

AN INEXPENSIVE LABORATORY AND FIELD CHAMBER FOR MANURE VOLATILE GAS FLUX ANALYSIS

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ABSTRACT. *Understanding the interactions between the environment and emissions from livestock manure is essential in developing management practices intended to minimize negative environmental consequences. However, protocols and equipment necessary to investigate these interactions at the laboratory or field-scale are cumbersome and can be expensive. An inexpensive dynamic flux chamber (cost < \$400 per unit) was designed to measure gaseous emissions from cattle manure in laboratory and field experiments. The hemispherical stainless steel chamber (emission surface = 0.08 m²) was constructed with an internal gas mixing fan. A port was attached to the chamber top, which facilitated the collection of headspace gas samples for greenhouse gases and volatile organic compounds (VOC) by solid-phase microextraction (SPME). The chamber was tested to evaluate flow characteristics and was found to perform very similarly to a continuous-flow stirred reactor. As such, concentrations measured at the sampling port were indicative of concentrations anywhere in the headspace. In laboratory and field applications, the inexpensive dynamic flux chamber was easy to use and required little operator input to quickly obtain multiple samples to measure the relative emissions of greenhouse gases, ammonia (NH₃), and VOC from multiple sites in cattle feedlot pens.*

Keywords. *Beef cattle manure, Dynamic flux chamber, Feedlot emissions, Feedlot surface, Odor, VOC.*

Many different gases such as NH₃, greenhouse gases, and VOC associated with malodor (volatile fatty acids, aromatics, sulfides, amides, and alcohols) are emitted from beef cattle manure, and managing these emissions has become an important issue for feedlot operators (Hutchinson et al., 1982; Baek et al., 2003; Duysen et al., 2003; Gay et al., 2003; McGinn et al., 2003; Koelsch et al., 2004). Direct measurements of gaseous emissions from feedlot surfaces provides essential information for the development of accurate dispersion models for predicting plume movement, which is useful for determining proper setback distances on new or expansion of existing operations (Nangia et al., 2001). Typical air dispersion models rely on input data such as meteorological, topographical and terrain, emission rates, and contributing area to predict plume movement and intensity. Meteorological data can be collected using local or on-site stations, whereas topographical and terrain data can be collected from digitized maps. Currently, considerable research effort has been spent refining emission measurements, which are primarily collected using

wind tunnel equipment (Meisinger et al., 2001; Duysen et al., 2003). Typical costs associated with building wind tunnels exceed \$2500 per unit. Furthermore, considerable expertise and operator input is needed to position and collect multiple measurements at field sites.

Emission rates and areas of emission within the feedlot are likely dependent on spatial, temporal, biological, and management variables. Managing feedlot pen surfaces for odor production, nitrogen transformations, greenhouse gas emissions, and dust potentials has been an active area of research (Woodbury et al., 2001; Razote et al., 2004; Miller and Berry, 2005; Parker et al., 2005). Similarly, research utilizing electromagnetic induction, GPS, and geostatistical imaging techniques for monitoring soil-crop dynamics (Eigenberg et al., 2002; Eigenberg and Nienaber, 2003) shows great potential for identifying zones within feedlot pens that may be contributing to gaseous emissions.

Understanding the complex interaction of environment and management on emissions is essential for predicting emission transport and fate. Robust techniques and inexpensive, versatile equipment are needed to link laboratory studies to field-scale verification. Unfortunately, the techniques and equipment necessary to investigate laboratory and field-scale situations are cumbersome or are prohibitively expensive. The objectives of this study were to design an easy to use, low-cost flux chamber for determining relative flux differences, evaluate this chamber for flow characteristics, and test the suitability of the chamber for laboratory and field studies.

MATERIALS AND METHODS

DYNAMIC FLUX CHAMBER DESIGN

Table 1 lists the parts and vendors used for construction of the headspace chamber system. A semi-hemispherical

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Mention of trade names or commercial products in this article is solely for the purpose of providing specific information and does not imply recommendation or endorsement by the USDA.

