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VIEWPOINT

Invited Contribution for the 2022 Anniversary Edition

Introduction of a guideline for measurements of greenhouse gas fluxes from soils using non-steady-state chambers

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Method: Soils represent a major global source and sink of greenhouse gases (GHGs). Many studies of GHG fluxes between soil, plant and atmosphere rely on chamber measurements. Different chamber techniques have been developed over the last decades, each characterised by different requirements and limitations. In this manuscript, we focus on the non-steady-state technique which is widely used for manual measurements but also in automatic systems. Although the measurement method appears very simple, experience gained over the years shows that there are many details which have to be taken into account to obtain reliable measurement results.

Aim: This manuscript aims to share lessons learnt and pass on experiences in order to assist the reader with possible questions or unexpected challenges, ranging from the planning of the design of studies and chambers to the practical handling of the chambers and the quality assurance of the gas and data analysis. This concise introduction refers to a more extensive *Best Practice Guideline* initiated by the *Working Group Soil Gases* (*AG Bodengase*) of the *German Soil Science Society* (*Deutsche Bodenkundliche Gesellschaft*). The intention was to collect and aggregate the expertise of different working groups in the research field. As a compendium, this *Best Practice Guideline* is intended to help both beginners

and experts to meet the practical and theoretical challenges of measuring soil gas fluxes with non-steady-state chamber systems and to improve the quality of the individual flux measurements and thus entire GHG studies by reducing sources of uncertainty and error.

KEYWORDS
carbon dioxide, chamber measurements, greenhouse gases, methane, nitrous oxide, soil gas flux

1 | INTRODUCTION

As a result of high versatility, seemingly easy application and their relative inexpensiveness, manual closed chamber measurements are a widely used and accepted technique for estimating the exchange of carbon dioxide (CO_2), methane (CH_4) and nitrous oxide (N_2O) between soils, vegetation and the atmosphere. Different approaches and chamber designs have been developed and were applied in studies with a wide range of research objectives. In this manuscript, we focus on the most widely used approach, the non-steady-state technique (Livingston & Hutchinson, 1995), where a (soil) surface is temporarily covered with a chamber, and the gas flux across the surface is calculated from the chamber headspace concentration change over time. Flux measurements with chambers require many decisions, each affecting the quality of the final flux data, which hence need to be considered carefully during planning.

The choice of temporal measurement resolution, the spatial layout and number of measurement replicates, the recorded ancillary data, as well as chamber design and handling can substantially affect the quality of the resulting flux data and their suitability for subsequent meta-analyses or modelling. Chamber designs are highly diverse and another major source of uncertainty regarding the absolute values (Pihlatie et al., 2013; Pumpanen et al., 2004). This has substantial consequences for upscaled flux estimates, derived emission factors and the inter-comparability of published flux data. Chamber designs are associated with specific technical limitations and should be adapted to the studies' objectives. Flow-through, non-steady-state chamber measurements require online gas analysers with a higher measurement frequency. This approach has been used for CO_2 for many years, and more recently also for CH_4 and N_2O. However, manual sampling of discrete gas samples taken at intervals from closed non-flow-through and non-steady-state chambers with subsequent gas analysis in the laboratory (e.g. by gas chromatography [GC]) is still a common way to determine CH_4 and N_2O fluxes. For this approach, the potential errors due to sampling and gas sample storage have to be considered, and those due to the later laboratory analyses. Practical experience and good quality assurance/quality control (QA/QC) routines are important to minimise the errors. Although the increasingly used 'fast analysers' offer many advantages, they are also associated with specific challenges. While flux estimation methods have been widely discussed with respect to suitability (Livingston et al., 2006; Parkin et al., 2012; Pedersen et al., 2010), little attention is often paid to the storage, screening and handling of flux data and ancillary measurements as well as to practices of flux and error estimation. The benefit of published high-quality flux data to the scientific community could be greatly enhanced by thorough and consistent reporting and description of the data (Rochette & Eriksen-Hamel, 2008).

For each of the above-mentioned steps, this document provides recommendations on how sources of error and uncertainty can be minimised and how the quality and reporting of CO_2, CH_4 and N_2O flux data may be improved. We provide standardised recommendations for manual and automated non-steady-state (i.e. closed) chamber measurements of CO_2, CH_4 and N_2O fluxes from soils in (1) terrestrial and (2) semi-terrestrial ecosystems including wetlands and nearshore environments. These include measurements and studies in different land uses such as agricultural fields, grasslands, natural, managed and restored peatlands and forests. Gas flux measurements at the surface of flowing water, lakes and oceans are not explicitly considered. Flux measurements of reactive gases (e.g. ammonia) require additional considerations, as do the applications using the isotopic signature of soil gases (Zaman et al., 2021), and therefore they are not explicitly discussed here.

With this guideline, we contribute towards a standardised procedure for greenhouse gas (GHG) flux measurements in order to improve the validity and comparability of GHG studies and to make users aware of potential error sources of GHG flux measurements from planning to data reporting and publishing. The recommendations given here are not meant to stand alone but build on general introductions to soils, soil gases and chamber measurements (Livingston & Hutchinson, 1995; Rochette & Hutchinson, 2015) and contribute to the existing QA/QC and standardisation initiatives for GHG measurements in general (ISO, 2019; Parkin & Venterea, 2010; Pavelka et al., 2018; USDA & NRCS, 2006), flux measurements of N_2O (de Klein & Harvey, 2012; de Klein et al., 2020; Rochette, 2011) and within the framework of GHG monitoring networks (Pavelka et al., 2018) or with special focus on automated measuring approaches (Subke et al., 2021) and isotopic approaches (Zaman et al., 2021). In addition to the existing standardisation efforts, we try to fill prominent gaps including practical recommendations and details regarding (1) the necessary considerations before starting the measurements (study and chamber design, planning), (2) the chamber measurement itself such as the handling of the chamber and gas analysis including tips for QA/QC for GC and (3) the workflow after the chamber measurement including available packages for flux estimation, limitations, artefacts and uncertainties, and will close with a short view on future developments and perspectives. This paper refers to a far more extensive Guideline (Fiedler et al., 2022) which was written by the authors after intensive discussions within the Working Group '*Soil Gases*' of the German Soil Science Society (*Deutsche Bodenkundliche Gesellschaft*; DBG) and colleagues abroad.

