



First experimental comparison of calorific value measurements of real biogas with reference and field calorimeters subjected to different standard methods

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ABSTRACT

This study presents the first comparison of experimental calorific value measurements of real biogas performed with three different calorimeters: one reference gas calorimeter developed at the French metrology institute (LNE) and two field calorimeters (Union Instruments CWD, 2005 CT and Cutler Hammer Calorimeter) at the German metrology institute (PTB). All measurement results obtained for calorific values agree within their measurement uncertainties. Uncertainties vary from 0.2% to 2.0% (coverage factor, $k = 2$) depending on the calorimeter and calibration procedure.

Two different standards (DIN 51899 and ISO 6143) were used to calibrate the field calorimeters and these have been compared. This comparison focuses on the calibration procedure, calibration frequency, number and composition of calibration gases and evaluation algorithm.

1. Introduction

The global concern about greenhouse gases, global warming and other environmental issues has been present since 1896 [1]. This concern is still of high relevance leading to new strategies like, most recently, the European Directive on Renewable Energies [2]. This directive aims at reducing the emission of greenhouse gases 20% (from those in 1990), using 20% of energy from renewable sources and increasing the energy efficiency in 20%. This led to an increment in use of renewable energies like solar, wind or biogas [3]. Concretely, biogas sector developed more than 6800 new biogas plants in Europe between 2010 and 2014 producing 4200 MW [4] from different sources as agricultural, forest, industrial or household feedstocks. Energy production by solar and wind technologies had a larger development than biogas production, but biogas is a direct energy that can be easily stored.

The commercial value of energy gases is given by their energy content and it is, among others, quantified by the gross calorific value. Therefore, accurate, reliable measurements of gas calorific value are of vital importance for the gas trading. This is highly challenging because the composition of biogas fluctuates significantly as shown in Table 1. Differences in concentration lead to a wide range of calorific values from 5.5 kW h m^{-3} to 8.5 kW h m^{-3} .

Indirect methods are often used to measure the calorific value for

natural gas. Composition measurements are performed usually by gas chromatography, and then the calorific value of the mixtures is inferred from the composition data and calorific value of the pure components issued from the standard ISO 6976 [6]. This standard covers only CO_2 concentrations up to 15% and other components, like water, lower than 0.0005%, therefore chromatography analysis requires some adaptation before to be suitable for biogas measurements. Furthermore, this method is prohibitively expensive for small producers. Not only the acquisition costs are high, but since it requires high quality gases for its normal use and the calibration process, the maintenance and running costs are significant as well. It also has the disadvantage that biogas might have minor impurities impossible to detect by chromatography. Other techniques, like NDIR for Non-Destructive Infra-Red analysis, are used to measure biogas and biomethane calorific value but the reliability of the measurements is weak as these techniques are not linear and mainly single-point calibrated.

Because of the measuring principle, direct methods like calorimetry display the calorific value of any mixture regardless of the composition within a reasonable working range. This measuring technique is also much simpler in terms of calibration process. Polynomial regressions are performed and for that different calibration gases with different calorific values are required.

This study continues a previous research presented by Haloua et al. [7] in a frame of an European metrology project on non-conventional

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Table 1
Average biogas composition [5].

Component	Concentration/mol·mol ⁻¹ ·10 ⁻²
CH ₄	50–75
CO ₂	25–45
H ₂ O	2–7
H ₂ S	2·10 ⁻³ - 2
N ₂	< 2
O ₂	< 2
H ₂	< 2

gases. This study takes part of a Joint Research Project named “Metrology for Biogas” partly dedicated to the characterisation of the thermophysical properties as the determination of calorific values of real biogas and biomethane samples.

This study aims to obtain reliable, reference methods to measure the calorific value of biomethane and biogas. Reference gas calorimeters can perform reliable traceable measurements of biogas and biomethane, although it requires some adaptations for renewable gases. For field calorimetry, a validated calibration process is required. In this study, two calibration standards (DIN 51899 [8] and ISO 6143 [9]) regularly used in natural gas processes have been executed and compared with a focus on the suitability for biogas, number of calibration gases, time required for the calibration process and accuracy of the measurements. Finally, calorific value measurements of a real biogas sample have been performed and compared with one reference gas calorimeter owned by the French metrology institute and two field calorimeters owned by the German metrology institute.

2. Description of the calorimeters

Three different gas calorimeters have been used for this research. One reference gas calorimeter at the French metrology institute (LNE) and two field calorimeters: Union Instruments CWD 2005 CT and Cutler-Hammer calorimeters at the German metrology institute (PTB).

2.1. The reference gas calorimeter

The reference gas calorimeter has been developed at LNE. Its development, validation, metrological qualification and use for pure compounds, synthetic and real gas mixtures have been disseminated in various publications [7,10–13].

