoculum and substrate is needed to determine quantities for a selected inoculum-to-substrate ratio (ISR) and for calculation of BMP.

1. Total solids (TS). Measure for the inoculum and all substrates, by drying for at least 24 hours at 105°C in triplicate.
2. Volatile solids (VS). Measure for the inoculum and all substrates by combusting the dried sample at 550°C for at least 2 hours in triplicate.

3.2 Test setup and duration

1. Samples. All BMP trials must include three types of samples: batches (bottles) with only inoculum (“blanks”), with microcrystalline cellulose as a positive control¹, and with substrate. Bottles with substrate (including positive controls) must contain inoculum.
2. Replication. All tests must include at least 3 batches (bottles) for each condition². The minimum number of batches (bottles) used in a BMP test with one substrate is therefore 9 (3 blanks, 3 cellulose, 3 substrate).
3. Duration. Terminate BMP tests only after daily CH₄ production from individual batches (bottles) during 3 consecutive days is < 1.0% of the net accumulated volume of methane from the substrate (substrate minus average of blanks). For manual or other methods where measurements are not made every day, termination can take place at the end of the first measurement interval of at least 3 days where the rate of production drops below the 1% maximum (or two or more intervals that sum to at least 3 days, all with rates below the 1% maximum). If different substrates are tested, each substrate can be terminated when the slowest of the 3 replicate batches (bottles) has reached the termination criterion. Blanks must be continued as long as the slowest (latest) batch (bottle) with substrate. Continuing tests beyond this 1% net duration is acceptable and can help ensure that validation criteria are met (Section 3.4).

¹ Other positive control substrates could be used in the future, but only cellulose has had extensive testing that was used to develop the validation criteria described below [2].

² If a bottle is lost through, e.g., breakage, resulting in $n = 2$ for any condition, results cannot be validated. Therefore it is prudent to include 4 blanks. Outliers can be eliminated if there is good reason to suspect there was an error in measurement (e.g., leakage) but the remaining number of replicates must be 3.

3.3 Calculations

1. Data processing. Standardized CH_4 volume (dry, 0°C , 101.325 kPa) are calculated from laboratory data using standardized methods, if available.³
2. BMP units. BMP should be expressed in standardized CH_4 volume (dry, 0°C , 101.325 kPa, referred to as “normal” volume) per unit mass of substrate VS added (often written as $\text{NmL}_{\text{CH}_4} \text{ gVS}^{-1}$).
3. Calculation of BMP. BMP of all substrates (including cellulose) is calculated by subtracting inoculum CH_4 production (determined from blanks) from gross (total) CH_4 production from substrate with inoculum, and normalizing by substrate VS mass. Calculations must follow a standardized approach⁴.
4. Calculation of BMP standard deviation. The standard deviation associated with each mean ($n = 3$) BMP value must include variability from at least both blanks and batches (bottles) with substrate and inoculum⁵.

3.4 Validation criteria

BMP results that meet *all* the following criteria can be described as “validated”. Otherwise, results are not validated, and tests should be repeated if possible, and otherwise, the lack of validation should be made clear in any reporting of the results.

1. All required components of the BMP measurement protocol listed above (Sections 3.1 through 3.3) are met.
2. Mean cellulose BMP is between 340 and 395 $\text{NmL}_{\text{CH}_4} \text{ gVS}^{-1}$.
3. Cellulose relative standard deviation (including variability in both blanks and substrate bottles) is no more than 6%.

References

- [1] Holliger, C., Alves, M., Andrade, D., Angelidaki, I., Astals, S., Baier, U., Bougrier, C., Buffière, P., Carballa, M., de Wilde, V., Ebertseder, F., Fernández, B., Ficara, E., Fotidis, I., Frigon, J.-C., Fruteau de Lacroix, H., S. M. Ghasimi, D., Hack, G., Hartel, M., Heerenklage, J., Sarvari Horvath, I., Jenicek, P., Koch, K., Krautwald, J., Lizasoain, J., Liu, J., Mosberger, L.,

³ Detailed descriptions of calculations are available for the following measurement methods in the DBFZ BMP methods collection (<https://www.dbfz.de/en/BMP>): volumetric (document 201), manometric (document 202), gravimetric (document 203), and gas density (document 204).

⁴ Calculation of BMP is described in detail in document 200 from the DBFZ BMP methods collection (<https://www.dbfz.de/en/BMP>).

⁵ See document 200. Inclusion of variability from substrate VS determination is optional.

- Nistor, M., Oechsner, H., Oliveira, J. V., Paterson, M., Pauss, A., Pommier, S., Porqueddu, I., Raposo, F., Ribeiro, T., Rüscher, F., Strömberg, S., Torrijos, M., van Eekert, M., van Lier, J., Wedwitschka, H., Wierinck, I. 2016, *Towards a standardization of biomethane potential tests*. Water Science and Technology 74: 2515-2522
- [2] Hafner, S.D., Fruteau de Laclos, H., Koch, K., Holliger, C. In preparation, *Improving inter-laboratory reproducibility in measurement of biochemical methane potential (BMP)*. Water