A2.1.3 – Review of the requirements for laboratory and field validations of instruments

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| Task  2.1 | Activity  A2.1.3 | Reporting date  01-10-2023 |
| Title  Review of the requirements for laboratory and field validations of instruments that are used to measure key impurities in biomethane | | |
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| Abstract  The report gives an overview of the requirements for in-laboratory and in the field validation of instruments used for impurities measurements in biomethane. The difference and similarities between the two types of measurements are given. General and specific requirements are given. | | |
| Key words  Gas sampling, biomethane, dynamic methods, static methods | | |
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| Acknowledgement  The project BiometCAP 21NRM04 has received funding from the European Partnership on Metrology, co-financed from the European Union’s Horizon Europe Research and Innovation Programme and by the Participating States. | | |
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# 1 - Introduction

Standards EN16723-1 [1] and EN16723-2 [2] require analysis in a laboratory and therefore require the collection and transport of a gas sample from the sampling point. Requirements for biomethane sampling including recommendations for maintaining the integrity of the biomethane sample, material compatibility and practical recommendations for dynamic and static gas sampling have been reviewed in the report A2.1.2. Once the gas is sampled and delivered, a suitable (validated) instrument should be used for measurements of compounds in the sample.

This short report covers another aspect of the gas analysis - instrument validation in laboratory and field conditions. Various field-related aspects which differ from laboratory environments such as installation, in-field instrument calibration, dynamic gas sampling and on-site safety are considered. Recommendations on how to include these aspects in the performance assessment protocol that will be drafted in activity A2.1.4 are given.

The report includes both input from the stakeholders and biogas plant operators and experiences gained from ENG54 Biogas [4] and 16ENG05 Biomethane [5] projects.

Biomethane contains methane as a major gas component. Most instruments are designed, calibrated, and sold for air, nitrogen, or another inert gas as matrix gases. Therefore, there are two major requirements for instruments used for impurities measurements in biomethane:

* instruments for field/laboratory measurements need to be designed for gas mixtures where CH4 content is high and up to 99% +. This is particularly important if these levels of concentration can cause some interferences to the measurements (e.g. lead to spectral interferences or influence onto gas thermal conductivity).
* chemically inert, corrosive-resistant (coated) materials for gas sampling line and instrument itself should be used. The line should be kept as short as possible with optional heating above 100 oC if necessary.
* optionally, a sampling probe shall be used to obtain the best sampling results.

# 2 – Laboratory validation of instruments

Laboratory validation of instruments is relatively straightforward because most of the (accredited) laboratories have a well-established infrastructure in place.

As the instruments, especially stationary bench-top versions, are normally used with a broad range of gases, it is important that the instruments have no memory effects from the previous use. Therefore, as a minimum, instrument purge with an inert gas like He, Ar, N2 or O2 andan optional heating of instrument’s key working elements should be done before utilization. The purging time shall be determined beforehand. The base line (or zero-line) (if relevant) shall be controlled.

If the instrument requires calibration, this should preferably be done using certified standards. The calibration should be done in advance and the fit-for-purpose of the instrument shall be demonstrated by estimating its performances prior the measurements. An uncertainty budget shall be calculated.

Some impurities (e.g. NH3, HCl etc.) tend to stick (adsorbed) on the surfaces of the sampling line which connects the certified standards (A) to the instrument (B) and in the instrument itself.

Therefore, if the instrument requires calibration, the calibration shall be done in the flow mode when the gas is continuously flowing from A to B. It is recommended to monitor/record continuously the response signal of the instrument at a given gas flow rate. This will allow the user to determine an effective response time of the whole system once the reading signal is stabilized. When performing the gas analysis, it is recommended, if possible, to keep the same gas flow as the one used during the calibration and validation. Surface adsorption reduces with temperature increase; therefore, it is recommended to always keep the instrument and the sampling line in a heated mode, when possible.

Other general requirements for instrument validation in the laboratory can be summarized as follows:

* general laboratory ventilation and temperature control;
* local ventilation points (suction points above the instruments or laboratory breadboards);
* sealed system (up to few bars): from a gas cylinder to the instrument;
* shut off valves with an easy access;
* standard (left)-threaded pressure reduction valves (for flammable gases);
* small gas flow rates through the instruments;
* solid ground connection for the instruments, i.e. no floating potential;
* hand-held sensors for reactive gases are recommended to control that there is no gas leak.

Collected gas (e.g. in gas bags) for laboratory analysis should typically be limited to low volumes (typically not more than 15 liters). Once the gas has been collected it is recommended to perform analysis as soon as possible and latest two days after the collection time to avoid risk for loss of component through adsorption and therefor biased (often underestimation) measurements.

