USDA-ARS GRACEnet Project Protocols
Chapter 3. Chamber-Based Trace Gas Flux Measurements[4]
November 2010 (replaces original version of April 2003)

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Scope:
This protocol addresses N_2O, CO_2 and CH_4 flux measurement by soil chamber methodology. The reactivities of other gasses of interest such as NO_x O_3, CO, and NH_3 will require different chambers and associated instrumentation. Carbon dioxide is included as an analyte with this protocol; however, when plants are present, interpretation of soil CO_2 flux data in the context of net GHG flux is not straightforward because soil CO_2 emissions do not represent net ecosystem CO_2-C exchange. This protocol adopts chamber-based flux methodology (the least expensive option available) in order to allow inclusion of as many sites as possible. Since micrometeorological techniques require expensive instrumentation, they will be used only at locations with current micrometeorological capability. In deciding on a chamber design, our goal was to adopt methodology which is sensitive, unbiased, has low associated variance, and allows accurate interpolation/extrapolation over time and space. Because of our inability, at this time, to precisely assess the extent of bias associated with a given chamber design and sampling protocol under the range of conditions which might exist, we have adopted our 'best guess' protocol. Assessment, refinement and/or modifications of this protocol may continue in the future. At some sites this may include evaluation of chambers against fluxes determined by micrometeorology or performing comparisons of alternate chamber designs. Recognizing that any measurement technique will have disadvantages, the best we can do at this time is to select a technique which minimizes potential problems. In addition, adoption of common methodology will aid in site inter-comparisons. To facilitate the adoption of a common technique, it is important to attain a common understanding of the potential shortcomings associated with chamber-based flux measurement techniques (Rochette and Eriksen-Haamel, 2008). The following section summarizes some of these issues.

Considerations for Chamber Construction and Deployment.
Several issues related to chamber techniques for gas flux measurement must be considered. These are discussed below along with recommendations to minimize potential problems.

1. Soil Disturbance: In the short term soil disturbance can occur upon installation of the anchor used to support the chamber. Longer term microclimate effects within the anchor have also been observed. Installed anchors may retain water and become flooded during high precipitation

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events. Humidity within the anchor may facilitate algal growth on the soil surface. Shading by the anchor may alter the temperature regime of the soil.

Recommendations: Install permanent chamber anchors at least 24 h prior to flux determinations. Minimize anchor or collar height to reduce micro environment perturbations. Move chamber anchors if soil microclimate effects are observed. Venterea et al. (2010) describe a chamber design using anchors that are nearly flush with the soil surface and thus would minimize microclimate effects (See Appendices III and IV). Pump excess or flooded water from chamber anchors as soon as possible. In situations where it is not feasible for chamber anchors to be installed in the soil, use temporary/portable chambers with a wind skirt (Matthias et al., 1980) may be considered.

2. Temperature perturbations: Temperature differences can have a marked effect on biological activity, and also cause gas expansion/contraction which can complicate flux calculations and create experimental artifacts if appropriate temperature corrections are not made. Absorption or dissolution of dissolved soil gasses is also impacted by temperature. The goal is to have the temperature regime within the chamber as similar as possible to the external temperature.

Recommendations: Use insulated chambers to help maintain a constant temperature regime during deployment. Reflective material can be used to construct or coat the chamber to reduce absorption of sunlight (e.g. double reflective insulation by Reflectix, Inc., www.reflectix.com). Keep chamber deployment time as short as possible without sacrificing detection sensitivity. Install thermocouple or thermometer in the chamber lid to monitor temperature changes throughout the incubation period.

3. Pressure perturbations: Wind passing over the chamber anchor may cause pressure-induced mass flow of gas into or out of the soil. Closed chambers placed on the soil can reduce natural pressure fluctuations. Sampling of gas within the chamber can cause mass flow of gas from the soil.

Recommendations: Pressure and sampling perturbations can be reduced using a properly vented closed chamber (Hutchinson and Mosier, 1981; Hutchinson and Livingston, 2001; Xu et al., 2006).

4. Humidity perturbations.   Humidity increases due to deployment of a chamber on the soil will effect trace gas concentrations in the chamber due to dilution by water vapor. For example, at $25^{o}C$ and 100% relative humidity, the maximum concentration of water vapor in the air is 0.0305 L/L. Thus, if the ambient relative humidity was zero, and placement of a chamber resulted in an increase in relative humidity within the chamber from 0% to 100%, the maximum dilution of other gases would be 0.0305 L/L or a dilution of 3.05%.   The potential underestimation of trace gas concentration in the chamber headspace due to dilution does not necessarily mean that the flux will be underestimated. If dilution changes the degree of curvi-linearity of the time course data, gas flux could be overestimated. The magnitude (and direction) of this effect depends on the magnitude of the soil gas flux as well as the chamber headspace height. In addition to trace gas dilution by water vapor other potential humidity effects include changes in soil water which could impact both soil biological activity and the amount of gas dissolved in the aqueous phase. Water vapor can also cause interferences in detection of other gases if a photoacoustic or other infra-red based analyzer is used.

Recommendations: Keep chamber deployment short. Relative humidity changes inside chamber could be estimated and used to potentially correct for dilution and/or gas solubility effects.

5. Gas Mixing: It is generally assumed that molecular diffusion is sufficiently rapid within the chamber headspace such that homogeneous gas concentrations exist when sampling (Livingston et al., 2006). However, this may not necessarily be true if large amounts of vegetation are present or the chamber volume to surface area ratio is large.

Recommendations: If it is deemed that mixing of the headspace gas is necessary, the best option is to fit the inside of the chamber with a gas distribution manifold connected to the sampling port. The manifold has a single port on one end (which extends out the top of the chamber) and multiple ports on the other end which accept small diameter teflon tubing (e.g., 1/16") that extend into the chamber. The narrow tubing from each of the multiple inner ports is extended to different points inside the chamber, so that when the sample is collected, gas is pulled from multiple points in the chamber. Manifolds can be purchased from Small Parts, Inc. (www.smallparts.com). An example part no. is B000P7KZ9Y (description = Stainless steel hypodermic tubing manifold, inlet - 13 Gauge, 6 outlets - 20 Gauge). The recommendation of placement of a small fan within the chamber, made in the previous version of this protocol is not advised. It has been observed that fans can induce pressure perturbations within the chamber.

6. Chamber Placement: In row-crop systems it is important that chambers be deployed to adequately represent the system. For smaller chambers this will necessitate placement of chambers in both the row and inter-row areas of the plot.   Alternatively, chambers with a larger footprint that provide more representative coverage of the system under study can be used, ideally utilizing chambers designed to cover the entire inter-row area. One goal of the GRACEnet project is to quantify ecosystem contributions to net trace gas flux; therefore, plants should be included inside chambers during flux determinations. There is some information indicating that N_2O emission may be facilitated by living plants (Chang et al., 1998; Chen et al., 1999; Smart and Bloom, 2001), however, this effect has only been observed under flooded conditions. Chambers must also be placed to sample other representative features of the system under study (e.g. tillage or fertilizer bands).

Recommendations: Inclusion of plants presents several problems. With regard to sensitivity, inclusion of plants would likely dictate that chamber height be increased, but an increase in chamber height results in an increase in chamber headspace volume and a corresponding decrease in flux detection sensitivity (minimum detectable flux limit is described below). Significant reductions in sensitivity might, in some cases, result in all the flux measurements being below the detection limit. In such cases, it is advisable to also measure bare soil fluxes (i.e. between rows in row-crop agriculture) using shorter chambers which have higher sensitivity. Results could then be reported as fluxes within a range of the bounds established by the two measurements. If it is not feasible to include plants at all growth stages, at least deploy chambers both within and between rows (in row crop agriculture). Inclusion of plants complicates interpretation of CO_2 flux data. Production of CO_2 by living plant tissue (both above and below ground) contained within the chamber cannot be considered in estimates of total GHG production unless annual photosynthetic CO_2 uptake is also measured.   Finally, when small chambers are used to sample distinct areas of the field that are not, in and of themselves, representative of the entire field (e.g. soil containing a fertilizer band vs. non-fertilizer band soil),

then a mathematical weighting of the fluxes must be performed in order to obtain the average field or plot flux.

7. Frequency and timing of flux measurements. Trace gas fluxes exhibit a high degree of temporal variability. Thus, the more frequently measurements are made, the more accurate the integrated seasonal/yearly cumulative flux estimate will be (Smith and Doobie, 2001; Parkin 2008). There are several components of temporal variability that must be considered including: i) diurnal variations, ii) seasonal variations, and iii) variations induced by perturbation (e.g., tillage, fertility, irrigation/rainfall, thawing).

Recommendations: To account for diurnal variability, measure flux at times of the day that more closely correspond to the daily average temperature (mid morning, early evening). A Q_{10} temperature correction procedure may used to adjust rates to the average daily temperature, but caution is warranted. The temperature correction procedure assumes that temperature variations are the primary factor driving diurnal flux variations, an assumption that may not be universally true. Selection of both the appropriate Q_{10} factor and the soil temperature (depth) to be used are critical. The time lag between gas production in the soil profile and gas flux from the soil surface will dictate the appropriate soil temperature to use in performing the Q_{10} flux correction. Finally, a wide range of Q_{10} values for N_2O have been reported in the literature Brumme et al., 1999; Dobbie et al., 1999; Dobbie and Smith, 2001; Machefert et al., 2002), so critical determination of the appropriate Q_{10} factor must be done. It is recommended that if a Q_{10} correction is performed, the original non-corrected fluxes should be reported as well. To account for perturbation-induced variations is recommended that fluxes be measured as soon as possible after the perturbation (such as rainfall, tillage, or fertility event), then daily for the next several days during and following the specific event. During the remainder of the year, gas flux measurements should be made at regular time intervals (every 1 or 2 weeks). It is highly recommended that fluxes be measured at least weekly and more frequently if resources allow (Parkin 2008).

7. Spatial Variability: Trace gas fluxes exhibit a high degree of spatial variability, and Coefficients of Variation associated with chamber-based fluxes commonly exceed 100%. Variability may also be a function of chamber size, and may be reduced by using larger chambers. Use of larger chambers can result in the physical 'averaging' of microsites, thus reducing variability (Parkin, 1987; Parkin et al., 1987). *Recommendations:* Use chambers with larger footprint to minimize small scale variability. Use as many chambers as possible. It is recommended that a minimum of two chambers per plot in plot scale studies. In landscape or field studies it is recommended that 'similar' landscape elements be identified and a sampling design employed where chambers are stratified by landscape element, soil type, or vegetation (Livingston and Hutchinson, 1995). In situations where identifiable hotspots may occur (e.g., urine patches in a grazed system) a sampling design will have to be developed to account for this. Gilbert (1987) gives some sampling guidelines when hotspots exist.