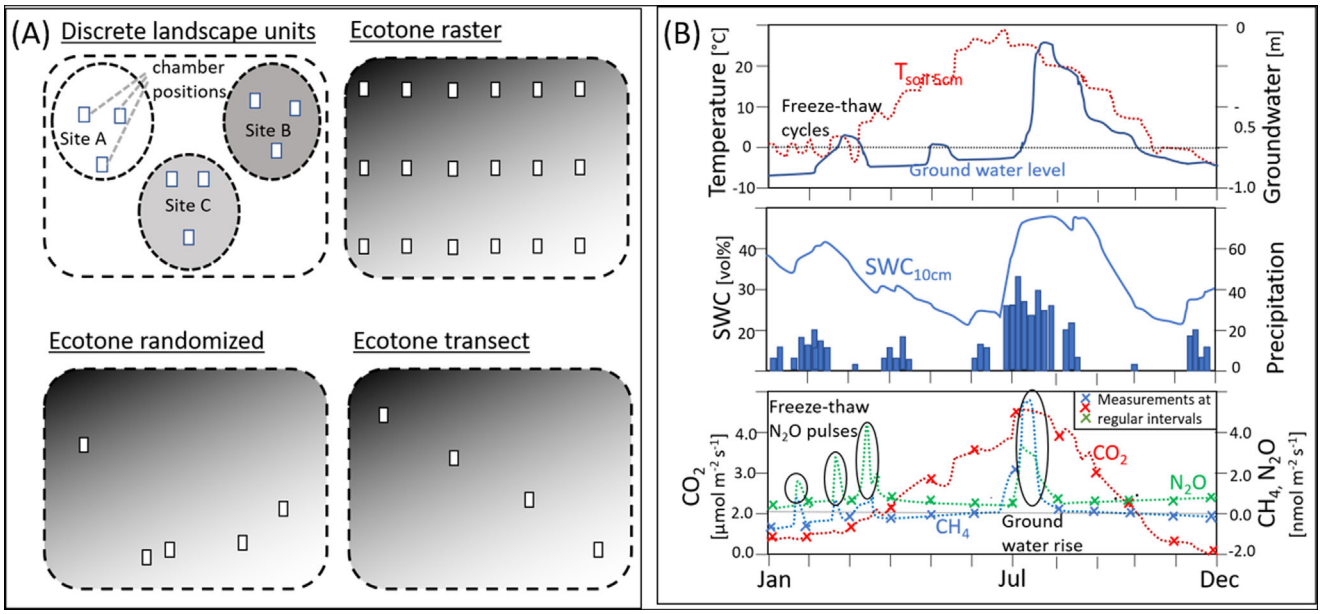

FIGURE 1 (A) Overview of different spatial layout types of chamber positions within different spatial structures (adapted from figure 1 of the full Guideline, Fiedler et al., 2022). (B) Exemplary (fictional) temporal evolution of (top) soil temperature and ground water, (middle) precipitation and soil water content (SWC) and (bottom) fluxes of CO_2, N_2O and CH_4.

2 | CONSIDERATIONS BEFORE STARTING THE MEASUREMENTS

2.1 | Study design

Studies involving field measurements of GHG fluxes require resources like time, investment and skilled personal. A well-planned study design is essential to achieve the desired research objectives. Chamber-based GHG measurements are often used to study GHG fluxes from different land uses or treatments, spatial patterns or individual objects (e.g., agricultural fields, plants, termite mounds, tree stems etc.) or for monitoring purposes. The study objective is not always the accurate quantification of an absolute GHG balance over time, but can also be the identification of differences between treatments or the drivers behind patterns. Yet, accuracy should always be an important objective.

2.1.1 | Addressing spatial variability

GHG fluxes vary spatially and temporally on the plot scale and are therefore different between ecosystems, landscapes and sites. Spatial and temporal variability can be very different for CO_2, CH_4 and N_2O and depend on the respective temporal and spatial scale. Landscapes and parts thereof are mosaics of discrete, more or less homogeneous units, which can be assessed by replicate chamber measurements (Figure 1A). The number of replicates per site or plot should be determined by the sample size required to identify statistically significant differences between these units, mainly depending on the (often unknown) spatial variability within the respective unit. While the relatively low spatial variability of CO_2 fluxes allows to work with less spatial replicates, this is more challenging for emissions of CH_4, and especially N_2O, where the potentially large variability (Folorunso & Rolston, 1984; Koch et al., 2014; Maier et al., 2017) would result in much higher replicate numbers. Even if such replicate numbers cannot be realised in long-term field studies, a pilot study assessing the spatial variability helps to plan a suitable design. The locations for pilot study measurements need to be selected using a predetermined grid, transects or by means of randomised locations (Figure 1A), depending on prior knowledge, proxy data and study objectives.

Depending on the result of a pilot study, spatial patterns or discrete units of GHG fluxes can be identified (Figure 1A), which allows finding proper measurement placements for the study, for example by addressing typical discrete units or laying a transect across a gradient. Topographical position, soil texture and vegetation units may be relevant proxies (Acosta et al., 2013; Maier et al., 2017). At least three spatially independent replicates need to be realised to derive simple statistical measures such as arithmetic mean and median. However, more independent replicates are preferable (Chadwick et al., 2014). Pooling of gas samples (Arias-Navarro et al., 2013) would allow to work with more spatial replicates with the same workload for the gas analysis, which is often a limiting factor. Yet, similar to working with larger chambers covering a larger surface area, a minimum of three independent measurements are still needed (Prosser, 2010).

2.1.2 | Addressing temporal variability

Chamber measurements give GHG flux estimates for the interval of the measurement. Automatic chamber systems (Subke et al., 2021)

allow for high-frequency measurements that cover possible temporal variability (Lammirato et al., 2018, 2021), for example with hourly measurement (at minimum at periods >10× closing time of the chamber). Manual measurements nevertheless play an important role since automatic systems require more infrastructure and resources. The sampling schedule for manual measurements should be flexible and account for hot moments such as land management measures (e.g. tillage, fertilisation, harvest), meteorological events (drought, strong frost and heavy precipitation, freeze–thaw N_2O pulses, CH_4 pulses due to ground water rises in Figure 1B) and plant phenology. Regular measurements every 1–2 weeks will cover background flux periods, which might be very relevant for the calculation of GHG budgets. Measurements should ideally not be interrupted for more than 1–2 weeks, because the uncertainty of GHG budgets is linked to measurement gaps (Lucas-Moffat et al., 2018). It is important to anticipate patterns and magnitudes of GHG fluxes to optimise the measurement schedule accordingly. For this, a preconception of factors controlling the processes behind the GHG fluxes is needed. The environmental controls have been reviewed for soil respiration (Vargas et al., 2011), gross primary production (GPP) (Anav et al., 2015), N_2O emissions (Butterbach-Bahl et al., 2013), N_2O uptake (Chapuis-Lardy et al., 2007) and CH_4 emissions from wetlands (Bridgham et al., 2013) and in forests (Feng et al., 2020).

