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A modified sulphur hexafluoride tracer technique enables accurate determination of enteric methane emissions from ruminants

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ABSTRACT

The sulphur hexafluoride (SF_6) tracer technique enables determination of enteric methane emissions from large numbers of individual ruminant animals. The objective of this research was to identify and correct substantial errors within the SF_6 technique. Six experiments were undertaken using respiration chamber, laboratory or SF_6 techniques. Experiment 1 used respiration chambers to demonstrate that the daily pattern of methane emissions from dairy cows was related to their pattern of feed intake. In contrast, the daily emission of SF_6 from these cows was constant and independent of the pattern of methane emission. This finding supports the contention that in order to accurately determine daily methane emissions using the SF_6 technique, it is necessary that gases are collected continuously at a constant rate for 24 h. Since development of the SF_6 technique in 1993, it has been propounded that capillary-tube flow restrictors achieved a constant rate of sample collection into evacuated gas collection canisters. Laboratory experiments 2, 3, 4 and 5 demonstrated that, when capillary-tube flow restrictors are used, the rate of sample collection declined and caused a bias of up to 15.6% in calculated methane emissions. This error was caused by an interaction between the declining sample collection rate and the pattern of an animal's methane emission over 24 h. In contrast, orifice plate flow restrictors maintained a constant sample collection rate at canister pressures <0.31 atm and thereby minimised the decline in sample collection rate. Experiment 5 also demonstrated that sample collection using orifice plate flow restrictors, combined with initial (<0.03 atm) and final (<0.49 atm) canister pressures, substantially reduced measurement error. Accuracy of the modified SF_6 technique, incorporating orifice plate flow restrictors for 24 h sample collection, was validated in Experiment 6. The mean (S.D.) methane yield (g CH_4/kg DMI) of eight cows did not differ ($P = 0.135$) when determined using the modified SF_6 technique 22.3 (1.44) or chambers 21.9 (1.65). In addition, the between-animal coefficient of variation for methane yield determined using the modified SF_6 technique (6.5%) was similar to that determined using

Abbreviations: atm, atmospheres; cc, cubic centimetre; DM, dry matter; DMI, dry matter intake; ID, internal diameter; OD, outer diameter; sccm, standard cubic centimetres per minute; SF_6, sulphur hexafluoride.

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chambers (7.5%). Consequently the modified SF_6 technique enables the statistical power of experiments to be increased or their size decreased. We conclude that the modified SF_6 technique reduced error associated with SF_6 release, sample collection and analysis. It is recommended that the modified SF_6 technique should be used in preference to the original SF_6 technique for determination of enteric methane emissions from ruminants.

1. Introduction

The emission of enteric methane from ruminants can be estimated using a calibrated tracer technique developed by Zimmerman (1993) and first deployed by Johnson et al. (1994). The tracer technique enables estimation of methane emissions from large numbers of individual ruminant and pseudo-ruminant animals without the need for their confinement.

A requirement of the technique is the release of a tracer gas (sulphur hexafluoride, SF_6) at a known constant rate into the reticulo-rumen of each animal. To achieve this, a slow-release device, known as a permeation tube, is placed in the reticulo-rumen (Zimmerman, 1993). The subsequent pattern of SF_6 emission from a ruminant animal has not been reported. However, it is well known that the rate of methane emission from ruminants can vary considerably throughout the day in relation to feeding (Aguerre et al., 2011; Grainger et al., 2007). Thus, the SF_6 technique requires a representative sample of emitted gases to be collected at a constant rate to ensure that an equal proportion of the sample is collected throughout the typically 24 h sampling period.

An animal's daily enteric methane emission is calculated from the measured gas mixing-ratio (concentration) of eructated and expired methane and SF_6 in a gas sample collected from a point near the mouth and nostrils.

Since the first reported application of the SF_6 technique by Johnson et al. (1994) the collection of gases from the ruminant animal has been achieved using evacuated sample collection canisters. This sampling method is reliant on simple components suited to deployment on livestock halters and harnesses. Gases are drawn into evacuated collection canisters without the use of electronic flow controllers or pumping systems. Instead, gases are typically drawn in by a partial vacuum *i.e.*, a low gas pressure within the sample collection canister (hereafter referred to as canister pressure) of <0.03–0.5 atmospheres (atm) (*e.g.*, Johnson et al., 1994).

The original gas sampling apparatus used for the SF_6 technique restricted the rate of gas inflow into an evacuated canister by placing a length of capillary tubing, with an internal diameter (ID) of 127 μm, between the sample collection point and the canister (Johnson et al., 1994). This capillary-tube created a leak into the canister enabling the canister to slowly fill with gases during a sample collection period of 2–6 h. Since 1994 the capillary-tube has become the standard method of flow restriction used within the SF_6 technique. All of the commonly cited publications describing the SF_6 technique have used capillary-tubes as flow restrictors and have stated that the resulting rate of air flow into evacuated canisters was constant throughout the sample collection period (*e.g.*, Johnson et al., 1994; Johnson and Johnson, 1995; Johnson et al., 2007). However, experimental data supporting this assertion have not been reported in any of the papers dealing with the SF_6 technique. Furthermore, the Hagen–Poiseuille Law (Pfitzner, 1976) states that gas flow through a capillary-tube is related to the driving pressure as described by Eq. (1);

$$Q = \frac{\pi \times \Delta P \times r^4}{8 \times \eta \times l} \quad (1)$$

where Q is gas flow rate through the capillary (m^3/s), ΔP is the pressure gradient across the capillary (Pascal), r is the inner radius of the capillary (m), η is the viscosity of the gas (Pascal-second), l is the length of the tube (m), and π and 8 are constants.

It follows from Eq. (1) that as an evacuated canister fills with collected air, the absolute pressure within the canister will increase and the rate of sample collection will decline. The Hagen–Poiseuille law contradicts the assertion in the context of the SF_6 technique, that the air sampling rate achieved using capillary-tubes is constant. Thus, it would seem that all of the methane measurement experiments conducted since 1994 that have employed a SF_6 technique, have failed to achieve a constant rate of sample collection. The impact this non-constant rate of sample collection has had on the accuracy of the SF_6 technique is difficult to assess. This is because the magnitude of the decline in sampling rate during a typical 24 h sampling period has not been documented. Uncertainty surrounding the magnitude of the potential inaccuracy is further compounded by the fact that the error induced by this phenomenon is almost certainly dependent on the diurnal pattern of methane emission of individual animals.

Since the initial use of the SF_6 technique, several research groups have made modifications to the gas sampling apparatus to improve its robustness. One widespread modification has been to use a short length of crimped capillary-tube as the flow restrictor (*e.g.*, Hegarty et al., 2007; Wims et al., 2010; Williams et al., 2011). Typically a short section of 127 μm ID capillary-tube, between 10 and 20 mm in length, is crimped to reduce its air conductance to the desired rate and it is positioned within a gas tight Swagelok tube fitting. The effect that crimping of capillary-tubes has on the rate of sample collection at different canister pressures has not been reported.