Recommended Protocol
Gas flux will be measured by static chambers deployed on the soil surface for a period of typically no more than 60 min. During chamber deployment, samples of the chamber headspace gas will be removed at regular intervals, and stored for later analysis by gas chromatography. Specific recommendations on chamber design, gas sampling and analysis, and flux calculations

are provided below. Investigators are encouraged to examine the referenced literature underlying these recommendations.

Minimum Requirements for Chamber Design:
1. Flux chambers should be fabricated of non-reactive materials (stainless steel, aluminum, PVC, polypropylene, polyethylene, or plexiglass.)
2. Material should be white or coated with reflective material, (mylar or painted).
3. Chambers should be large enough to cover at least 182 cm^2 of the soil surface, and have a target height of 15 cm (height can be decreased to increase sensitivity or increased to accommodate plants).
4. Chambers should contain a vent tube, at least 10 cm long and 4.8 mm in diameter (e.g., 1/4" stainless steel tubing). See Fig. 1 for details. Alternatively, Xu et al. (2006) describe a novel circular vent tube designed to completely eliminate wind-induced pressure gradients.
5. Chambers should have a sampling port to enable the removal of gas samples. Possible options include: butyl rubber septa or a nylon/polyethylene stopcock.

Recommended Design:
Chambers should have two parts; a permanent anchor, driven into the soil and a flux chamber cap which contains the vent tube and sampling port. Anchors are fabricated so that they can accommodate the flux chamber during measurement phase. Anchors and chambers can be made of 20 cm (or larger) diameter PVC. Alternatively, anchors can be made of thin-walled stainless steel or aluminum to minimize physical disturbance upon insertion. The vent tube is necessary to avoid pressure perturbations (and subsequent mass flow) when chambers are installed on the anchor, and when gas samples are collected. Photographs of several chamber designs are presented in Appendix III and descriptions of chamber construction are provided in Appendix IV. Some of the supplies and vendors of materials for chamber construction are provided in Appendix II.

Fig.1. Optimum vent tube diameter and length for selected wind speeds and enclosure volumes as described by Hutchinson & Mosier (1981).

Chamber deployment
Anchors: Anchors should be installed at least 8 cm into the ground and extend no more than 5 cm above the surface. Permanent anchors should be installed at least 24 h prior to first flux measurement. There are no fixed guidelines regarding how long anchors can (or should) be left in place. In cultivated systems, chamber anchors are typically removed prior to cultivation, planting, or fertilizer application, and then replaced. In grassland studies anchors have been left for over 10 years with no apparent deleterious effects. One advantage of leaving anchors in
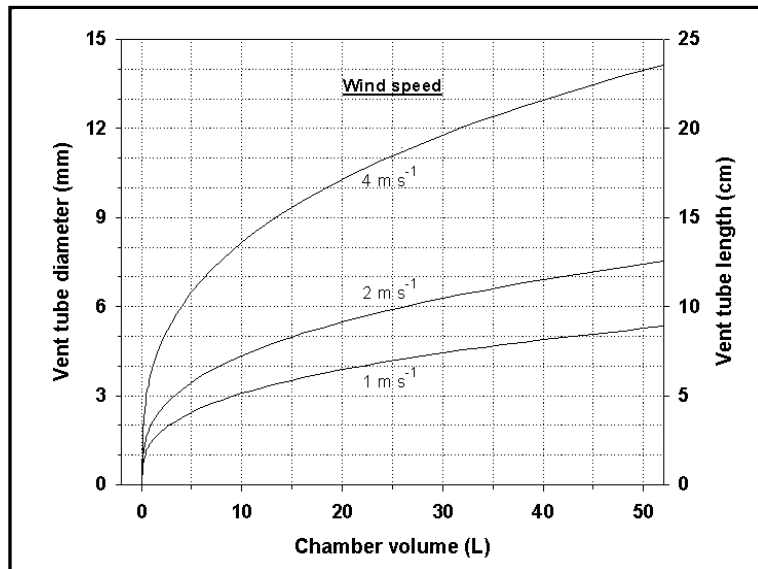
place is that soil disturbance and root damage are minimized. However, there have been reported problems with microclimate effects within the anchors left in place for extended periods. For example, changes in humidity or shading can cause algal growth, and in heavy or compacted soils ponding of rainwater can occur. This is not a desirable situation. It will be up to the investigator to determine how often chambers should be moved.
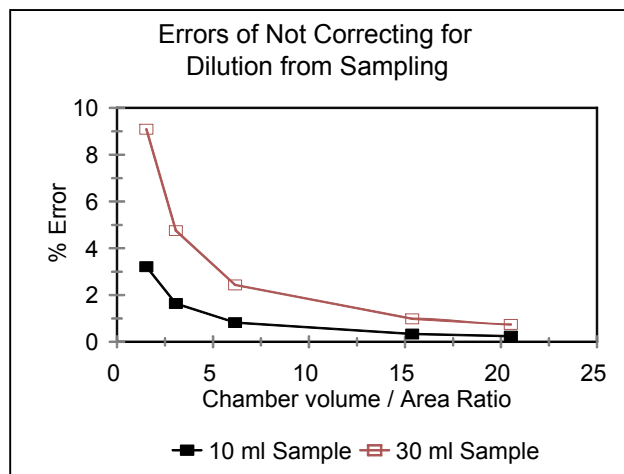

Fig. 2. Percentage underestimation of flux due to headspace dilution as a result of sampling. Presented as a function of chamber size and gas sample size.

Gas sampling: Fluxes are measured by determining the rate of change of trace gas concentration in the chamber headspace. In most cases trace gas concentrations are determined by physically removing a gas sample from the chamber headspace for analysis in the laboratory. Gas samples should be withdrawn at regular intervals during the chamber deployment. Chambers should be in place no longer than 60 minutes. The shorter time the deployment time, the smaller the chamber-induced biases, but deployment must be long enough so that sensitivity is not compromised. At least 3 time points are required for flux calculation: time 0, and two additional points, equally spaced in time (e.g. 0, 30, 60 min. or 0, 20, 40 min). *[Note: Sampling is performed at regular intervals to facilitate flux calculation by Eq. 1. However, more samples can be collected, and models exist for the analysis of data not collected at equi-spaced time intervals. see Flux Calculation Section, below. Using more than 3 time points will decrease uncertainty in flux calculations, but with an obvious trade-off in additional labor].* Sampling is performed by inserting a polypropylene syringe into the chamber septa and slowly removing a gas sample. Mixing of headspace gas by pumping the syringe before sampling is not recommended as pumping may cause pressure

perturbations and/or excess dilution of headspace gas by entry of outside air through the vent tube. The gas volume removed at each time point is dictated by the specific gas analysis technique to be used. Typically, from 5 to 30 ml are removed. If the syringe is equipped with a stopcock, the sample can be stored directly in the syringe for a short time. Alternatively, the gas sample can be transferred to a previously evacuated glass vial sealed with a butyl rubber septum. It is recommended that enough gas be injected into the evacuated vial to produce an overpressure. This overpressure facilitates the subsequent removal of a gas sample for analysis. It should be noted that each time a headspace gas sample is removed from the chamber outside, air flows into the chamber through the vent tube. This results in a dilution of the analyte in the chamber headspace. The error
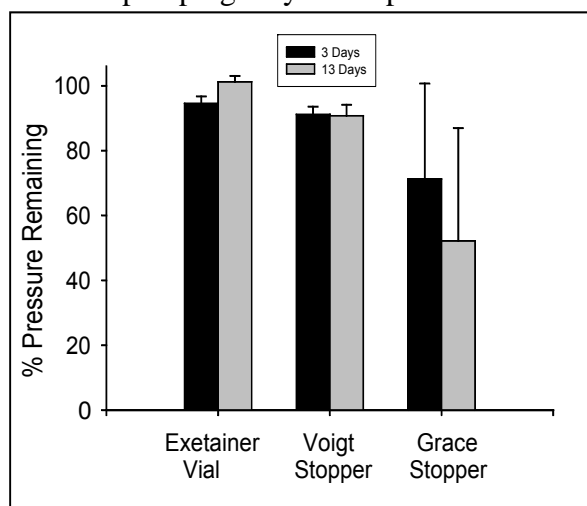

Fig. 3. Percent retention of 250 mbar overpressure in Exetainer vials and crimp top serum vials sealed with butyl rubber stoppers. Crimp-top vial stoppers were obtained from two different sources. Error bars indicate standard deviations.

associated with this dilution effect is a function of both the sample volume withdrawn and the chamber Volume/Surface Area ratio (Figure 2). Correction for this dilution effect should not be necessary for chamber Volume/Surface Area ratios >10 and sample volumes < 30 ml. Prolonged storage (>2 d) of gas samples in polypropylene syringes is not recommended as leakage can occur. An example of a gas sampling protocol is presented in Appendix I. In some situations instrumentation may be available for real-time analysis of headspace gasses. Infra-red gas analyzers have been successfully used for determining soil CO_2 fluxes. Photoacoustic analyzers reportedly can measure a suite of gasses (e.g. CO_2, N_2O, CH_4, H_2O). However, we recommend extreme care in such circumstances. The combination of the overlap of the absorption spectra of the different analytes, combined with the large range in analyte concentrations (e.g. CO_2 ~ 380 ppm vs. N_2O~ 320 ppb) in air pose potential problems for precise estimation of fluxes. Interferences of water vapor with CH_4 determinations have been noted (Parkin, unpublished). Similarly, CO_2 interferences with N_2O have been observed (Akdeniz et al., 2009). If photoacoustic analyzers are used it is highly recommended that calibrations be performed with mixed gas standards where the relative concentrations of the different gasses are changed. Alternatively, if N_2O is the analyte of interest, a soda lime trap can be installed to scrub CO_2 from the gas sample stream.