In general, GHG studies should account for the following factors:

- Air and soil **temperatures** and **soil moisture** are usually in temperate climates the most important controls of biological processes, especially soil respiration (Figure 1B).
- **Photosynthesis** potentially affects all GHG fluxes via root exudation which accelerates microbial activity. This can be especially relevant if gas-transport mechanisms within plants are driven by the rate of photosynthesis (Günther et al., 2014; van den Berg et al., 2020). Photosynthesis reduces the effective foliar respiration ('Kok effect') which needs to be taken into account when opaque chambers are used (Atkin et al., 1998; Wehr et al., 2016).
- Freeze–thaw cycles, drought–rewetting cycles, a changing (high) ground water table and situations when aerated soils become (nearly) water saturated are often drivers for **hot moments** for CH_4 and N_2O fluxes (Figure 1B) (Kuzyakov & Blagodatskaya, 2015; Luo et al., 2012).
- **Management factors and events**, like fertilisation, pesticide application, tillage, harvest, crop choice as well as grazing, can be a major control of GHG fluxes. Changes in water table (e.g. CH_4 pulse in Figure 1B) and soil moisture must also be considered. Measurements should be conducted directly before and repeatedly immediately after any such events.

2.1.3 | Ancillary data and measurements

It is important to measure and document all parameters needed to understand and plan the study design and schedule, especially those that are needed for the chamber measurement itself (GHG concentrations, air temperature and pressure, relative humidity) but also photosynthetically active radiation (PAR) when transparent chambers are used. Additional general climate data (precipitation, wind speed, temperature) and soil type, pH and texture and variables such as soil temperature, soil water content and water table depth are usually also needed. Depending on the study, nutrient concentrations (e.g. nitrate) or dissolved organic carbon in the soil pore water can be of great value when modelling is intended or as explanatory variables. Information about vegetation (growth, species composition, leaf area, rooting depth) is needed several times a year for crops. Photos of the chamber bases at each measurement date can be helpful for documentation and quality assurance.

2.2 | Chamber design

2.2.1 | Fundamentals of non-steady-state chambers

Non-steady-state chamber measurements rely on chamber systems that temporarily cover the surface of the soil. The gas flux between the soil surface or the soil–vegetation system and the atmosphere, $\Phi_{gas,s\text{-}v\text{-}a}(t)$, is determined by measuring the change in gas concentrations over time inside the chamber (headspace), which is assumed to ideally correspond to the net molar gas flux $\Phi_{gas,net}(t)$, given that gas fluxes between inside and outside of the chamber $\Phi_{gas,leak}$ are negligible (Figure 2A).

The gas flux $\Phi_{gas,s\text{-}v\text{-}a}(t)$ can be approximated by

$$\Phi_{gas,s-v-a}(t) \cong \frac{dn_{gas}(t)}{dt} \times A^{-1}, \quad (1)$$

where A is base area of the chamber, and $n_{gas}(t)$ amount of gas molecules in the chamber at time t. Based on the chamber volume V_{ch}, air pressure $p_{air}(t)$, air temperature $T_{air}(t)$ and the concentration of the gas of interest $x_{gas}(t)$, the amount of gas molecules $n_{gas}(t)$ can be calculated using the ideal gas law, where R is the ideal molar gas constant:

$$n_{gas}(t) = \frac{p_{air}(t)V_{ch}}{RT_{air}(t)} x_{gas}(t). \quad (2)$$

The concentration of the gas of interest is measured either by taking discrete samples that are transferred in vials to the laboratory for analysis using, for example, GC, or by circulating air in a closed loop from the chamber to the gas analyser and back. Increasing water vapour concentration due to evapotranspiration leads to dilution of the gas of interest (Pérez-Priego et al., 2015), which can be corrected if water vapour concentration $x_{H_2O}(t)$ is also measured:

$$x_{gas,corr}(t) = x_{gas,uncorr}(t)\frac{1 - x_{H_2O}(t_0 = 0)}{1 - x_{H_2O}(t)}. \quad (3)$$

Since the gas concentration measurements must be representative for the entire headspace volume, it is necessary that the chamber headspace is well mixed, for example by a small fan (Christiansen et al., 2011; Rochette & Hutchinson, 2015).

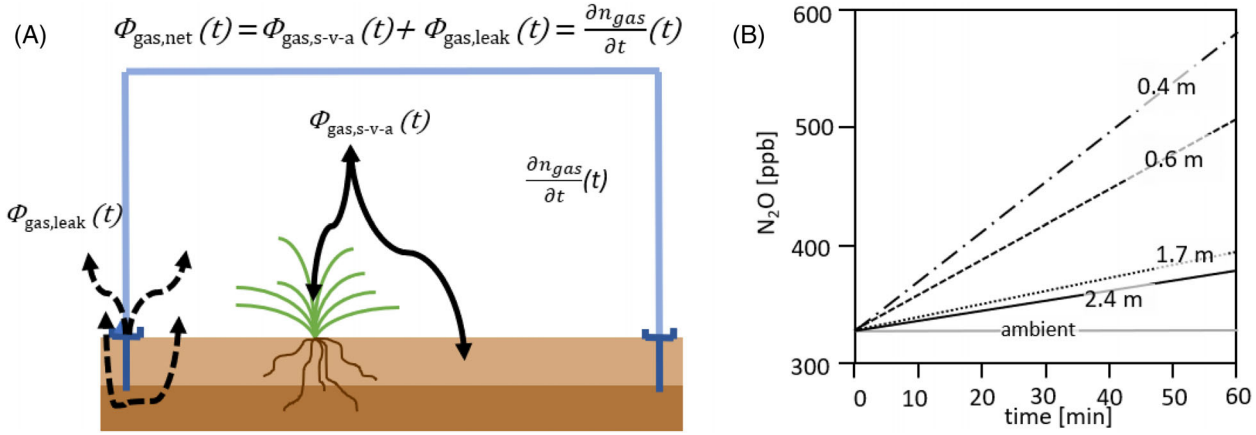

FIGURE 2 (A) Schematic of the general principle of a closed chamber. The net gas flux can be expressed as the sum of the gas fluxes between the soil–vegetation system and the atmosphere $\Phi_{gas,s\text{-}v\text{-}a}(t)$ and the gas flux between inside and outside of the chamber $\Phi_{gas,leak}(t)$. (B) N_2O concentration change during typical chamber-closure times at a fixed efflux of 200 μg m^{-2} h^{-1} for different chamber heights (Huth, 2016)

2.2.2 | Chamber construction and geometry

Typically, chambers are composed of two components: a base, which is permanently inserted in the soil in a mechanically stable way, and a chamber top, which is put on the base only during the flux measurement. Chambers can have different geometries and sizes depending on the research question, study design and the structure of the ecosystem under investigation. Chamber systems should be composed of gas-tight and nonreactive materials, and be constructed so that the headspace volume is not significantly altered during chamber closure. An airtight seal between the base and top of the chamber is required and should be checked regularly (Hoffmann et al., 2018). The construction should minimise condensation of water inside the chamber, and for automated systems also within the flow through tubes going to the gas analyser (e.g. by heating cables). The base area of the chamber should be as large as possible considering the practical handling and the desired spatial resolution of the study (Kaiser et al., 1996). The smaller the base area, the larger the relative disturbances along the edges of the base. A base area–perimeter ratio of ≥10 cm is considered as very good, whereas a ratio of ≤2.5 cm is considered as very poor (Healy et al., 1996). The higher the air-filled porosity of the soil and the longer the chamber deployment period, the deeper the chamber base has to be inserted (Rochette & Eriksen-Hamel, 2008). A ratio of insertion depth to chamber deployment period of ≥12 cm h^{-1} is considered as very good, whereas a ratio of ≤5 cm h^{-1} is considered as very poor.