The target objective for calorific value uncertainty for the biogas sample is 0.1%–0.4% ($k = 2$). A coverage factor $k = 2$ provides approximately 95% confidence.

Fig. 1 presents a general overview of the set-up of the calorimeter based on Rossini principle [14,15] initially operating with pure compounds.

The calorific value of a fuel gas is directly proportional to the adiabatic temperature rise of the calorimetric water bath $\Delta T_{ad,comb}$ resulting from the combustion of a defined quantity of this gas. The superior calorific value H_s is calculated by equation (1)

$$H_s = \frac{C_{cal}\Delta T_{ad,comb} + K}{m_{gas}} \quad (1)$$

where m_{gas} is the mass of the gas to be burned (kg) and K (kJ) represents the sum of energetic corrections inherent in a combustion experiment as the electrical energy released for ignition, the energy loss due to water vapour leaving the calorimeter during the combustion and the energy loss due to the difference of the gas temperatures at the inlet and outlet of the burner. The heat capacity of the calorimeter C_{cal} (kJ·K⁻¹) is calculated from an electrical calibration step in which electrical energy dissipation by Joule effect is performed at the nearest of the combustion site. The determination of heat capacity obtained by electrical dissipation in the calorimeter through $C_{cal} = E_{cal}/\Delta T_{ad,cal}$

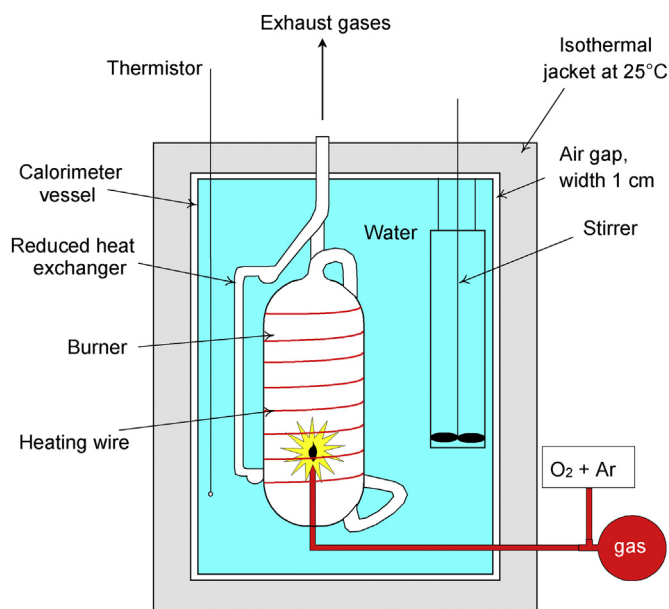


Fig. 1. Schematic drawing of the reference gas calorimeter.

guarantees traceability of each determination of the calorific value to the SI units. Electrical energy E_{cal} is assessed to be equivalent to the combustion and accurately measured.

Taking into account the method for heat capacity determination, equation (1) becomes:

$$H_s = \frac{E_{cal}}{m_{gas}} \frac{\Delta T_{ad,comb}}{\Delta T_{ad,cal}} + \frac{K}{m_{gas}} \quad (2)$$

Uncertainty budgets are calculated for each term of equation (2) using it as the mathematical model. The uncertainty propagation law according to the “Guide to the Expression of Uncertainty in Measurement” (GUM) [16] was applied to it. Equation (3) enables a simplification for the uncertainty calculations:

$$H_s = \frac{E_{cal}}{m_{gas}} Z + K \quad (3)$$

Measurement process of the reference gas calorimeter is highly detailed in Refs. [7,10–13]. However, the reference calorimeter requires some modifications and improvements to perform reliable measurements of real biogas.

The reference gas calorimeter at LNE has been validated and used for synthetic gas mixtures and pure compounds. Measuring the thermophysical property as the gross calorific value of raw biogas was highly challenging for the whole instrument's safety: deposit of soot particles or embedding of hard-to-remove materials can occur at the walls of the burner made of glass during the combustion and the burned gas analysing system (Flame Ionisation Detector analyser and IR/Chemiluminescence analysers) could be damaged by the nature of the burned exhaust gases. Choice has been made to keep the burner as it is and to sample the exhaust gases in a Tedlar bag (40 L) in order to analyse them with a Fourier-Transform Infrared Spectroscopy (FTIR) gas analyser calibrated with a synthetic reference mixture CO, CO₂, CH₄ and C₂H₄.

Because of the sampling conditions at the raw biogas production site in Finland, the sampling pressure was limited at the outlet of the biogas plant. Three 50 L bottles have been filled for calorific value measurements at PTB and LNE. LNE received one bottle which, after verification, has an absolute pressure of $p_{abs} = 3$ bar. Several major difficulties arose for the experiments with the reference calorimeter with this pressure:

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