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Table 1. Parts list and vendors for dynamic flux chamber (approximate purchase date Jan. 2004).

Item	Vendor	Part Number	Qty.	Total Cost (\$)
SS chamber	Wal-Mart	--	1	5.00
Air pump	SKC, Inc.	222-2301	1	130.00
Midget bubbler	Supelco	64834-U	1	50.00
12V battery	Batteries.com	272388	1	25.00
Circulating fan	Newark	91F7443	1	10.00
Bulkhead elbows	Parker Inst.	4-4C5BZ-SS	4	60.00
Union tee	Parker Inst.	4-4-4JBZ-SS	3	45.00
Hose to pipe adapter	Parker Inst.	4-4B2HF-SS	1	5.00
		4-4FH2B2-S		
Bulkhead male	Parker Inst.	S	1	10.00
Male connector	Parker Inst.	4-4FBZ-SS	1	7.00
1/4 SS tee	--	--	1	3.00
Misc. elec. connectors	Newark	--	--	15.00
Misc. tubes	--	--	--	15.00
Total:				\$380.00

stainless steel (SS) vessel (7.6 L volume, 0.32 m diameter, 0.08 m² exposed surface area) was used as the dynamic flux chamber (fig. 1A). A port was fixed at the chamber top using a 6.5 mm national pipe thread (NPT) SS bulkhead fitting. The bulkhead fitting was sealed with an O-ring, and the stem protruded into the chamber headspace approximately 25 mm. The external connector was attached to a 6.5 mm NPT SS tee (fig. 1B). One end of the tee was fitted with septa to allow insertion of an SPME portable field sampler (Supelco, Bellefonte, Pa.) for collection of VOC or a syringe needle for collecting permanent gases, such as O₂, CO₂, CH₄, and N₂O (fig. 1E). The assembly was oriented such that an SPME fiber would pass through the tee toward the chamber to minimize breakage during insertion. The second connector of the tee was fitted with a 6.5 mm hose barb fitting (fig. 1B). The hose barb was attached to a 0.25 m long, 6.5 mm diameter, polyvinyl chloride (PVC) tube, which was connected to a midget bubbler (fig. 1C). The bubbler was contained inside a 23 × 85 mm glass vial filled with 12 mL of a 10 mM H₃PO₄ ammonia trap solution. The vacuum side of the bubbler was connected to the inlet port of a battery-operated air pump. A nylon membrane syringe filter with a 0.45 μm pore diameter was placed in-line to limit inadvertent pump contamination with acid trap solution (fig. 1D). The pump (fig. 1E) discharge port was connected to two 6.5 mm diameter compression fitting tees using two 0.25 m, 6.5 mm diameter, rigid-wall vinyl tubes. Four 0.10 m long, 6.5 mm diameter, rigid-wall vinyl tubes distributed the discharge to four 6.5 mm, 90° elbow bulkhead compression fittings. These fittings segmented the circular chamber into quadrants (fig. 1F). The elbow fittings were placed such that the air inlet was approximately 50 mm above the feedlot/sample surface (fig. 1F). Inside the headspace of the chamber was a 40 mm, 12 V axial-flow fan moving approximately 130 L min⁻¹ (fig. 1G). The fan was suspended in the center of the headspace approximately 70 mm above the feedlot/sample surface. Fan airflow direction was from surface up to the chamber top. Fan power was supplied through a bulkhead connector located at the chamber top (fig. 1H).

Three possible airflow configurations can be utilized with the chamber system: recirculating, static, or flow-through. For our field application, the static configuration was used to compare CO₂ emissions, whereas the recirculating configura-

tion was utilized to compare ammonia and VOC emissions from a feedlot pen surface. In the static configuration, the air pumps were turned off and the emitted gases were allowed to build up in the chamber over time. At 5 min increments, headspace samples were collected to measure the change in CO₂ content with time.