The chamber must be high enough for the plants so that they are neither folded nor clipped. Disturbance of the plants may have effects on photosynthesis and respiration as well as on the transport of other trace gases, for example on gas transport through aerenchyma of wetland plants (van den Berg et al., 2020). On the other hand, the chamber height also directly affects the time required for a given flux to reach a certain concentration. With a given precision of the gas analysis, higher chambers may result in longer closure times when flux rates are low (Figure 2B). As several gases with different flux rates and different concentration measurement precision are often simultaneously analysed, a compromise must be found. The effective chamber volume has to be accurately determined especially considering the microrelief of the soil surface.

2.2.3 | Avoiding artefacts

Although it should be avoided, air temperature inside the closed chamber as well as barometric pressure can change during the time of chamber closure. A perfectly airtight chamber would generate artefacts. For example, the volume expansion of air induced by a temperature increase might lead to a (very slow) mass flow of air in the direction to the soil pore space, which would interfere with the diffusional fluxes. Thus, it always should be considered to use a chamber vent (Hutchinson & Mosier, 1981). To avoid systematic flux artefacts caused by wind-induced pressure gradients (Venturi effect) (Maier et al., 2019), different vent designs and solutions have been proposed (Xu et al., 2006). During the placement of the chamber, short-time pressurisation of the chamber headspace is likely to occur, which can lead to irregularities (Christiansen et al., 2011; Davidson et al., 2002) which cannot be avoided by regular permanent vents. We recommend using an additional closable larger installation vent, which can be opened during chamber placement and closed immediately after placement. Nevertheless, leakage must be avoided and can be tested using smoke cartridges inside the closed chamber (Hoffmann et al., 2018).

Chamber measurements require proper mixing of air inside the chamber which is usually achieved by a small fan or circulating air in flow-through chambers. Several studies have shown that the strength of turbulences inside the chamber headspace influences the flux estimate (Lai et al., 2012; Reicosky et al., 2008). Ideally, chamber measurements would not affect the mixing of air in the atmosphere and the gas exchange between soil and atmosphere. A chamber system with a too strong fan used in calm weather might 'flush' more gas from the soil by mixing the previous gradient of gas concentration next to the soil surface. A fan with weak circulation speed will not be able to mix the chamber air properly. No standard method is so far available to correct for these effects of changes in turbulence.

As a presumption, the flux is not significantly affected due to the chamber measurement process. This calls for system designs and routines, which minimally perturb both the gas fluxes during individual measurements as well as due to repeated measurements. This is the case if the chamber system is designed so that it minimises all possible side impacts on the radiation balance, air humidity and temperature in the chamber and the temperature of the soil. Mechanical disturbances of vegetation and soil, particularly at the edges of the chamber, should also be avoided. Closing the chamber should be avoided during significant precipitation events (Luo et al., 2012; Maier et al., 2019) to ensure that the soil water status inside and outside of the chamber remains similar in the long term.

2.3 | Summary of tips and considerations before the measurement

- **Study design:** A sufficient number of **spatial replicates** of measurements is essential for an effective study. This number depends on the ecosystem studied and its inherent spatial variability and patterns. At least three spatially independent replicates need to be realised, but more replicates are preferable.
- **Study design:** The **temporal resolution** of measurements must account for the anticipated temporal variability of fluxes, which are often very different between the respective GHG. Processes producing or consuming CO_2, CH_4 and N_2O respond differently to environmental drivers like soil moisture, precipitation, air-filled pore space, freeze–thaw cycles and water table depths. Including such parameters in the monitoring and planning scheme allows monitoring low background fluxes as well as high fluxes during critical events (hot moments). These parameters can also be used as explanatory variables and help filling gaps.
- **Chamber design:** Chamber systems should consist of a permanently installed base, a chamber with permanent vent, a closable larger installation vent and a fan (or other equivalent). The base area should be as large as possible and as small as necessary to allow for easy handling and the desired spatial resolution. The chamber height should be large enough for the plants in the chambers and small enough to allow for short chamber closure, taking into account the given precision of the gas concentration analysis. Chamber systems should be carefully and systematically checked prior to field campaigns (i.e., sealing, headspace mixing, optical transmissivities etc., using an extensive check list)
- **Additional sampling of (background) data:** Relevant ancillary data must be collected along with the chamber measurements.

3 | MEASURING SOIL–ATMOSPHERE GAS FLUXES USING NON-STEADY-STATE CHAMBES

3.1 | Chamber handling and gas sampling

3.1.1 | Short versus long chamber closure periods

Short chamber closure periods minimise the disturbance of the studied ecosystem (de Klein & Harvey, 2012; Rochette & Hutchinson, 2015). Longer closure periods result in a lower detection limit for gas fluxes, which is of particular importance for low fluxes of N_2O (Lammirato et al., 2021). The duration of closure periods should be based on the expected flux magnitude and the precision of the gas analysis. Periods of 20–60 min for GC-based measurements and 3–5 min for online analysers are common.

Soil–atmosphere gas fluxes are assumed to be mainly driven by diffusion in the soil, although non-diffusive fluxes can be important under special circumstances (e.g. wind-induced pressure-pumping, ebullition). Closed chamber methods rely on measuring the gas flux into the chamber based on the change in gas concentration while the chamber is closed. Since this diffusive gas flux is driven by the concentration gradient between soil and soil surface, the continuous increase in gas concentration in the chamber reduces the flux (Figure 3). To account for this artefact, flux estimations can be (1) restricted to a period at the beginning of the closure for which the concentration change can be assumed approximately linear (Figure 3). Another option (2) is to account for nonlinear gas concentration change by fitting a nonlinear function to the data to estimate the 'undisturbed' slope at the moment of chamber placement (Livingston et al., 2006; Pérez-Priego et al., 2015). The time until quasilinear concentration changes become nonlinear depends on many factors such as flux rates, effective chamber height, air-filled porosity and gas concentration profiles in the soil. When fluxes of different gas species are studied, the most suitable option (1 or 2) for the respective gas species should be selected.

3.1.2 | Chamber handling

Chamber bases should be installed so that their upper edge is close to the soil surface. The installation of chamber bases disturbs the soil structure and may cut roots, thus biasing the natural gas flux (Heinemeyer et al., 2012). In agricultural settings, bases should be installed into bare soil, for example shortly after seeding (de Klein & Harvey, 2012). Installation in permanent grassland damages roots that need to regrow over weeks to months which must be considered if measurements start right after installation. The base needs to be installed at least several hours before the first measurement.