# 3 - Validation of instruments in the field

There is one major difference between validation work in the laboratory and in the field related to biogas production. Contrary to measurements in laboratory, the measurements in the field are normally performed in an outdoor environment at non-stabilized ambient conditions such as temperature and humidity. Moreover, the measurement equipment (incl. instruments) can be exposed to wind/rain or sudden and strong wind/rain gusts. Most of instruments are rated for +10 oC + 35 oC operation conditions and therefore, the best-case scenario is when the measurements are performed in late spring to early autumn time of the year. Some (especially portable) instruments classified as “rugged” can be used/designed for outdoor use and the user is advised to refer to a user guide/manual for the instrument prior its use.

IR-based and GC-based instruments require use of an in-build IR light source (IR-based) or detectors and heating parts to perform the measurements and e.g. wind gusts or direct sun light can influence their performance and potentially cause various side effects such e.g. baseline change, stability problems or long time responses.

For outdoor measurements it is recommended to:

* have a wind/rain/sun protection;
* perform measurements at temperature and humidity conditions recommended for the particular instrument;
* perform periodic base-line and reference measurements (with e.g. He, N2 and certified standards) to account for possible variations that may affect the signal (such as ambient temperature, especially during long-time field measurement campaigns). It is also recommended to use a handheld thermometer to monitor possible ambient temperature changes during the measurements.

Similarly, as for the laboratory instrument validation discussed in the section 2, possible memory and bias effects, gas flow rate, length of the sampling line and its effect on the response time need to be considered. When certified standards are used in the field, the influence of ambient temperature variations on reference gas composition needs to be considered. This can mostly be critical for dynamic gas generators used on-site for calibration/validation of the instruments.

For gas sampling from a pipe, the position of the sampling probe (if any) needs to be considered. If the probe is not in the middle of the gas flow, then the sample may not be representative of the gas it is allocated to, with regards to impurities composition. This consideration is relevant for sampling in both pressurized and non-pressurized environments.

Sampling line and the instrument shall be validated in the laboratory before the field measurements. Uncertainty budget shall be calculated for the whole system (probe + sampling line + instrument).

Other general requirements for in- field instruments validation can be summarized as follows:

* all measurements should be done under supervision;
* non-ATEX rated instruments should be placed in non-ATEX areas. ATEX-approved instrument can be used in ATEX areas after formal permission (from plant) to do the measurements. Every component installed shall be carefully checked;
* a shut off valve (before measurement system/instrument) should be agreed/installed by the production site;
* suitable reduction fittings between gas intake and the instrument should be agreed with the plant in advance (e.g. from DIN20 to ¼”, or 6 mm);
* gas-to-system and gas-to-vent flow direction, i.e. no back gas flow and no air flow towards the instrument;
* gas vent point away from any AC/DC (power) elements and building entrances;
* solid ground connection for all equipment, i.e. no floating potential;
* sealed (leak-proofed up to 8 bars, i.e. about x2 safety factor) system from gas intake to gas-to vent points;
* no electrical elements in direct contact with the gas, unless they are ATEX classified;
* no O2/air, only inert gases such as Ar, N2, CO2 etc. gases for instrument purge;
* gas sampling at low flows (maximum 1 nl/min recommended)

# 4 - Safety

There are no specific safety requirements or risk assessments to work at biogas production plants (at least based on information obtained from several biogas plants in Denmark, Sweden, Norway, and Finland).

General, common sense, safety rules are:

* visit/work at biogas production plant should be agreed in advance and the presence of personal from the plant is required;
* general protection (working safety clothes with reflective elements, shoes, protection glasses and optionally helmet);
* connections to the plant´s gas lines should be done under supervision from plant’s personal;
* no smoking, no alcohol, no drugs are allowed;
* knowledge of a common meeting point (in an emergency case).

# Conclusion

The report discusses aspects that need to be consider when installing instruments onsite: installation, onsite calibration, dynamic gas sampling and on-site safety. Some of these aspects are common for laboratory and field analyzers used for quality assessment of biomethane. The major requirements are to take all precautions to avoid leakages both in or out of the instruments, to carefully select the materials for the sampling line and the relevant parameters (such as flow rates). For instruments installed onsite, discussions with the plant are a must to select sampling points, ensure that the conditions at the sampling points are compatible with the instrument´s requirements (for example with regards to connections sizes, flow, pressure, and temperature), select dates with appropriate weather conditions (can imply backup plans). Any operation needs to be performed in the presence of personnel from the plant who have an extended knowledge of the plant including the locations of ATEX zones if needed for the installation and operations of the instruments.

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