The recirculating configuration using an ammonia acid trap involved drawing headspace gas through the top exit port (fig. 1B) to the inlet of the midget bubbler (fig. 1C). The air was bubbled through a known volume of acid for a specified time to remove ammonia, and the ammonia-free air exited the bubbler to the inlet of the battery-operated air pump (fig. 1C). The air was then pumped to a tee fitting (fig. 1E) that distributed the airflow to four inlet ports located at the bottom of the chamber (fig. 1F). Since the volume of air removed was returned to the chamber, there was minimal pressure gradient from outside the chamber to inside. Removal efficiency of the ammonia trap was checked by connecting three bubblers in series and passing a known mass of ammonia through the series of bubblers. The concentration of ammonium present in each acid trap was determined, and it was found that the first trap removed >98% of the ammonia. Therefore, it was determined the air being returned was effectively ammonia-free after passing through one trap. Relative flux rate was determined by measuring the mass of ammonia collected and normalizing it to the surface area of the chamber and the collection time.

The flow-through configuration was utilized for laboratory studies investigating ammonia and VOC emissions from animal manures and standard solutions. The airflow path for the flow-through configuration was similar to the recirculating configuration in that chamber air was drawn through the exit port to the midget bubbler, but the air then exited the bubbler to the inlet port of the air pump, which discharged the air to the atmosphere. Return air to the chamber was supplied by a compressed air tank to the tee (fig. 1E), which distributed the pure air to the four inlet ports. The inlet airflow was balanced to equal the exit airflow.

The chamber was portable enough to be moved between sample locations so that two people could collect up to 40 sites within a pen in approximately 3 h at a 30 min collection time for each location. To effectively sample a feedlot pen, the animals were restricted from the sampling area for less than 3 h, which reduced stress on the animals. The large number of points within a pen somewhat compensated for the relatively small surface area of the chamber and provided an adequate number of data points for further geostatistical analysis, such as cokriging with electromagnetic induction mapping data (Woodbury et al., 2005, Eigenberg et al., 2005).

METHANE TRACER STUDIES

Four tracer studies were conducted to test the flow properties of the dynamic flux chamber. The chamber was placed on a flat plate so that the total headspace volume tested was 7.6 L. The chamber was operated in a flow-through configuration with outside air entering the chamber through the side ports, mixing with headspace gases, and then leaving the chamber through the top exit port. Flow rates through the exit port for the four tracer studies were set at 1.18, 1.15, 1.14, and 1.14 L min⁻¹, respectively. A methane pulse of 50 cm³ was injected through an inlet port. Total injected methane mass was adjusted for atmospheric pressure at the time of the