Vials, septa and storage: Brooks (1993) evaluated several storage protocols and found that red rubber stoppers such as found on commercially available evacuated blood vials were the worst. Parkin (1985) observed that red rubber absorbed N_2O. Recently, Glatzel and Well (2008) tested the integrity of red rubber stoppers, grey butyl rubber stoppers, and Exetainer vials (with grey butyl rubber septa) after repeated needle punctures. These investigators observed pressure losses of approximately 94%, 84% and 30% following 5 needle punctures of butyl rubber stoppers, red rubber stoppers, and Exetainer septa, respectively. However, we have observed marked differences in quality of grey butyl rubber stoppers obtained from different venders. We repeated the Glatzel and Well experiment with two batches of grey butyl rubber stoppers on glass crimp-top serum vials and with Exetainer vials. The septa or stoppers were punctured 5 times with a 22 gauge needle and the vials were then injected with room air to achieve a 250 mBar overpressure. Pressure in the vials was determined after 3 d and 13 d (at room temperature and pressure) with a pressure transducer. In our experiment the Exetainer vials (Labco Limited part # 938W) and 6 ml serum vials (Alltech Associates) with grey butyl rubber septa obtained from Voigt Global (part # 73828A-RB) maintained > 90% of the overpressure for 13 d, and had low variability (Fig. 3). However, average pressure retention of the Grace stoppers was 71% at 3 d, and dropped to 60.8% at 13 d. The Grace stoppers were also highly variable, as indicated by the standard deviation bars. Rochette and Eriksen-Haamel (2008) characterize the use of Exetainer vials (which have grey butyl rubber septa) as the "best" practice, and use of vials with other butyl rubber stoppers as 'good'. We agree with this recommendation. In our evaluation of butyl rubber stoppers obtained from a variety of different venders we observed marked differences in efficacy of sample integrity for different batches of butyl rubber stoppers. Because of the variability in the quality of butyl rubber stoppers on the market, **it is highly recommended that the rubber stoppers of each new batch be tested for the ability to hold vacuum and pressure.** Additionally, we have observed that gas leaks can occur around the stopper in crimp top serum vials if the crimp is not applied tightly. When a manual crimping tool is used variability among individuals can be a factor and care must be taken to assure adequate crimping pressure. A recommended strategy for dealing with gradual loss of pressure

from vials requiring storage prior to analysis is to overpressurize them initially (as recommended above), and then immediately prior to analysis de-pressurize them by inserting a small needle through each septum for a few seconds.   This procedure will ensure that all vials are at the same (ambient) pressure at the time of analysis, and also allows for an assessment of sample integrity (i.e., samples that have not held pressure are suspect). This procedure also accounts for the fact that different vials are likely to leak at different rates. Standards must be treated in the same way, and the sample injection system (e.g. autosampler) used must be capable of handling samples at ambient pressure. This procedure has been used successfully over several years (Venterea et al., 2005; 2010).

Gas Analysis: Samples should be run as soon as possible after collection. Gas chromatography will be used for analysis of N_2O and CH_4 (electron capture detector for N_2O and flame ionization detector for CH_4). Specific method of gas sample injection into the GC will depend upon the specific instrumentation available at each location. However, it is recommended that the GC be fit with a sample valve to minimize injection error and thus increase analytical precision. To account for problems associated with GC drift it is recommended that: i) samples from individual chambers are run in sequence (e.g. t0, t1, t2,) rather than segregating all the samples by time (i.e. all t_0 samples run in sequence, then all t_1 samples run in sequence, etc.) and ii) standards are run periodically throughout the sample run (e.g. every 10 to 20 samples).

Standards: Standards should be prepared each sampling time. Standards should be handled in a manner similar to samples with regard to collection and storage. Preferably samples should be prepared in the field (i.e. injected into glass vials, or collected in syringes). Several different standard concentrations should be run, as detector response may be nonlinear. The range of standards should bracket the concentrations found in samples [e.g., N_2O; 0.1, 1.0 and 10 ppm; CH_4; 0.5, 1, 2, and10 ppm). Standard curves are then used to convert the GC output of the samples into units of ppm. It has been noted that occasionally the stated concentration on purchased standard gasses may be erroneous (A. Mosier, pers. comm.). With some gas chromatographs, oxygen has been observed to influence N_2O detection sensitivity when measured with an electron capture detector (Parkin, unpublished). The specific gas chromatograph used should be checked for this effect, and the make-up gas of all standards should reflect the gas composition of the atmosphere (i.e. approx 20% O_2 and 78% N_2). It is recommended that funds be allocated in the GRACEnet project to purchase a common NIST-certified standard gas mixture to be used to check standards at all locations.

Data Analysis
Flux Calculations: Fluxes are calculated from the rate of change of the concentration of the analyte of interest in the chamber headspace. Since the units associated with the gas standards will typically be ppm, when the standard curve relationship is applied to calculate gas concentrations of the samples, the resulting unit of the analyte is also ppm. The units, ppm, are typically on a volume per volume basis (which is the same as a mole per mole basis). Volumetric parts per million (ppm(v)) has units of μL trace gas L^{-1} total gas.   For example a 1 ppm (vol/vol or mol/mol) N_2O standard will contain $1\mu L$ N_2O / L of gas. If the rate of change of headspace trace gas concentration is constant (i.e. ppm(v) vs. time data is linear), then linear regression can be used to calculate the slope of the concentration vs. time data. The slope of the line is the trace gas flux. Thus, a regression of ppm(v) vs. hours will result in a slope with units of μL gas L^{-1} h^{-1}.

Multiplying the slope by the chamber volume (L) and dividing by the chamber surface area (m^2) will result in a flux with units of μL trace gas m^{-2} min^{-1} . If the rate of change of headspace trace gas concentration is not constant (i.e. ppm(v) vs. time data is curvi-linear), then linear regression may not be appropriate. Curvi-linear concentration data with time is attributed to a build up of the analyte concentration in the chamber headspace (Hutchinson and Mosier, 1981), which alters the diffusion gradient and the resulting flux (Hutchinson and Mosier, 1981), or to horizontal movement of gas in the soil (Livingston and Hutchinson, 1995), or to leakage from the chamber (Stolk et al., 2009). To account for this effect, Hutchinson and Mosier (1981) proposed an algorithm as an alternative to linear regression (Eq. 1).

$$f_o = (C_1 - C_0)^2 / [\, t_1 \text{ x } (2 \text{ x } C_1 - C_2 - C_0)] \text{ x } \ln[(C_1 - C_0)/(C_2 - C_1)] \qquad \text{Eq. [1]}$$

where f_o is the flux at time 0, C_0, C_1, and C_2 are the chamber headspace gas concentrations (ppm(v)) at time 0, 1, and 2, respectively, and t_1 is the interval between gas sampling points (h). The resulting units of f_o are: μL trace gas $Liter^{-1} h^{-1}$. In order to convert these units to μL trace gas $m^{-2} h^{-1}$, f_0 must be multiplied by the chamber volume (Liters) and divided by the chamber surface area (m^2)

In addition to the Hutchinson and Mosier (HM) method, there have been several alternative methods proposed for the analysis of curvi-linear data. The quadratic procedure described by Wagner et al., (1997) involves fitting a quadratic equation to the concentration vs. time data (Quad method). The flux is then computed as the first derivative of the quadratic equation at time zero. Pedersen et al. (2001) developed a stochastic diffusion model that is an extension of the HM method and does not require equi-spaced data points, and can accommodate more than three data points. The non-steady-state diffusive flux estimator (NDFE) developed by Livingston et al. (2006) is a 3 parameter model in which f_0 can be derived from concentration vs. time data by non-linear regression. Recently, Pedersen et al., (2010) developed a technique designated as the HMR model, which is a modification of the Hutchinson/Mosier technique to account for horizontal gas diffusion and/or chamber leaks. Similar to the Pedersen stochastic model, the Quad method and the NDFE model, the HMR technique can be used with data sets of 3 or more points. However, as alluded to by Pedersen et al. (2010), the practice of evaluating a 3 parameter model (i.e. HMR or Quad or NDFE) with only 3 or 4 data points is not optimal. Such situations may result in parameter estimates being non-significant.

What is the best method? Several criteria must be considered in the selection of an analysis technique to apply to a given data set. Past studies have evaluated some of the aforementioned methods with regard to the bias (accuracy) associated with the calculated flux estimate (Livingston et al., 2006; Venterea et al 2009; Venterea 2010; Pedersen et al., 2010). However, in addition to bias, the variance associated with the calculation method must also be considered. Every analytical technique for gas measurement has an associated error. In the case of gas chromatography, the precision (coefficient of variation) of the gas measurements is often in the range of 1 to 6% when small (0.2 to 1.0 ml) gas samples are used. The error associated with gas measurement (as well as other sampling errors) can result in the occurrence of "noisy data" (Anthony et al., 1995), and this "noise" induced by sampling and analytical variability can introduce a variance component to the flux estimation method. Thus, in addition to bias, the variance of the flux estimation method should also be considered.

A statistical analysis by Venterea et al. (2009) demonstrated that clear trade-offs exist between bias and variance in selecting a flux-calculation scheme, with linear regression having greater bias but less variance compared with the HM and Quad methods. Parkin and Venterea (manuscript in preparation) investigated these issues further, using Monte Carlo simulation to evaluate the bias and variance of linear regression, the HM method, and the Quad method when applied to data sets of 3 or 4 points, with chamber deployment times of 0.5 h, 0.75 h and 1.0 hour, and different degrees of data curvi-linearity. Monte Carlo simulations were performed by constructing simulated N_2O chamber data using the method described by Venterea et al. (2009). This analysis was applied over a range of analytical precisions (1% to 6%). When an estimation method has both bias and a variance component, the appropriate selection criterion is the Mean Square Error (MSE) which combines the bias and variance (Eq. 2) (DeGroot, 1986).

$$MSE = Variance + Bias^2 \qquad\qquad Eq. 2$$

Our analyses showed that there is not a simple answer to the question, "Which flux calculation method is the best?" The MSE of a given flux calculation method is dependent upon three factors: 1) the magnitude of the underlying flux, 2) the degree of data curvi-linearity, and 3) the analytical precision. For example, Fig. 4 shows the MSE associated with linear regression, the Quad method and the Hutchinson/Mosier method across a range of simulated N_2O fluxes for a given chamber height, deployment time, and GC precision. The points where the curves intersect indicate decision points for the different calculation methods. Below 22 μg N m^{-2} h^{-1} linear regression has a lower MSE than either the Quad or HM methods, thus it is the method of choice. At fluxes between 22 and 52 μg N m^{-2} h^{-1} the Quad method has the lowest MSE, and for fluxes > 52 μg N m^{-2} h^{-1} the HM method has the lowest MSE and should be used. These flux decision points are only valid for data sets with a certain degree of curvi-linearity (controlled, in part, by chamber height, deployment time, and soil characteristics), and an analytical precision of 2%. As data curvi-linearity and analytical precision change, the decision points for the different methods also change. While quantifying analytical precision is relatively straightforward, characterizing the degree of data curvi-linearity is not. Here we propose a calculation that can be
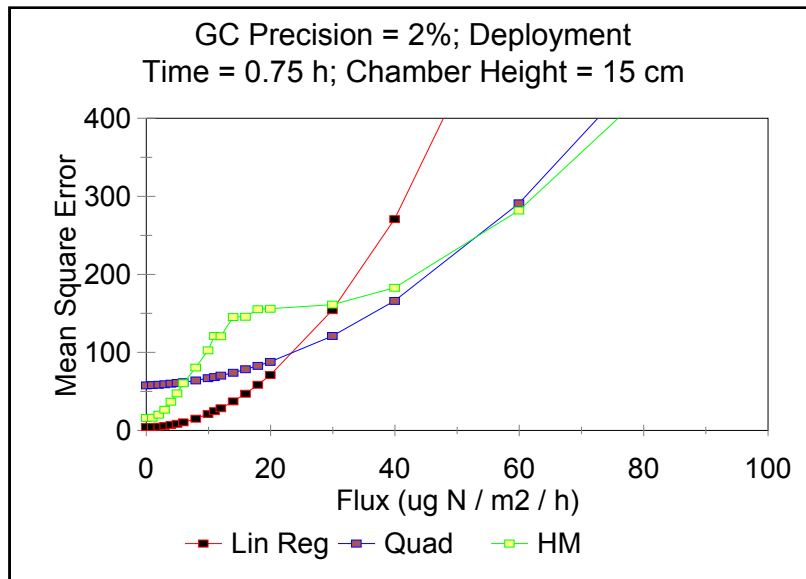