Walking next to a chamber during measurement must be avoided as it may cause a pumping effect, damages surrounding vegetation and compacts the soil. In wetlands and peatlands, boardwalks need to be installed to facilitate the measurements and to avoid ebullition. When closing the chamber, it is important to carefully lower the chamber top

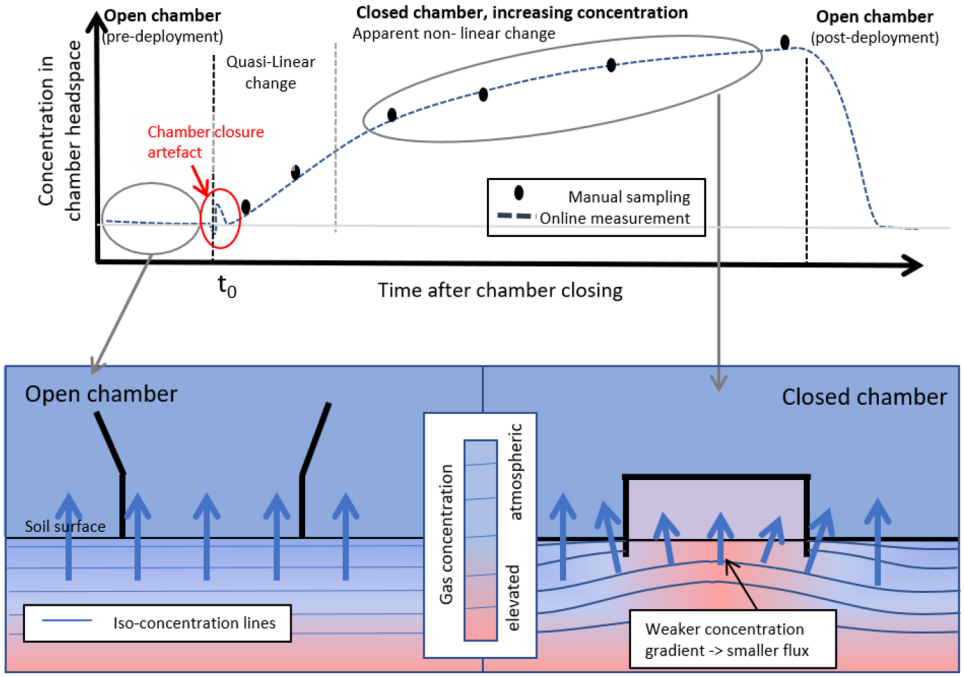

FIGURE 3 (Top) Schematic evolution of the gas concentration, for example CO_2 in a non-steady-state chamber, displayed as online measurement (dashed line) and manual samples measurements (points). (Bottom) Cross section of a chamber measurement. As soon as the chamber closes (t_0), the gas concentration in the closed chamber increases approximately linearly. After the concentration gradient at the soil–atmosphere interface decreases, the flux into the chamber decreases.

onto the base to avoid excessive pressure shocks, which can be avoided by using an additional closable larger installation vent. Between measurements, the chamber must be completely flushed with ambient air. Especially transparent chambers need to be cleaned regularly.

3.1.3 | Manual gas sampling for lab analysis

Manual gas sampling can be done with syringes or evacuated or non-evacuated vials. Pumping with syringes prior to sampling is not recommended. It is advantageous to use a sampling method that allows creating over-pressure in the sample containers, for instance, by sampling with syringe and then pushing the sample into an evacuated vial with a smaller volume than the syringe. Manual sampling from the chamber headspace is usually carried out with low frequency, i.e. few gas samples (four to six) are taken from the closed headspace after defined intervals of, for example, 0, 5, 10, 20 and 30 min. Different types of vials are available from different manufacturers, which always should be tested for airtightness. Practicability is an important reason why many researchers tend to use smaller vials that can easily be set onto autosampler systems for the GC.

More gas samples taken during one chamber measurement allow for a better flux estimate. Yet, the trade-off between sampling and analysis cost, statistical accuracy and study design has to be considered. The quality of the concentration measurements has important implications for the reliability of the flux estimation. Generally, at least four samples are recommended to ensure reliable flux estimation. The first gas sample from the chamber headspace should be taken as soon as possible after chamber placement because it cannot be assumed that the headspace air composition equals that of ambient air (Rochette, 2011). In special cases, it can be effective to use two-point flux estimates if—in turn—the total number of conducted flux measurements can thus be increased (Chadwick et al., 2014). This procedure allows for reducing the overall uncertainty due to spatial variability while allowing for higher uncertainty of individual flux estimates (Jungkunst et al., 2018).

3.1.4 | Gas sampling with online gas analysers

Online gas analysers allow for continuous gas measurements in the field. Besides their multiple advantages, there are also challenges to consider. Gas is sampled via tubes from the chamber headspace, analysed with a frequency of ≈0.05–1 Hz and pumped back to the chamber in a closed loop. A time delay between the concentration change in the chamber and the analyser has to be accounted for. An additional fan is recommended for larger chambers to ensure proper mixing of the chamber headspace. The length of the connecting tubes should be similar at the inlet and outlet of the analyser, in order to account for the flow resistance of the tubes. The material of the tubes should be appropriate for the gas of interest; silicone tubes must not (!) be used due to the highly permeability for CO_2. Special tubes are recommended for studying the isotopic composition of gases (e.g. PTFE, Tedlar© or aluminium-coated tubes). Longer tubes should be thermally insulated

TABLE 1 Detectors that can be used for measurement of GHG

Analyte	Common detector	Alternatives
N_2O	ECD	HID, mass spectrometer
CH_4	FID	HID, TCD
CO_2	FID + Methaniser	ECD, TCD, HID

to avoid condensation of water inside the tubes. The use of particle and water filters at the analyser inlet is highly recommended.

3.2 | Gas chromatographic analysis

3.2.1 | Gas chromatograph laboratory infrastructure

Many aspects of GC analysis strongly depend on the available instrumental set-up. Yet, some general recommendations can be given. A suitable laboratory infrastructure is required, that is a good gas supply system, with gas pipes of minimal length and ideally purifiers installed directly at the GC gas inlets. GC systems should be operated in a laboratory with controlled temperature. An uninterruptible power supply stabilising voltage and current should be installed to avoid damage due to power blackouts. GC systems include specific columns and detectors that allow for separation and quantification of gas species. An autosampler system allows measuring a sequence of samples automatically. Air samples contain water vapour, which can deteriorate the system's performance. Therefore, a GC system must include a backflush system to separate the analytes from undesired components.