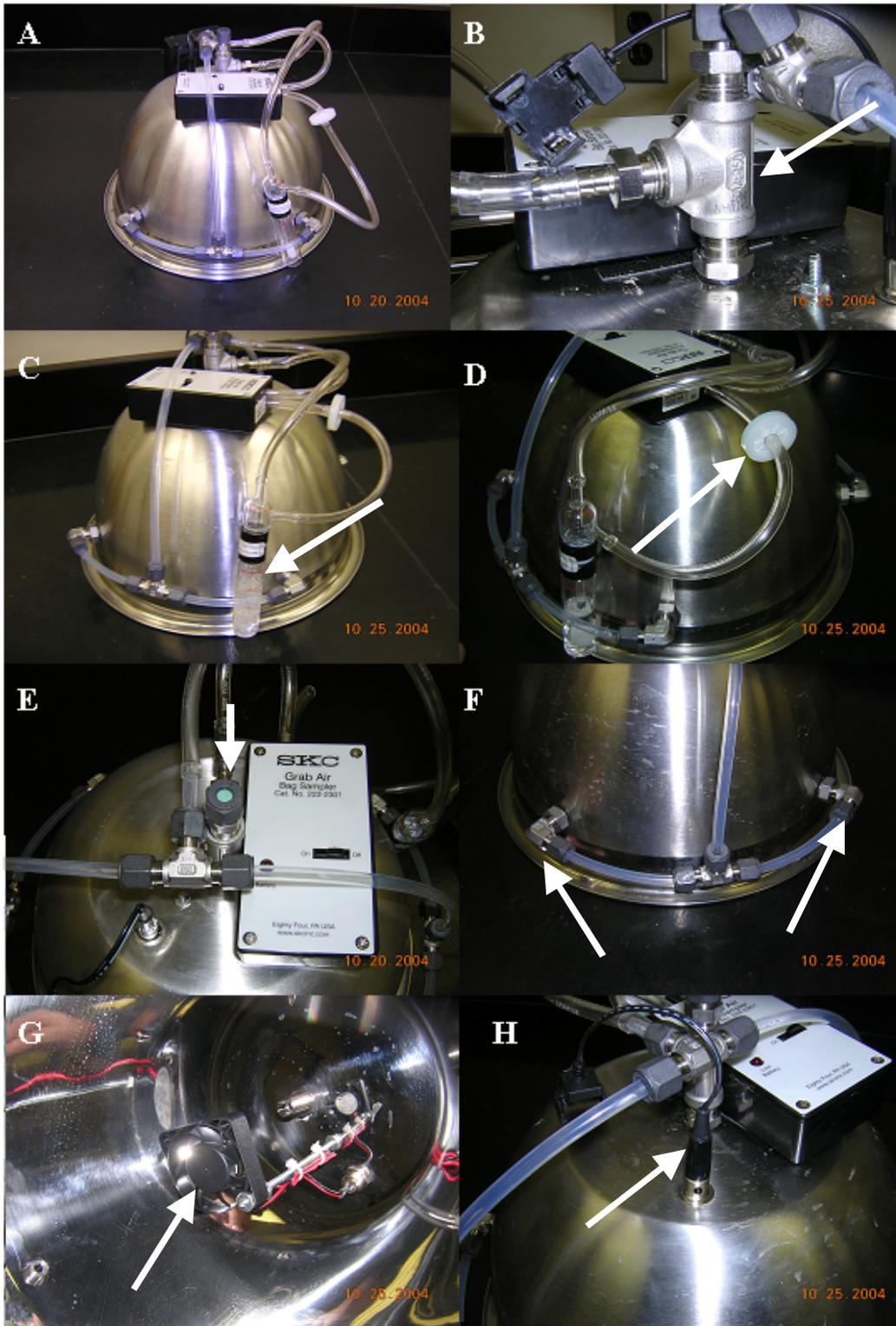


Figure 1. Flux chamber setup for ammonia analysis: (A) general setup of the chamber, (B) exit port tee fitting, (C) ammonia trap, (D) filter to prevent acid contamination of the air pump, (E) septa port, (F) inlet ports, (G) internal mixing fan, and (H) bulkhead power connector for the internal mixing fan.

test. Several air samples were taken by syringe through the chamber septa port. Sampling intervals were variable (0 to 40 min), with short intervals at the beginning and longer intervals at the end of the tracer study. Total sampling times were sufficient to exchange the chamber volume six or more times. Several initial samples were taken before the pulse injection to

establish background methane concentrations. Methane concentrations were determined relative to standard mixes (Scotty Specialty Gases, Plumsteadville, Pa.) using an SRI 8610C gas chromatograph (SRI Instruments, Torrance, Cal.) equipped with helium ionization and thermal conductivity detectors, as previously described (Miller and Berry, 2005).

FIELD APPLICATION

Four chambers were constructed and tested at the experimental cattle feedlot at the USDA-ARS U.S. Meat Animal Research Center in south-central Nebraska. Chambers were placed on the feedlot pen surface along a transect leading from the edge of the pen to the top of the central mound, running parallel to the feed bunk. Chambers were operated in recirculating configuration (1 L min^{-1}) for 20 min, which recirculated the headspace gases through an acid trap, through the filter, into the pump, and then back into the chamber in order to trap NH_3 . At the same time, a pre-conditioned SPME fiber was exposed to the airstream at the SS tee (fig. 1B) for a 20 min exposure in order to sample VOCs emitted from the feedlot surface.