Fig. 4. Mean Square Errors associated with different calculation methods over a range of simulated N_2O fluxes. 3-point curvi-linear data were generated with the algorithm of Venterea 2009 using a Tao value of 1.0).

used to empirically quantify data curvi-linearity as a tool to aid in selection of the appropriate calculation method (Eq. 3).

$$\text{Data Curvi-linearity Index} = (C_1 - C_0)/(C_2 - C_1) \qquad \text{Eq. 3}$$

where C_0, C_1, and C_2 are headspace gas concentrations for 3 equi-spaced time points (time 0, time 1, time 2). [*Note: the constraint of equi-spaced time points is required for Eq. 3, however a more generalized form of the data curvi-linearity index can be calculated as the slope of the ½ of the time course data divided by the slope of the second ½ of the time course data.*]
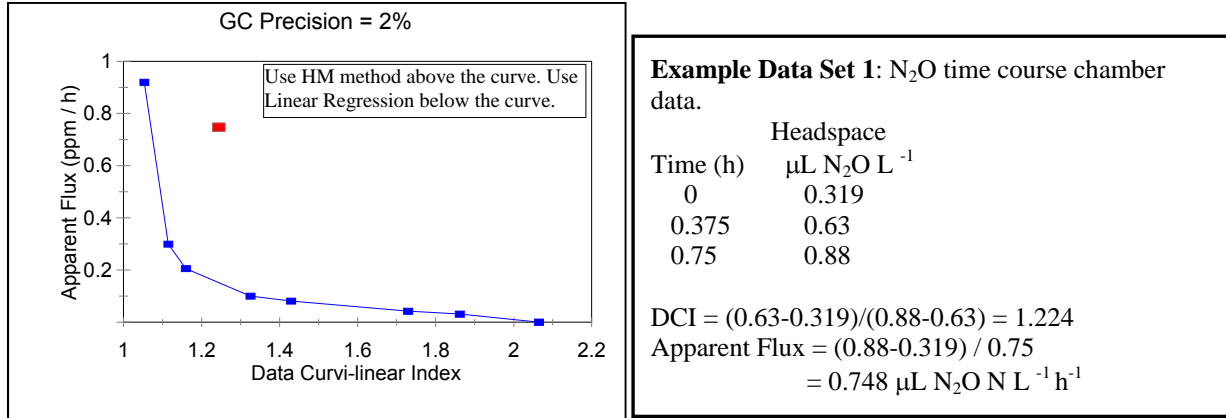

Fig. 5. Decision criteria curve for 2% precision.

Example Data Set 1: N_2O time course chamber data.

Time (h)	Headspace µL N_2O L^{-1}
0	0.319
0.375	0.63
0.75	0.88

DCI = (0.63-0.319)/(0.88-0.63) = 1.224
Apparent Flux = (0.88-0.319) / 0.75
= 0.748 µL N_2O N L^{-1} h^{-1}

Figures 5 and 6 show the decision curves for the HM method vs. linear regression as a function of the Data Curvi-linearity Index (DCI) and the apparent N_2O flux. The blue curves in the figures delineate the points where the MSE of the Hutchinson/Mosier method equals the MSE of linear regression. Above and to the right of the curves, the MSE of the HM method is less than the MSE of linear regression, while in the regions below and left of the curves MSE of linear regression is less than the MSE of the HM method. In these plots, the apparent N_2O flux was calculated as: $(C_{end}-C_0)/Td$, where C_0 and C_{end} are headspace gas concentrations (µL N_2O L^{-1}) at time 0 and the end timepoint, respectively, and Td is the total chamber deployment time (h). To illustrate how Figs. 5 and 6 can be used to determine which calculation method should be used for a given data set, two examples are provided. In example data set 1, the DCI is calculated to be 1.224, and the apparent flux is calculated to
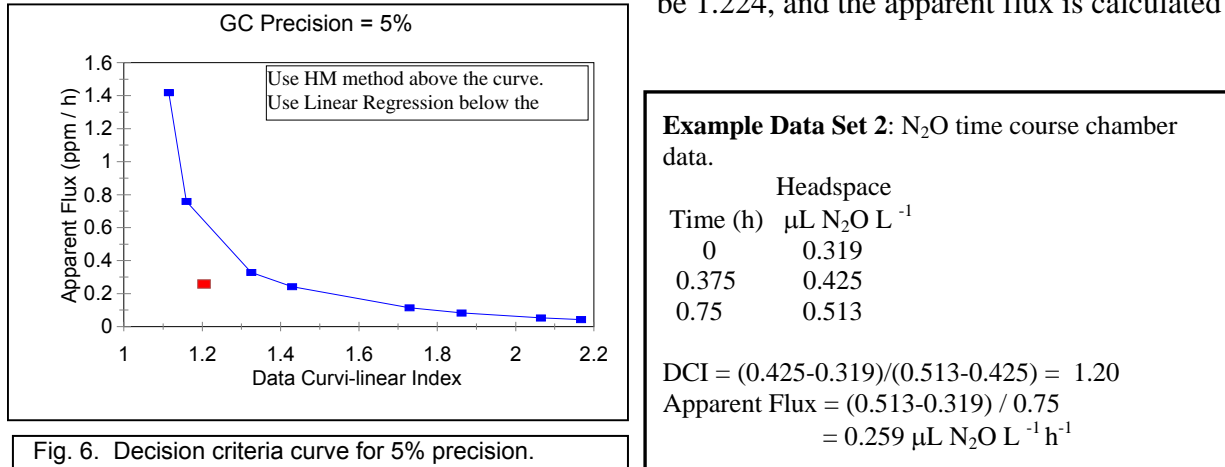

Fig. 6. Decision criteria curve for 5% precision.

Example Data Set 2: N_2O time course chamber data.

Time (h)	Headspace µL N_2O L^{-1}
0	0.319
0.375	0.425
0.75	0.513

DCI = (0.425-0.319)/(0.513-0.425) = 1.20
Apparent Flux = (0.513-0.319) / 0.75
= 0.259 µL N_2O L^{-1} h^{-1}

be 0.748 μL N_2O L^{-1}. If the GC precision is 2%, then this point (1.244, 0.748) is plotted on Fig 5. (shown as a red square). It is observed that the red point lies above the curve in Fig. 5, thus linear regression should not be used. The HM calculated flux for example 1 data is 0.923 μL N_2O L^{-1} h^{-1}. In the example data set #2, the calculated DCI is 1.20 and the apparent flux is 0.259 μL N_2O L^{-1} h^{-1}. If the GC precision happened to be 5%, then this point (1.20, 0.259) is plotted on Fig 6 (red square). Since this point falls below the curve linear regression would be the method of choice over the HM method. The decision criteria curves shown in Figs 5 and 6 are only for linear regression vs. HM method with 3 timepoint data sets. Families of decision criteria curves are currently being generated for the Quad and HM methods for 3 and 4 point rate data with GC precisions in the range of 1% to 6%

Fig. 7. Theoretical underestimation of published chamber fluxes as a function of soil volumetric water content (θ), chamber volume to surface area ratio (Hc), deployment time (Td), flux-calculation (FC) scheme, and soil physical properties. From Venterea (2010).

(Parkin and Venterea, manuscript in preparation). These curves will be added to this document when they become available. Once the precision of a given GC system is known and its associated decision criteria curve has been generated, it should be relatively straightforward to code the selection of the best flux-calculation technique into a spreadsheet-based calculation system using conditional (If/Then) statements applied to each individual set of chamber data. This will require developing an empirical functional relationship between DCI and apparent flux (Parkin and Venterea, manuscript in preparation).

Bias corrections and soil property effects: Linear regression, and to a lesser extent the HM and Quad flux models, will generate negatively biased flux estimates even if correlation of the chamber data with the models are very high (Livingston et al., 2006; Venterea et al., 2009). Thus, fluxes estimated above are still expected to underestimate the actual pre-deployment soil-to-atmosphere fluxes. Additionally, the degree of bias and the extent of data curvi-linearity will increase with increased air-filled porosity in the soil underneath the chamber. This phenomenon occurs because as trace gas accumulates in the chamber following chamber closure, trace gas also accumulates in the air-filled soil pores, and a greater proportion of the total emitted trace gas will accumulate in the soil pores as the air-filled porosity increases (Venterea and Baker, 2008). This could result in important experimental artifacts, especially when soils under study differ with respect to bulk density and/or water content (for example when evaluating effects of tillage or organic amendments) (Fig. 7). This effect can also invalidate inter-site flux comparisons. In order to deal with these issues while avoiding the complications of using the NDFE model, Venterea (2010) developed a spreadsheet-based method for correcting the bias in flux estimates made using linear regression, HM, or Quad. This method accounts for the effects of soil properties, and therefore requires information regarding soil bulk density, water content, texture, and temperature at the time of flux-measurement. Measurement or estimate of these factors will necessarily introduce additional potential errors, but researchers wishing to make such
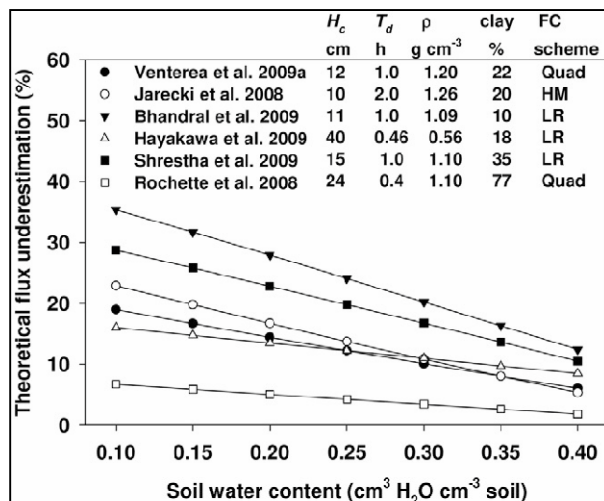
corrections and attempt to estimate "absolute" fluxes can utilize the method of Venterea (2010). Example spreadsheets can be accessed at http://www.ars.usda.gov/pandp/people/people.htm?personid=31831 or from the on-line version of the Venterea (2010) article.