Different detectors can be used for the three gases—N_2O, CO_2 and CH_4 (Table 1). N_2O is commonly detected using an electron capture detector (ECD), which contains a radioactive nickel foil, that is special radiation safety rules apply. The best sensitivity can be achieved in a gas mixture of 90% argon (Ar) and 10% CH_4, but nitrogen or helium carrier gases are often sufficient. Care should be taken regarding a good separation of N_2O and CO_2. A (pulse discharge) helium ionisation detector (HID) is a suitable alternative to detect N_2O. It can additionally measure other gas species, including CO_2, CH_4, N_2 and O_2. Helium (He) of extremely high purity is then required as carrier gas. This high purity can practically only be achieved by means of a gas purifier. Coupling a mass spectrometer with the GC is another option to measure N_2O.

CH_4 is usually detected using a flame ionisation detector (FID), which requires a gas supply of hydrogen (H_2) and synthetic air. A thermal conductivity detector (TCD) is an alternative for detection of higher than atmospheric CH_4 concentrations.

CO_2 can be detected using an FID after reduction to CH_4 (using a 'methaniser') with H_2. CO_2 can also be detected with an ECD (Loftfield et al., 1997), which may result in a conflict with optimisation for N_2O detection but simplifies the GC system and saves costs. TCD and HID are alternative CO_2 detectors.

3.2.2 | Calibration and quality control

Standard gases should be mixtures of trace concentrations of the three GHGs in synthetic air. Their quality is crucial for overall measurement quality and should be certified with ±1% analytical tolerance (or even less). A minimum of four standards are recommended, which should span the concentration range of typical gas samples equally. Calibration models should be selected according to the detector characteristics in the planned measurement range. Special attention should be given to the lower end of the calibration curve, since all gas samples will be in this range if fluxes are small. Strong nonlinearity in the calibration curve is usually an indicator of problems. It is highly recommended to calibrate the GC before each autosampler run, and to add calibration standards in the middle of long runs (>60 samples). Detection limits can be estimated following ISO procedures (ISO, 2000, 2008). Most GC software programs allow for calculating uncertainty of sample concentrations.

For quality control purposes, basic diagnostic values such as the baseline signal or calibration parameters should be monitored and recorded weekly. Regular tests should check possible sample carryover and possible changes in the repeatability of a close-to-ambient standard 10 times in a row. A coefficient of variation <0.5% is ideal (de Klein & Harvey, 2012; Harvey et al., 2020). Values of 1%–2% are acceptable, whereas a value >3% usually indicates problems that need to be fixed. Autosampler runs must always include control samples. Gas samples from the same flux measurement should be analysed in direct succession in order to minimise possible drift effects. Samples should be stored at laboratory temperature, in dry conditions and not exposed to sunlight, for example in a cupboard. Vials should be thoroughly tested, for example, for maintenance of overpressure, (near) vacuum and gas composition over time. Tests should be repeated if new septa are ordered, since the composition of the septa may change between production units and they may absorb or emit GHGs. A comparison found some slight advantages of Exetainers® over crimp top vials (Glatzel & Well, 2008).

3.3 | Online gas measurements

3.3.1 | Analyser types and manufacturers

The scientific question, experimental design and chamber system determine the gas concentration range and the accuracy and precision needed for the analysis of the respective gas species. Meeting these requirements is essential for any method of gas analysis including online gas analysers. Online gas analysers differ in many features, for example number and types of gases that can be measured, concentration ranges and precision, measurement frequency, minimum sampling volume and air flow, drift and temperature or matrix sensitivity, size, weight and power consumption.

Infrared gas analysers (IRGAs) have been available for decades and have facilitated CO_2 online measurements substantially and were

included in many automated systems. IRGAs cover a large range from small sensors to larger systems.

During the last 15 years, new technologies enabled the development of fast gas analysers for measuring CH_4, N_2O as well as their isotopes and isotopologues that are suitable for online chamber measurements. Many rely on laser technology by measuring the absorption in narrow and precise bandwidths of radiation. They are usually operated in a continuous flow-through mode and have a high measurement frequency (>1 Hz) and precision. Many of these analysers available today allow for the measurement of several gas species at once. Laser technology is advancing rapidly and the established and new manufacturers are developing analysers that might become interesting for GHG measurements in the future: Additional modules have been developed for small discrete volumes of gas samples (e.g. 20-mL gas vials) that allow using these analysers in a way similar to the traditional laboratory GC.

Mobile field gas analysers using other technologies such as photoacoustic spectroscopy or Fourier transform infrared spectroscopy are able to measure multiple gas components at once (Warlo et al., 2018), but require a sequential gas sampling, which therefore limits the sampling and measurement frequency (<0.05 Hz).

3.3.2 | Practical limiting factors and typical challenges of laser-based gas analysers

Online gas analysers allow for checking and optimising chamber measurements directly in the field, that is to choose the optimal chamber deployment period. The higher sampling frequency (compared to sampling with vials) yields more data points, which allows for much shorter chamber deployment periods and better minimum detection limits at the same time (Brümmer et al., 2017). It also allows for an easier identification of the linearity of the initial concentration change in the closed chamber (Figure 3). The usually low maintenance requirements of online gas analysers also make them suitable for automated measurements. Weight and power consumption of some models have been substantially reduced to facilitate mobility.

Yet, many online gas analysers for CH_4 and N_2O are expensive and rather heavy (compared to gas sampling with vials), and not all models are ready yet for truly mobile applications in harsh environments. Power supply can be a limiting factor for manual and automatic measurements in remote areas. Power blackouts can result in data loss and may even damage the operating internal computer system. Particles and water in the air stream can severely damage the gas analysers. For these reasons, routine backups on external devices and protective filters are highly recommended for online analysers. Interference with other gases can result in a loss of precision or even drifting measurement values (Gralher et al., 2018; Warlo et al., 2018). Temperature can affect the analysers' performance, so it is important to warm up analysers before starting the chamber measurements and to maintain stable conditions while measuring. When working with closed chambers in a flow-through set-up with online analysers, the volume of all tubes and the effective internal volume (corrected by the pressure) of the analyser need to be accounted for. Since the measurement cavities of some laser analysers operate at a very low pressure, any leakage along the cavity results in a constant intrusion of ambient air into the system, which can substantially affect the flux measurement. It is important to regularly check the analyser for airtightness. Vibrations can be very problematic for gas analysers and should be avoided during measurements. Analysers should thus be transported very carefully.

3.4 | Summary of tips for chamber handling and gas analysis

- **Chamber handling:** Initial pressure fluctuations during chamber placement should be avoided.
- **Chamber handling:** The chamber deployment period should be as short as possible and as long as necessary.
- **Manual gas sampling:** Manual sampling can be done with syringes or vials. Pumping with syringes prior to sampling is not recommended. At minimum, four air samples should be taken during a chamber measurement.
- **GC analysis:** The GC system should be optimised, and stable and suitable conditions and routines in the laboratory should be maintained. The quality of the measurements and GC system should regularly be checked. Calibration gases and routines should be adapted to the needs of the study.
- **Online gas analysers:** Online gas analysers can enhance the efficiency and quality of chamber measurements, yet it is essential to consider the technical limits (detection limits, precision, minimum detectable fluxes (MDFs), mobility, temporal resolution) and the available resources (e.g. manpower, time, money). Online gas analyser systems should be regularly checked for leaks, as well as data should be checked directly in the field.