After collecting the NH_3 and VOC samples, the pump was turned off and the chamber was operated in static configuration, where headspace gases were mixed by the fan and the pump did not move headspace gases through the acid trap. The headspace gases were sampled at 5 min intervals for 10 min. Ammonia content of the liquid in the acid trap was measured using a Technicon (Tarrytown, N.Y.) autoanalyzer by a modification of the indophenol blue method (Bundy and Meisinger, 1994). The VOCs sorbed to the SPME fiber were measured by gas chromatography-mass spectroscopy and compared based on the total peak area (ion current) in the chromatogram. Briefly, the exposed SPME fiber was inserted into the inlet (300°C) of an Agilent 6890 gas chromatograph (Agilent Technologies, Palo Alto, Cal.), and the desorbed VOCs were separated on a polyethylene glycol column ($30 \text{ m} \times 0.25 \text{ mm}$ with a $0.25 \mu\text{m}$ film) using a temperature ramp program (flow = 2 mL min^{-1} ; initial temp. = 50° for 3 min, first temp. ramp = $30^\circ\text{C min}^{-1}$ to 170°C , second temp. ramp = $20^\circ\text{C min}^{-1}$ to 240°C , hold 240°C for 1.5 min). Compounds were detected by electron ionization (EM voltage = 1388 V , MS quad temp. = 150°C , MS source temp. = 230°C , scan mode of 30 to 550 amu) with an Agilent 5973 mass spectrometer and identified using the NIST 1998 reference library. The CO_2 content of the air samples was measured by the gas chromatograph system described to measure CH_4 . Flux rates for CO_2 were calculated based on chamber footprint, headspace volume, and sampling time, whereas flux rates for NH_3 were calculated based on chamber footprint, sampling time, and mass of NH_3 in the acid trap.

RESULTS AND DISCUSSION

Tracer study pulse concentration curves (BTC) are illustrated in figure 2a. The first sample was taken 5 s after injection. The abrupt rise and subsequent decrease in methane headspace concentration indicates that the pulse was evenly distributed during this first interval. Methane concentrations decreased abruptly, and then asymptotically approached background concentrations by 1000 s. Cumulative mass percentages of methane collected for each tracer study are illustrated in figure 2b. Calculated percentages of methane collection were determined by summing the area under the methane concentration curve defined by the individual sampling time interval. Calculated cumulative recovery percentages for the tracer studies ranged from 93.0% to 103.8% (table 2) and averaged 98.8% recovery, which did not differ from the expected 100% recovery ($P = 0.65$). Percent recovery, however, was very dependant on the