Converting from Volumetric Units to Mass Units: A flux calculated from either from linear regression or a non-linear model will have units of μL trace gas m^{-2} h^{-1} (when ppm(v) is regressed against time in hours). An additional calculation must be performed in order to covert flux values from a volumetric basis to a mass basis. To perform this conversion the ideal gas law is used (Eq. 4).

$$PV = nRT \qquad\qquad \text{Eq. 4}$$

where P = pressure, V = volume, n = the number of moles of gas, R = the gas law constant, and T = temperature.

Sample Calculation*: Altitude = 1000 feet; Air temperature = 20°C*
To convert μL gas to μMol (value of 0.965 atm was obtained from Table 1 and ^{o}C was converted to ^{o}K by adding 273).

1 μL trace gas * 0.965 atm / ((0.08206 L atm Mol^{-1} ^{o}K^{-1}) * (273 + 20)^{o}K) * 1 L/10^6 μL * 10^6 $\mu Mol/Mol$ = 0.0401 μMol trace gas

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The ideal gas law quantifies the relationship between pressure, volume, mass and temperature of a gas. The ideal gas law constant (R) can be expressed in many different forms, but when R = 0.08206, the units are L atm Mol^{-1} ^{o}K , and the corresponding units of P, V, N and T are atmospheres, liters, moles, and degrees Kelvin, respectively. The goal of applying Eq. 4 is to convert μL trace gas to μMol trace gas. To do this, one must have knowledge of both the air temperature and atmospheric pressure. Table 1 shows atmospheric pressures at different elevations.   With knowledge of the temperature and altitude the ideal gas law is applied to convert μL of the trace gas to μMol of trace gas. For example, at an altitude of 1000 ft., and at an air temperature of 20°C, we calculate from Eq. 4 that 1μL of trace gas contains 0.0401

Table 1. Relationship between altitude and atmospheric pressure. Pressure is given in 3 different units.

Alt (ft)	mm Hg	psi	atm
0	29.92	14.7	1.000335
1000	28.86	14.18	0.964949
1320	28.54	14.02	0.954061
2000	27.82	13.67	0.930244
2640	27.14	13.33	0.907107
3000	26.81	13.17	0.896219
3960	25.77	12.66	0.861513
4000	25.84	12.69	0.863555
5000	24.89	12.22	0.831571
5280	24.47	12.02	0.817961
6000	23.98	11.78	0.801629
6600	23.25	11.42	0.777131
7000	23.09	11.34	0.771687
7920	22.15	10.88	0.740384
8000	22.22	10.91	0.742426
10560	20.11	9.88	0.672334

µMol of trace gas (see sample calculation box). Thus, 1 ppm (1 µL/L) trace gas contains 0.0401 µMol trace gas per L of air. If temperature is changing significantly during chamber deployment (by more than about 5° C per hr), and temperature corrections per above are not applied, this will cause errors in calculated fluxes due to expansion (temperature increase) or contraction (temperature decrease) of chamber headspace gas.

Minimum detection limit and non-significant fluxes: Often field fluxes are low, thus it is important to have an idea of the minimum detection limit (MDL). To determine the MDL we performed Monte Carlo simulations over a range of analytical precisions and chamber deployment times (Parkin and Venterea, manuscript in preparation).   Results of these studies for N_2O are presented in Tables 2 and 3 for 3-point and 4-point data sets, respectively. The limits presented in Tables 2 and 3 are for positive fluxes. The detection limits for negative fluxes can be obtained by multiplying the values in Tables 2 and 3 by -1. These "negative" MDLs will then represent the upper limit for gas consumption processes which are manifested as negative fluxes. There are several options available to handle data that falls below the MDL (or within the detection limit band). These options include: 1) report the value as "below the detection limit", 2) report the value as zero, 3) report some a value between zero and the MDL (such as ½ the MDL), or 4) report the actual measured value even if it falls below the MDL (Gilbert, 1987). **We recommend that, in reporting trace gas studies in this project, option 4 be adopted - report the measured value along with the stated MDL.**

Table 2. Minimum Detection Limits (α = 5%) for Hutchison/Mosier (H/M) procedure the Quadratic procedure (Quad) and linear regression (L.R.) for different chamber deployment times.̃ Three equi-spaced data points for each flux determination.

Analytical Precision (%CV)	Minimum Detection Limit (ppb / hour) : 3 data point rates								
	0.5 Hour Deployment			0.75 Hour Deployment			1.0 Hour Deployment		
	HM	Quad	LR	HM	Quad	LR	HM	Quad	LR
1	34.6	64.1	17.7	23.5	43.0	11.8	17.5	22.0	8.9
2	70	129	35.5	46.6	85.9	23.7	34.8	44.0	17.9
3	105	193	53.3	69.4	130	35.7	52.3	66.0	26.7
4	140	258	71.1	93.4	173	47.5	70.4	88.1	35.7
5	176	322	88.9	117	215	59.1	88.2	110	44.6
6	213	387	107	141	258	71.3	106	132	53.5

Table 3. Minimum Detection Limits (α = 5%) for Hutchison/Mosier (H/M) procedure the Quadratic procedure (Quad) and linear regression (L.R.) for different chamber deployment times. Results are for 15,000 Monte Carlo simulations at each analytical precision and deployment time. Four equi-spaced data points for each flux determination (t_0, t_1, t_2, t_3). For the H/M flux calculations the average concentration of the t_1 and t_2 data points was used.

	Minimum Detection Limit (ppb / hour) : 4 data point rates								
Analytical Precision (%CV)	0.5 Hour Deployment			0.75 Hour Deployment			1.0 Hour Deployment		
	HM	Quad	LR	HM	Quad	LR	HM	Quad	LR
1	21.2	47.0	16.9	14.1	31.1	11.3	10.6	23.3	8.48
2	42.3	93.4	33.8	28.1	61.9	22.5	21.1	46.8	16.9
3	62.7	138	50.8	42.1	92.5	33.6	31.5	69.3	25.4
4	84.4	186	67.3	55.8	123	44.9	42.2	93.4	33.7
5	105	232	84.5	69.5	154	56.2	52.8	116	42.2
6	126	278	101	83.0	184	67.4	63.6	177	50.7

Non-Detects: As was noted above, analytical and sampling error introduces variability that can result in "noisy" dat. Fig. 8 shows the 17 possible data patterns that can be obtained from 3 point sample sets. One consequence of noisy data is that the HM model will often not be applicable. The HM model will only work if the quantity $[(C_1 - C_0)/(C_2 - C_1)] > 1$ (Fig. 8, panels 2 and 3) or the quantity $[(C_1 - C_0)/(C_2 - C_1)]$ is between zero and 1 (Fig. 8, panels 8 and 9). In 13 of the 17 possible data patterns shown in Fig. 8, the HM model will fail. In cases of HM failures, the investigator can: 1) designate the flux as zero, 2) use linear regression, or 3) use an alternate method (i.e. Quad, NDFE, HRM). Until the non-linear models (NDFE and HMR) can be further evaluated, at this point in time we recommend that the investigator use linear regression (option 2) or the Quad method when the HM model fails. Often an outlier may be present, due to sampling or analytical problems (i.e. vial leakage, chamber leakage, sample mix up, or change in GC detector sensitivity, humidity or temperature perturbations). Critical judgment is required to

Fig. 8 Possible data patterns for 3- point flux measurements
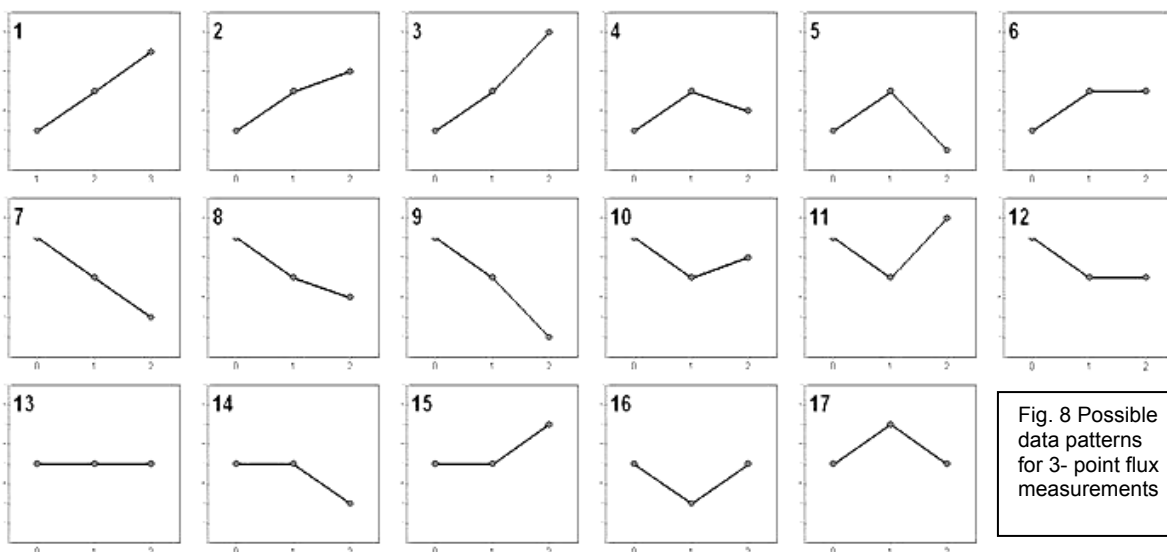
disregard outliers. If N_2O, and CO_2 analyses are performed on the same sample (N_2O with an electron capture detector and CO_2 with a thermal conductivity or methanizer + flame ionization detector or infra-red detector), then often a sampling or analytical problem can be diagnosed by comparing N_2O and CO_2 data for each timepoint. For example, if data pattern 4 or 5 (Fig. 8) is observed for both N_2O and CO_2, a likely explanation would be chamber or sample vial leakage, since, in opaque chambers, consumption of atmospheric CO_2 is not typical. Similarly, if CO_2 data patterns like those of panels 7, 8, or 9 were observed and the CO_2 concentration of last time point were near ambient, this may indicate a sample mix up (i.e. t_0 exchanged with t_2). Temperature or humidity changes during chamber deployment may produce patterns similar to those of panels 4, 5, and 6. If the investigator cannot discount outliers based on experience and judgment of past performance of the site, instrument function, or chamber efficacy, the most conservative approach would be to use linear regression on all the data. If noisy data proves to be a persistent problem, evaluation of GC precision, chamber design, septa reactivity/integrity, and sampling protocols should be performed. Also, collection of 4 (or more) gas samples during the chamber deployment will yield improved flux estimates.

Quality assurance /Quality control:
Standards and standardization: Standards should be prepared with each set of samples. Linearity of the detector's responses should not be assumed, thus a range of gas concentrations for each trace gas be run. Standard gasses should be prepared in an "air matrix" unless it has been previously determined that GC detector response is not sensitive to O_2. GC drift can occur during a run, thus it is recommended that check standards be run every 10 or 20 samples to determine, and if necessary, correct for GC drift. It is highly recommended that a NIST-certified tank containing CO_2, N_2O and CH_4 be purchased by the project and used to evaluate the standard gases used at all locations.