4 | AFTER THE MEASUREMENTS: WORKING WITH THE DATA

4.1 | Flux estimation

4.1.1 | Estimating diffusive fluxes—Linear or nonlinear?

Different calculation methods can be used to calculate fluxes from the measurement of the time course of the gas concentration within the closed chamber. A simple linear regression model (LM; Table 2) can be used to estimate the flux (Lundegårdh, 1927), providing the change in gas concentration in the closed chamber is linear. Since concentration measurements can include substantial errors due to sampling and detection problems, robust linear regression (Yu & Yao,

TABLE 2 Regression methods used for flux estimations using non-steady-state chambers

	Method	Reference
LM	Linear regression model	Lundegårdh (1927)
LMR	Robust linear regression	Yu and Yao (2017)
HM	Nonlinear model of Hutchinson & Mosier	Hutchinson and Mosier (1981)
HMR	HMR implementation by Pedersen	Pedersen et al. (2010)
QM	Quadratic model	Wagner et al. (1997)
EM	Exponential model	Kutzbach et al. (2007)
NDFE[a]	Non-steady-state diffusive flux estimator	Livingston et al. (2006)

[a]Only for bare soil.

2017) is recommended to identify outliers. To consider the effect of a changing concentration gradient between soil and chamber headspace over time (Figure 3), a nonlinear regression model was developed (Hutchinson & Mosier, 1981) (HM; Table 2) and later modified (HMR; Table 2) (Pedersen et al., 2010). Further detailed physical consideration of the soil–chamber–atmosphere environment led to the development of the *Non-steady-state Diffusive Flux Estimator* (NDFE; Table 2) (Livingston et al., 2005, 2006). Gas transport modelling of chamber designs allows evaluating chamber set-ups (Sahoo & Mayya, 2010; Well et al., 2019); however, they are not always easy-to-use. In contrast to these physics-based estimation approaches, there are nonlinear empirical approaches (EM, QM; Table 2), which simply fit regression models to the chamber data in order to derive the pre-deployment or initial gas concentration change.

Chamber measurements with online analysers often demonstrate the nonlinearity in the concentration change over time (Kroon et al., 2008; Kutzbach et al., 2007). A comparison showed that increasing chamber height, area and volume significantly reduced flux underestimation by reducing the nonlinearity in the concentration change (Pihlatie et al., 2013). Nonlinear models often yield better statistical fits than linear regression due to the higher number of fitted parameters which does not necessarily indicate a lower uncertainty of the flux estimate (Levy et al., 2011). Regression analysis should not be conducted on less than four concentration measurements to get a reliable flux and uncertainty estimate. More data points reduce the uncertainty of the flux estimate. Thus, online measurements should be preferred over manual sampling. If a gas is manually sampled, a first gas sample immediately after chamber placement should be taken because the accuracy of the concentration at this initial sampling time is crucial for overall flux accuracy (Rochette, 2011).

The uncertainty associated with choosing a regression model for estimating the slope of the concentration increase at t_0 in the chamber headspace is the most important source of uncertainty for gas flux estimation based on manual gas sampling (Levy et al., 2011). We recommend using routines that test different flux estimation approaches and determine the most suitable method for each chamber measurement based on selected criteria (e.g. Hüppi et al., 2018; Venterea et al., 2020), so that reproducibility can be ensured. Many research groups developed calculation packages for this purpose (Table 3). There is no general consensus regarding the most appropriate method for estimating fluxes based on closed chamber data. A good option is to adjust the measurement design to promote increased linearity (Venterea & Baker, 2008), for example by a higher chamber and a shorter chamber deployment period (or only data from the initial minutes of a measurement). Practically, it is often necessary to find compromises in the sampling scheme when several gases are studied (CO_2, CH_4 and N_2O), as the respective gases have different flux rates and thus different accumulation behaviour in the chamber. While small fluxes (e.g. N_2O) might result in a quasi-linear increase, larger fluxes (e.g. CO_2) may require nonlinear flux estimation, or using only the concentration measurements of the first minutes.

4.1.2 | Detection limits, artefacts and trade-offs

When fluxes are small, the quality of the $\partial ngas/\partial t$ estimation deteriorates and eventually fails. It is important to know the sensitivity of the experimental set-up and to know MDF. Fluxes below the MDF value are not necessarily zero, but they cannot be detected with the required statistical confidence (Christiansen et al., 2015; Nickerson, 2016). MDF depends mainly on the analytical precision of the instrument, P_I, the

TABLE 3 Methods for flux estimations based on non-steady-state chamber data

Flux estimation package	Reference	Methods (for abbreviations, see Table 2)
R-Script-CH4_version_1.0.R[a]	Hoffmann et al., 2017	LM
flux	Jurasinski, et al., 2014	LM, LMR[b]
gasfluxes	Fuss et al., 2020; Hüppi et al., 2018	LM, LMR, HMR, with automatic decision function
HMR	Pedersen et al., 2010	LM, HMR, with semi-automatic decision
FluxCalR	Zhao, 2019	LM
chamberflux	Eckhardt & Kutzbach, 2016; Kutzbach et al., 2007	LM, EM, polynomial models; ordinary least-squares regression, many model selection criteria

[a]Detects ebullition.
[b]Uses median-based linear regression.

deployment period of the chamber, t_c, and the sampling periodicity p_s, but also on the dimensions of the applied chamber system such as the chamber height (V_{ch}/A) (Equation 4):

$$\mathrm{MDF} = \left(\frac{P_I}{t_c \sqrt{\frac{t_c}{p_s}}} \right) \left(\frac{V_{ch} p_{air}}{A R T_{air}} \right). \quad (4)$$

Thus, reducing the chamber height improves the MDF, yet it also increases the probability to get nonlinear increases in chamber measurements of high fluxes. Rounding significant digits of the concentration may result in a larger MDF and should be avoided.

Ebullitive fluxes may contribute substantially to ecosystem fluxes of CH_4, for example, in flooded peatlands (Green & Baird, 2013). Gas bubbles trapped below the water or peat surface may be artificially mobilised by vibration or pressure fluctuations during chamber deployment, so that extreme care is required when fluxes are measured. Different methodologies have been proposed to quantify ebullitive fluxes, either by modifications to the technical design or by detection of irregularities in the concentration increase in regular chambers. Online measurements can be used to detect such irregularities, that is sudden increases of the chamber headspace CH_4 concentration (Hoffmann et al., 2017). In a similar way, artefacts during the first few seconds after chamber placement can be identified, which may result from pressure changes induced by chamber closure (Figure 3). It is recommended to discard these data points.