Ancillary Measurements
In addition to the measurements prescribed by soil sampling protocol additional measurements are recommended.
1. *At the times fluxes are measured:* 1) air temperature, 2) soil temperature (5 cm), and 3) soil water content (0-6 cm).
2. *At the time of chamber anchor installation:* 1) bulk density, 2) soil texture, 3) organic C and N., 4) pH, 5) anchor height above the soil (used to compute chamber headspace volume), 6) soil nitrate and ammonium (0-10 cm). [*Note: It is desirable that soil nitrate and ammonium be determined throughout the year at time intervals deemed appropriate by the individual investigator as dictated by resource availability and plot constraints.*]
3. *Year round:* The following metereological should be collected year round at a frequency of at least once per day: 1) precipitation, 2) air temperature, 3) relative humidity, and 4) solar radiation.

Advice and Consultation
Several USDA-ARS investigators involved in GRACEnet have experience in trace gas analysis and flux measurement. These people have agreed to serve as resource contacts for investigators with questions on GC operation, soils chambers, gas sampling, flux calculation, field variability, and data calculations and interpretation.

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Appendix I. Example of Trace gas Flux Sampling Procedure
A set of 12 Anchors placed in pairs (in-row and inter-row). For each set of 12 chambers:
1. Lay out Chambers, Vials, Syringes by each anchor
2. Install 5 cm temperature Probes (1 in each plot). Air temperature and chamber temperature probes are placed in first plot only.
3. Take ambient air gas sample
4. Start Measurement (t 0)
a. Start at plot #1
      1. Record Temperatures, Start Stop Watch
      2. Place chamber on anchor #1 (vent facing downwind)
      3. Remove 10 ml gas sample
      4. Inject sample into vial
      5. Flush syringe with Air 2x
      6. Place chamber on anchor #2
      7. Remove 10 ml gas sample
      8. Inject sample into vial
      9. Flush syringe with air 2x
b. Move to the pair of chambers in the next plot
      1. Record time on stop watch
      2. Place chamber 3 on anchor
      3. Remove 10 ml gas sample
      4. Inject into vial
      5. Flush syringe with Air 2x
      6. Place chamber 4 on anchor
      7. Remove 10 ml gas sample
      8. Inject into vial
      9. Flush syringe with air 2x
c. Move to next plot
      2. Repeat steps 4b.1 through 4b.9 (above)
d. Repeat step 4c until all 12 chambers are in place and have been sampled for time 0
5. First Time Point (t 1)
a. Move to plot #1 (chamber 1)
      1. Record Soil Temperatures, record chamber temperature and air temperature.
      2. Insert syringe into chamber septa
      3. When stopwatch shows t-1 time (e.g. 20 minutes), remove 10 ml Gas sample
      4. Inject gas sample into appropriate vial
      5. Flush syringe 2x
      6. Move to next chamber, repeat steps 5a.2 - 5a.5, above.
      7. Continue until all chambers have been sampled for time 1
5. Second and third time points (t 2 and t-3)
a. same as step 5 above.
6. Remove all chambers, Move to next set of 12 anchors. Repeat steps 1-5
7. When all plots have been done, one person collect all chambers and place in truck other person take soil moisture readings in each plot (4 measurements/plot).

Appendix II: Potential Vendors for Supplies*

Sample Vials and Stoppers:
Option1. Exetainer vials, screw cap 12 ml vials that have a butyl rubber septa-same idea as the serum vials and butyl rubber stoppers-just cheaper and more or less disposable-can buy new screw caps and septa relatively cheaply. Exectainer vials are purchased through Labco Limited (Brow Works, Copyground Land, High Wycombe, Buckinghamshire. HP123HE, United Kingdon (phone 44-1494-459741) (fax: 44-1494-465101) (Email: sales@labco.co.uk or enquiries@labco.co.uk).
Option 2. Glass serum vials 6.0 ml (22 x 38 mm) Alltech, 2051 Waukegan Rd, Deerfield, IL 60015 (vial stock # 98768)
Butyl rubbber stoppers; 20 mm round bottom (part # 73828A-RB).   *Aluminum crimps* (20 mm); (part # CTO20NAT), Voigt Global Distribution, Inc. P.O. Box 1130, Lawrence, KS 66044. 877-484-3552. www.vial-seals.com

Standard Gases: Scott Specialty Gases: Phone: 877-715-8651.
http://www.scottecatalog.com/scottgas.nsf/web/appsnatgas

Disposable syringes and needles: Beckton-Dickenson. http://www.bd.com/hypodermic/

Syringe stopcocks: www.coleparmer.com

Reflective Mylar Tape or insulation: www.uline.com, www.reflectix.com

Gas Manifolds: Small Parts, Inc. 800-220-4242, www.smallparts.com . An example is part no. B000P7KZ9Y (description = Stainless steel hypodermic tubing manifold, inlet - 13 Gauge, 6 outlets - 20 Gauge).

*Reference to a trade or company name is for specific information only and does not imply approval or recommendation of the company or product by the U.S. Department of Agriculture (USDA) to the exclusion of others that may be suitable.

Appendix III. Examples of some chambers.
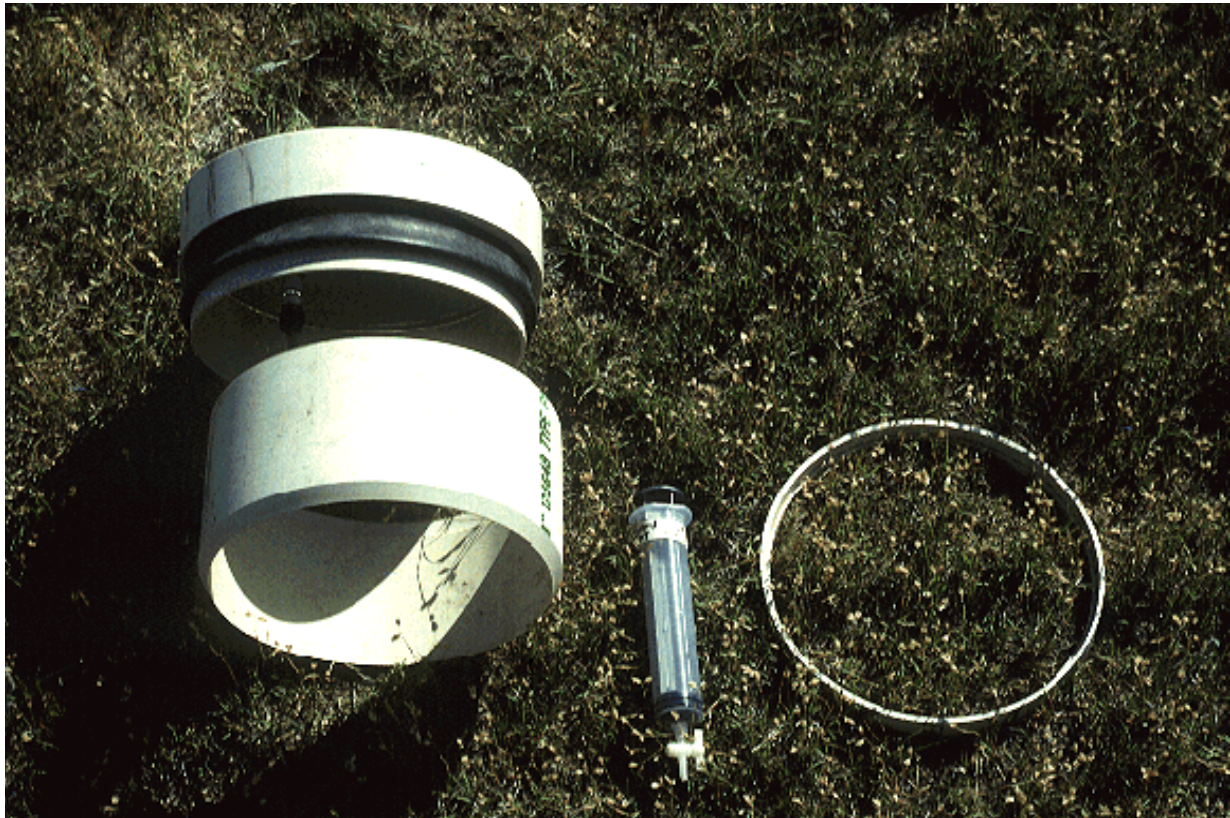
PVC soil anchor and chamber used by Hutchinson and Mosier.

Rectangular Chambers used at Ft. Collins, CO Location.

1Gas sample collection from chambers at Ames, IA location. Thermometers are measuring air and chamber headspace temperatures.

Example of temporary/portable chamber used by Parkin et al, (2005). Chamber has an attached polethylene skirt held in place on the soil surface with a length of chain. As shown, the chamber is monitoring soil CO_2 flux by recirculating gas through an infrared analyzer. Gas samples can be withdrawn through septum in top of chamber for N_2O and CH4 analyses.