4.2 | Working with flux data

Study objectives, design and data structures are diverse. In many cases, annual GHG balances are derived from time series of chamber measurements, but shorter studies focussing on, for example, processes or spatial patterns are also common. High inter-annual variability and uncertainties of annual balances illustrate the need to ensure the quality of later meta-analyses and GHG reporting. A general, standardised, best solution for all studies does not exist since the specific challenges to each study vary widely. Yet, some general recommendations can be given. We recommend estimating fluxes directly after the measurement to check flux uncertainties and plausibility compared to prior measurements. A well-developed QA/QC working protocol addressing all points discussed previously in this manuscript should be followed.

4.2.1 | Dealing with uncertainty in flux estimations

Flux estimates include uncertainty that should be included in the following evaluation instead of simply discarding flux estimations where the regression does not meet the required significance level. However, if there is a good reason to assume that the flux estimates are erroneous, for example due to documented problems during sampling or concentration measurement, they should be discarded. It is noteworthy to mention that fluxes below the MDF should not be treated as zero fluxes or be discarded.

4.2.2 | Greenhouse gas balancing and treatment evaluation

Manual chamber measurements often result in time series with very sparse data. Automatic chambers typically yield a higher temporal resolution but may still contain considerable data gaps due to technical failure or weather-induced breaks. These gaps have to be filled in a meaningful way to derive seasonal or annual balances. Too few measurements result in high uncertainty of the derived GHG balances (Gana et al., 2018; Lucas-Moffat et al., 2018). Strategies to fill the gaps reach from simple averaging and linear interpolation (mostly CH_4 and N_2O fluxes) to more advanced empirical modelling (mostly CO_2 fluxes) based on physiological processes to pure statistical models like neural networks. These approaches depend on parameters, like soil or air temperature, soil moisture, radiation and so forth, that are available at higher temporal resolution without gaps (Liu et al., 2022; Ooba et al., 2006). The availability and sampling of these proxy data must be included already in the planning of the study design.

Many gap filling approaches rely on statistical modelling. One widely applied approach for CO_2 fluxes is to use separate models for GPP and ecosystem respiration (R_{ECO}). GPP is mainly controlled by PAR and by the amount of photosynthetically active tissue (Chapin et al., 2011) (if temperature or soil moisture is not limiting). R_{ECO} is mainly driven by temperature and soil moisture (Janssens et al., 2001). Several models for both parameters have been proposed in the literature (see *flux* package for an overview). In the absence of significant relationships between fluxes and available environmental parameters, fluxes can either be averaged and multiplied by the covered time or integrated over time (Koebsch et al., 2013). Averaging was used for many early GHG studies, but integration over time is better in case of pronounced seasonality. Yet, both approaches do not allow estimating the interpolation error. An uncertainty estimate of the annual flux can be derived by the statistical procedure of resampling the measured fluxes (bootstrapping). The uncertainty of the individual flux estimate can be included in such a bootstrapping approach that uses multiple flux data sets based on the observed standard error of the flux estimate. Annual fluxes should first be calculated for each chamber base before averaging across chamber bases. As there is no standard procedure to obtain uncertainties for annual or seasonal flux estimates, it is important to clearly describe the methodology used. Steps towards a structured evaluation of the different methods (Liu et al., 2022) are valuable and should be further developed.

4.2.3 | Studying processes and spatial patterns

Soil–atmosphere gas fluxes vary spatially which is often addressed by using replicates to calculate representative average values. Instead of simple average values, regionalisation methods (e.g. kriging) can be

used if spatial patterns can be detected. Understanding the spatial variability and the interdependence of GHG can help to understand the underlying processes (Jurasinski et al., 2012; Maier et al., 2021; Warner et al., 2017). Chamber measurements are also used to study transport, transformation and allocation in the soil–vegetation–atmosphere system. Manipulation experiments such as trenching (Subke et al., 2006) or the use of isotopically labelled material or natural isotope abundance approaches allow separating different contributing processes (Brüggemann et al., 2011; Well et al., 2019). Chamber measurements are also used to quantify gas fluxes from tree stem surfaces (Barba et al., 2019; Maier et al., 2018). Even though the focus of such studies might be very different, recommendations generally remain the same to consider the uncertainty in flux estimates and the importance of documentation of how flux estimates were derived and aggregated.

4.3 | Data handling and documentation

Storage of all data in a database is strongly recommended to handle the large data volume and complexity of data sets acquired in GHG studies. Raw data should always be stored alongside any derived or aggregated data, in order to allow for re-analysis. Data with limited quality should be flagged rather than deleted. Storage of all raw data, aggregated data and metadata required for flux estimation including the necessary documentation and subsequent data analysis ensures transparency and repeatability, and thus better science. A table of data and parameters relevant for GHG studies and recommended for internal documentation, ranging from the description of sites and plots to the chamber set-up and the flux estimation routines, is given in the full Guideline as an example (Fiedler et al., 2022). While a complete and structured documentation is essential for all studies, data reporting for publication represents a selection thereof which should include all essential information that allows others to comprehend and reproduce the interpretations of the published study. Minimum requirements include details about *Location*, *Site information*, *Study design*, *Chamber design*, as well as the actual measurements, that is gas sampling and analysis methods including the calibration of the measurement devices and the method of flux calculation and data analysis.

All data should be regularly saved on a durable storage medium in order to ensure an appropriate backup practice. We encourage researchers to archive their data sets for a minimum time at least 10 years as recommended, for example, in the Protocol of the German Science Foundation (DFG) for Good Scientific Practice (Deutsche Forschungsgemeinschaft, 2013). Ideally, all data should be published open-access alongside the respective publications. This will improve the availability of GHG flux data for meta-analyses, data interpretation and thus the overall value for the scientific community. We also recommend publishing experimental data (additional to data reported in a publication) in online data repositories and data journals which can then be used for meta-analyses and GHG emission modelling. Here, additional information is often needed in predefined formats. More detailed lists of data relevant for publications in journals or online data repositories are included in the full Guidelines as an example.

5 | CONCLUSIONS

In recent years, measurement technologies have substantially improved, for example, with laser systems that enable online measurements with higher measurement frequency and accuracy, which are also used more frequently with automatic chamber systems. Through the combined use with other measurement methods, for example with eddy covariance or with continuous environmental parameters of climate stations or soil parameters, which can be used as predictors for modelling approaches, an ever-better data basis can be created. Nevertheless, even with the technical progress mentioned above, a targeted and efficient use of the available resources is necessary, taking into account the respective study objectives and challenges, as well as a critical discussion of the implicit uncertainties.

Although this guideline focuses on GHG measurements primarily in terrestrial ecosystems, many of the considerations discussed can also be applied to challenges in other environments, such as measurements of gaseous exchanges between the atmosphere and aquatic systems or measurements of reactive gases (e.g. ammonia or biogenic volatile organic compounds), and is also highly relevant for studies of the isotopic composition of gas fluxes. Hence, the presented guideline contributes to improving the quality and post-usability of current and future measurement studies.

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DATA AVAILABILITY STATEMENT

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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