Stainless steel chamber tops (above) and anchors (below) used in St. Paul, MN. Chamber anchors are nearly flush with soil surface. See Appendix IV for construction details. Photos on following page shows inside of chamber top and sample collection method.
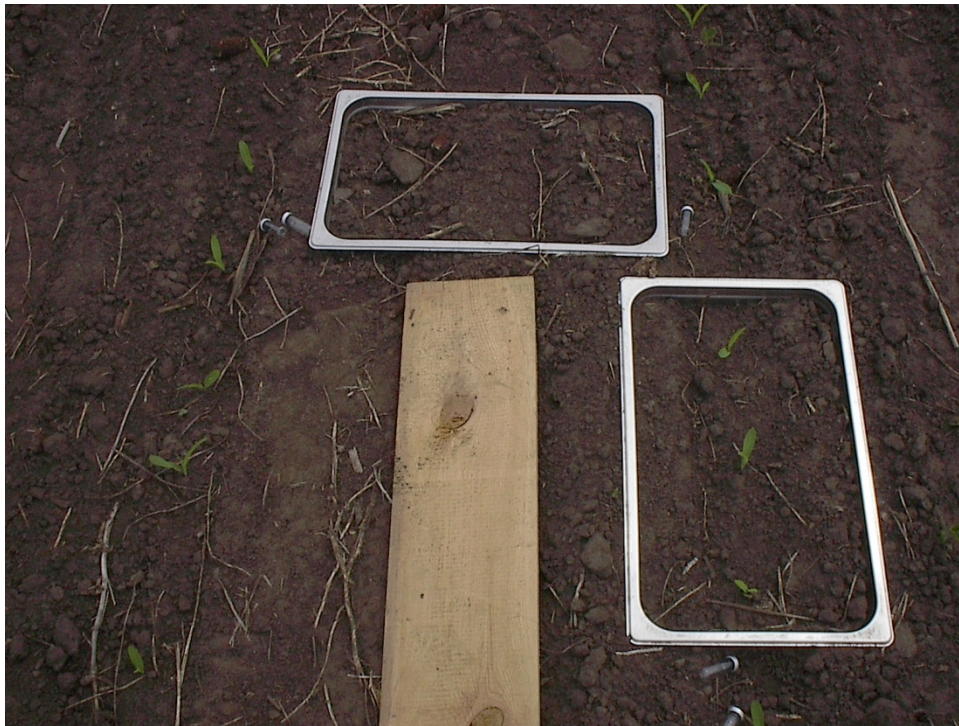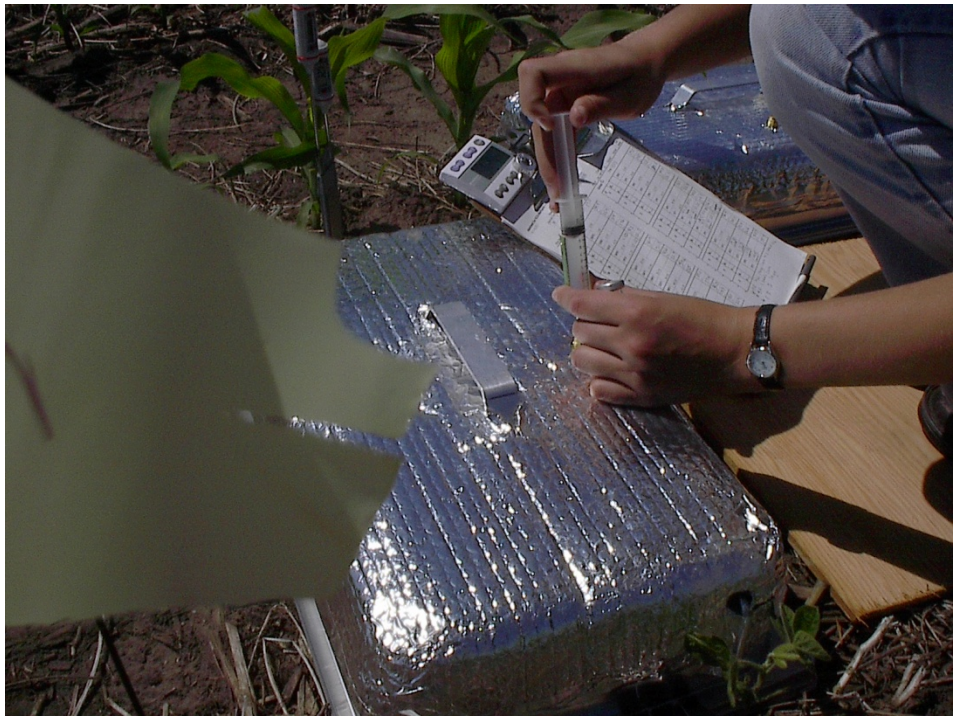
Appendix IV. A. Example of circular chamber construction used at the Ames, IA location.

CHAMBER CONSTRUCTION

Cut a 10 cm length of PVC pipe for the chamber top and a 15 cm length for the ring that will serve as the chamber anchor. Using a router with a 45 degree bevel chamfer bit, make a reasonably sharp edge on the anchor PVC ring. This will make it easier to insert into the soil.

The 10 cm long PVC ring will be used for the lid. Make a threaded 7/16" hole about 1" in from the edge of the ring. This side will be the top of the lid.

Trace the outside of the ring onto the PVC sheet (1/4 " thick) and cut out this circle. Apply PVC primer to the outside of this circle and to the thin edge of the ring. When dry, apply the cement to those same areas and attach the PVC circle to the top of the lid. Weigh it down with something to get a good seal.

After the cement has set, drill a ½" hole in the PVC circle approximately halfway between the center of the circle and the outside edge. The 20 mm butyl rubber stopper will go in here.

Attach a 15 cm piece of SS tubing to the straight union fitting and screw the fitting into the threaded hole in the ring. This is the chamber vent and will be inside the lid.

Cut an approximate 7 cm wide strip of the tire tube(make sure this is cut so as to get one continuous piece). Put this around the bottom of the lid. It will fit very snugly. Half of the tube strip will be on the lid and half will be hanging off the bottom. Now tape the rubber strip, which is on the lid, to the outside of the lid using the duct tape.

Put overlapping strips of reflective mylar tape on the chamber, top and side, so that it is nearly totally covered on the outside. Fold over the rubber strip so that the edge of the ring is showing. On this edge of the ring, the weather strip will go.

Place a thin layer of contact cement on the edge of the chamber. When the cement becomes tacky, apply the weather strip.

Place the butyl rubber stopper in the chamber tip and secure with duct tape.

Photographs of construction details are shown below.

MATERIALS LIST

PVC pipe, 12" diameter, schedule 40
Straight union fittings, ¼" PP
Tractor tire tube, 15.5R38

PVC sheet, grey, ¼" thick, grade 1 type 1
Metalized Mylar Film tape, 2" width, silver
PVC Purple Primer
PVC cement
Rubber Weatherseal, 3/8" wide x ¼" thick, "D" profile
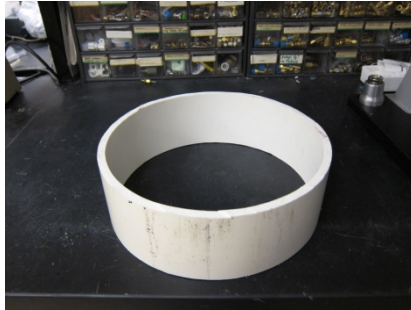Stainless Steel Tubing, ¼"
20mm Butyl Rubber Stoppers
Duct Tape
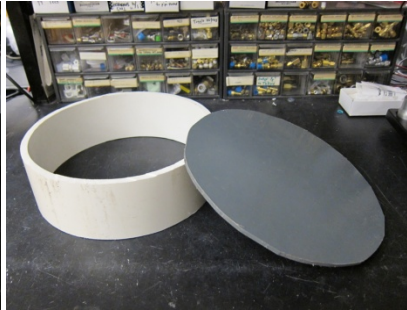
Fig. 1. PVC ring 30.3 cm inner diameter, 10 cm long.

Fig. 2. Circle cut from ¼" thick PVC sheet.
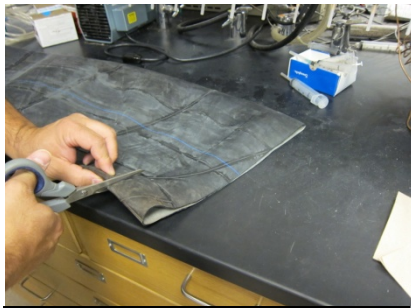

Fig. 3. PVC circle glued to PVC ring.

Fig. 4. Strip cut from truck tire inner tube (7 cm wide).
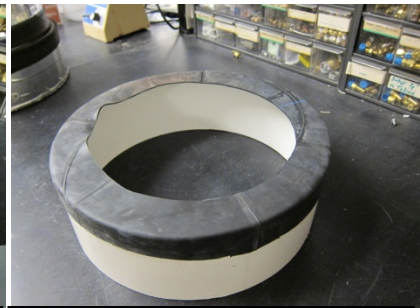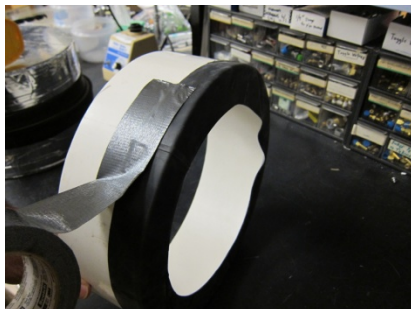

Fig. 5. Inner tube strip placed on PVC ring.

Fig. 6. Inner tube strip on PVC ring.

Fig. 7. Inner tube strip is taped into place.
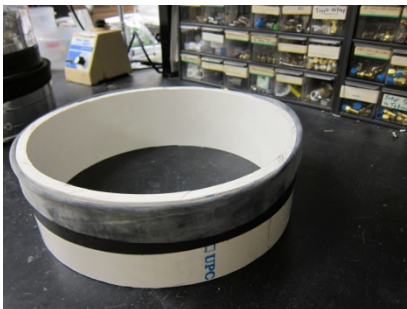
Fig. 8. Inner tube strip folded back onto PVC ring.

Fig. 9. Thin layer of contact cement applied to PVC edge.
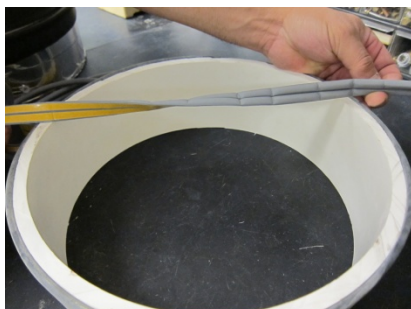
Fig. 10. Weather strip to be applied to PVC edge.

Fig. 11. Weather strip is first separated to single strand.
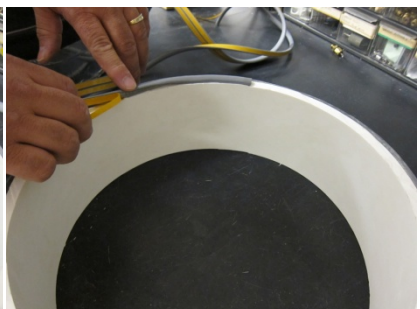
Fig. 12. Weather strip being applied to edge of PVC ring.

Fig. 13. Edges of weather strip are sealed with glue.

Fig. 14. Hole is drilled in side of chamber top.

Fig. 15. Hole is tapped to accept vent tube fitting.

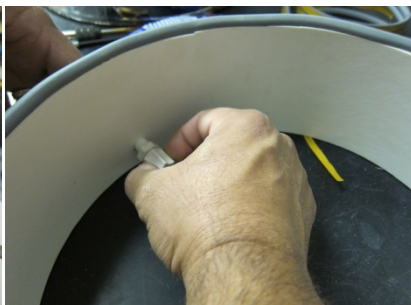Fig. 16. Plastic union serves as vent tube fitting.

Fig. 17. Plastic union screwed into hole in chamber top.

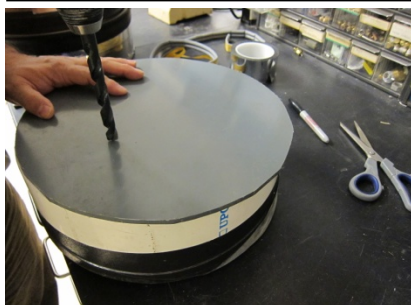Fig. 18. Stainless steel (1/4") is attached to plastic union.

Fig. 19. Hole drilled in top for sampling septa.

Fig. 20. Reflective mylar tape applied to chamber side.

Fig. 21. Reflective mylar tape applied to chamber top.

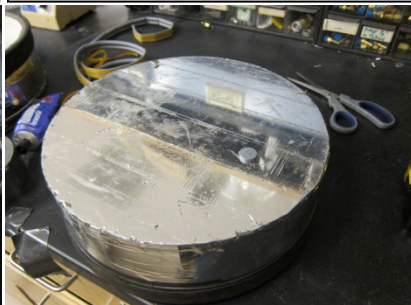Fig. 22. Butyl rubber septa placed in chamber top hole.

Fig. 22. Butyl rubber septa in top.
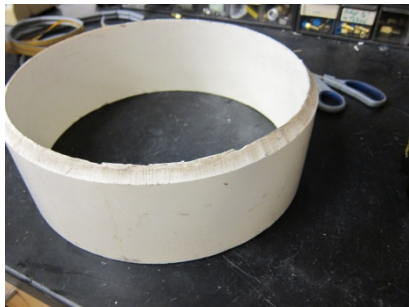

Fig. 23. Septa held in place with tape.

Fig. 24. PVC anchor ring (15 cm), one edge beveled.

Fig. 25. Chamber is being placed on anchor ring.

Fig. 25. Chamber on anchor – inner tube is folded up.

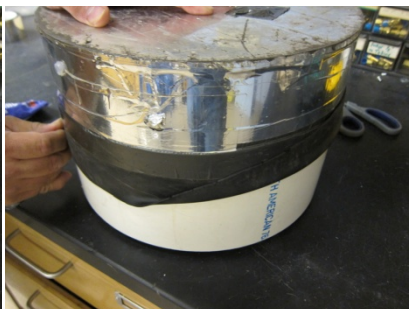Fig. 26. Inner tube being folded down onto anchor.

Fig. 27. Inner tube being folded onto anchor.
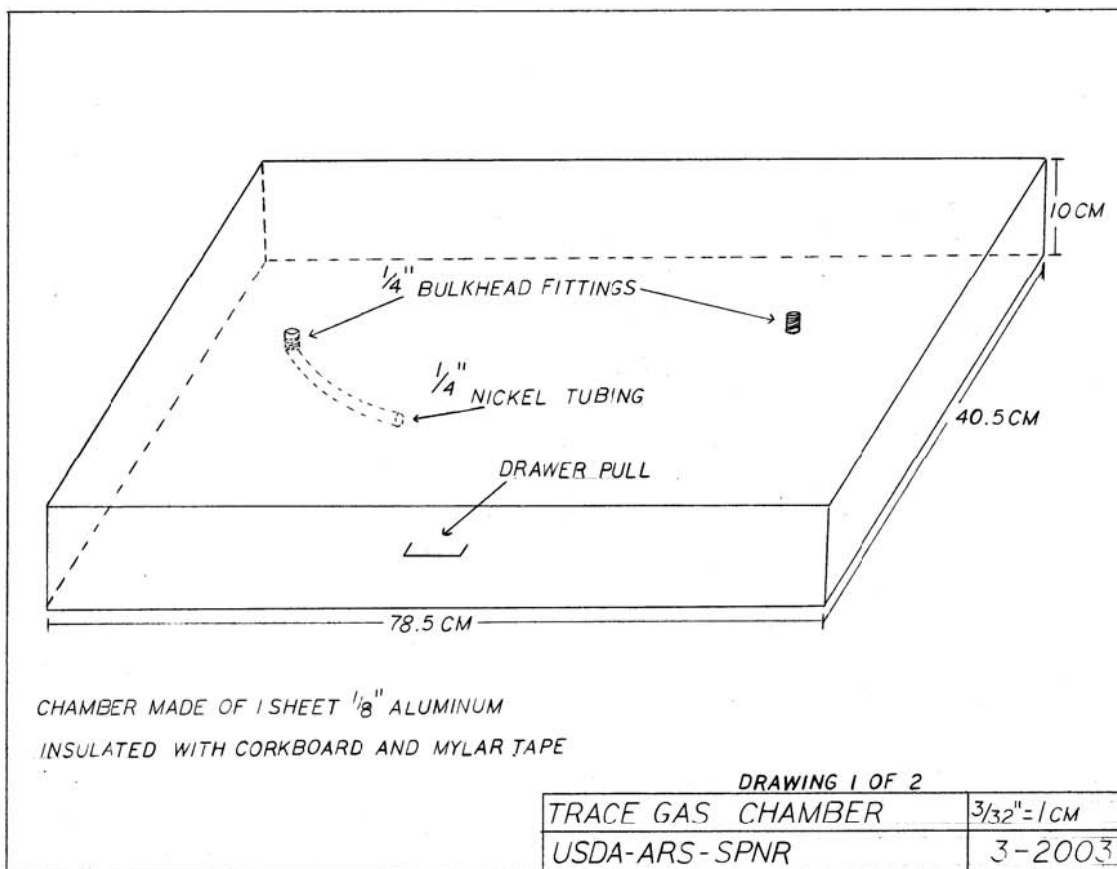

Fig. 27. Chamber in place on anchor.

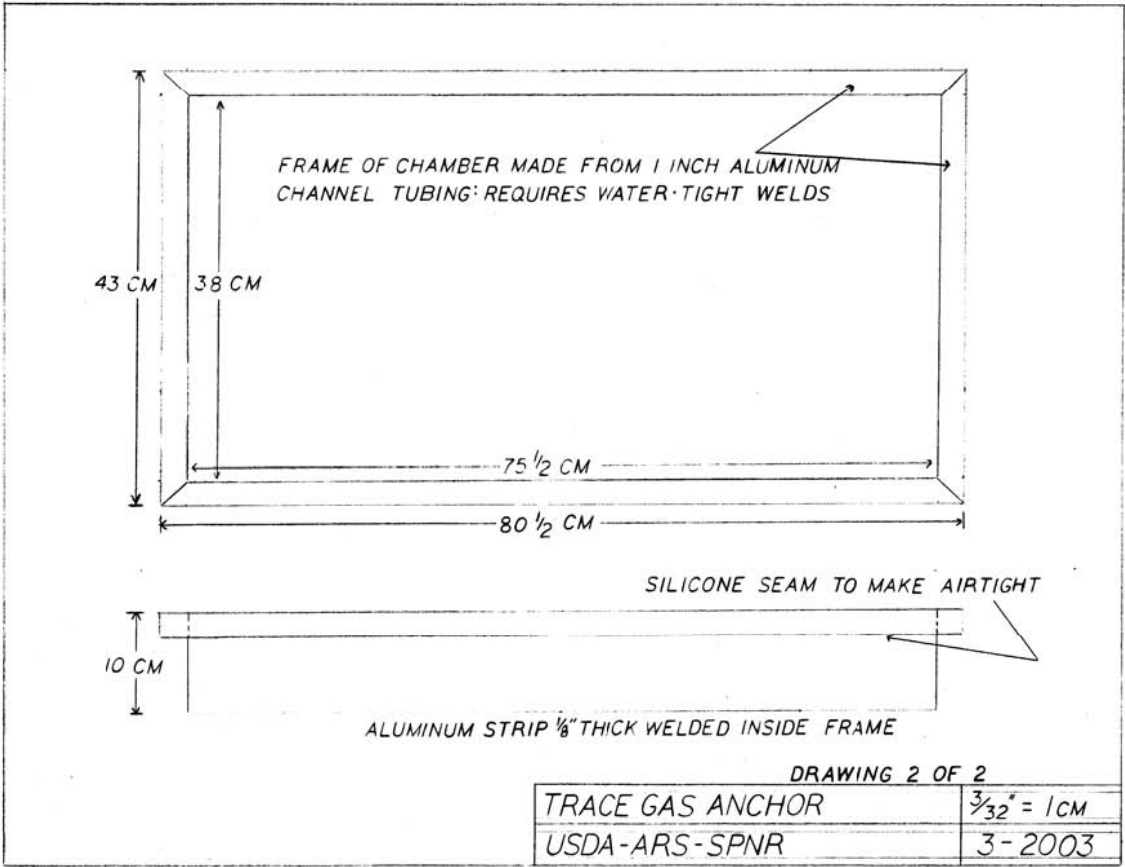Fig. 28. Chamber should have tight fit to anchor.

B. Schematics for rectangular chamber construction used at the Ft. Collins, CO Location.

Rectangular aluminum Chambers: Made from sheet aluminum. These can be made any size to fit the field situation.

Anchors: Made from sheet aluminum with a trough to hold water that has been welded on top. The anchors are inserted 10 cm into the soil.

Chamber: Made from sheet aluminum to desired dimensions. Two holes, to accommodate Swagelock fittings for vent tube and gas collection septum are drilled and tapped in each chamber top.

10 CM

1/4" BULKHEAD FITTINGS

1/4" NICKEL TUBING

40.5 CM

DRAWER PULL

78.5 CM

CHAMBER MADE OF 1 SHEET 1/8" ALUMINUM
INSULATED WITH CORKBOARD AND MYLAR TAPE

DRAWING 1 OF 2

TRACE GAS CHAMBER	3/32"=1 CM
USDA-ARS-SPNR	3-2003

FRAME OF CHAMBER MADE FROM 1 INCH ALUMINUM
CHANNEL TUBING: REQUIRES WATER·TIGHT WELDS

43 CM   38 CM

75 1/2 CM

80 1/2 CM

SILICONE SEAM TO MAKE AIRTIGHT

10 CM

ALUMINUM STRIP 1/8" THICK WELDED INSIDE FRAME

DRAWING 2 OF 2

| TRACE GAS ANCHOR | 3/32" = 1 CM |
| USDA-ARS-SPNR | 3-2003 |

Description of construction for stainless steel low-profile chambers used at St. Paul, MN ARS location (see photos in Appendix III)

Chamber anchors and tops are fabricated using 20-gauge rectangular stainless steel "steam pans" equipped with a flange around the edges (Superior Products, St. Paul, MN). Anchors are made by cutting out the bottom section of the pan resulting in a frame measuring 0.50 m X 0.29 m X 0.086 m deep, which is inserted into the soil so that the flange is nearly flush with the soil surface. Chamber tops (0.50 m X 0.29 m X 0.102 m high) are further fabricated by attaching weather-stripping material (EPDM) to the flange to serve as a gasket, covering the outer surfaces with reflective insulation (Reflectix, Markleville, IN), and installing a vent tube (3.5 mm ID X 0.15 m long) horizontally on one side and a septum-lined sampling port in the top. The sampling port is connected on the inside of the chamber to a manifold (Part no. STCM-13-20/4, Small Parts, Inc., Miramar, FL) which in turn was connected to 4 sections of FEP tubing (0.8-mm ID X 0.2 m long) (Cole Parmer) with one section of tubing secured in each quadrant of the chamber. Additional details including additional photographs and links to material suppliers can be found on-line at:

http://www.ars.usda.gov/pandp/docs.htm?docid=19008