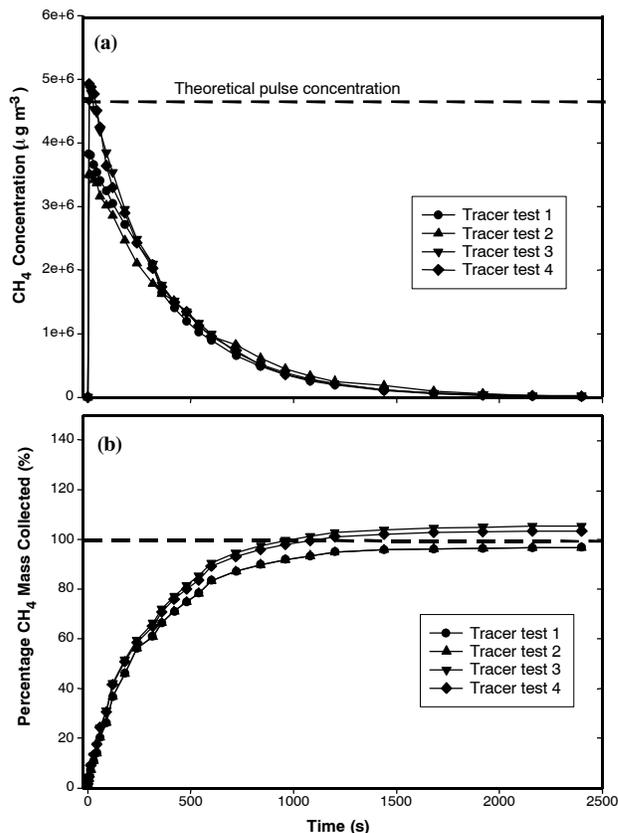


Figure 2. (a) Tracer study pulse concentration curves and (b) cumulative mass collected curves.

ability to precisely measure the initial pulse mass entering the chamber, which likely explains the variation observed between tests.

Measured pulse concentration curves of an ideal continuous-flow stirred reactor are typified by a steep rise in concentration following injection, and then a rapid decrease in concentration. Following the rapid decrease phase, the concentration asymptotically approaches the background level. Deviation from the ideal reactor may be explained by injection of the tracer in only one inlet port, and not at the chamber center. During the initial 5 s of mixing time, some of the tracer may have preferentially been removed from the chamber, affecting peak concentrations and recovery. However, this process appears to be negligible. Using physical measurements of the dynamic flux chamber and pulse test data, values were determined for the calculated retention time (RT_C) and the experimentally determined retention time (RT_E) (table 2). Although RT_C values were slightly higher on average, a two-sample paired t-test demonstrated that RT_C did not differ from RT_E ($P = 0.38$).

Table 2. Calculated (RT_C) and experimentally (RT_E) determined retention times and pulse recovery percentages for each tracer test.

Measured Flow (L min^{-1})	RT_C ^[a] (s)	RT_E (s)	Pulse Recovery (%)
1.18	386	390	96.4
1.15	396	395	93.0
1.14	400	394	103.8
1.14	400	392	101.8

[a] $RT_C = \text{chamber volume (7.6 L)/measured flow} \times 60 \text{ s/min}$.

Table 3. Spatial variability in the emission of NH₃, CO₂, and VOC from a cattle feedlot pen.

Constituent	Pen Site ^[a]			
	1	2	3	4
NH ₃ flux (μg m ⁻² min ⁻¹)	6.1	10.1	195.4	15.6
CO ₂ flux (μg m ⁻² min ⁻¹)	208.6	305.4	132.7	495.3
VOC flux ^[b]				
Total ion current (TIC) × 10 ⁵	23.0	20.9	170.5	20.5
Methyl sulfides (% of TIC)	0.0	0.0	16.0	0.0
Ketones (% of TIC)	0.0	15.6	8.8	17.2
Alcohols (% of TIC)	4.9	5.8	1.7	9.9
Volatile fatty acids (% of TIC)	39.5	11.4	3.0	10.2
Aromatics (% of TIC)	55.6	67.2	70.5	62.7

[a] Pen sites were roughly 3 m apart, forming a transect parallel to the feed bunk going from the fence line (site 1) to the top of the central mound (site 4).

[b] Five compound classes were identified by mass spectroscopy. Methyl sulfides included dimethyl sulfide, dimethyl disulfide, and dimethyl trisulfide. Ketones included acetone and 2-pentanone. Detected alcohols were limited to ethanol. Volatile fatty acids included acetic, butanoic, and dodecanoic acids. Aromatics included butylated hydroxytoluene, phenol, 4-methyl phenol, and benzoic acid.

Application of the dynamic flux chambers in the laboratory for the routine comparison of VOCs from different manure sources is one example of its utility in the research laboratory (Miller and Woodbury, in review). Application in the field environment was also achieved in the cattle feedlot (table 3) and demonstrated the versatility of the equipment. A range of NH₃ and CO₂ emissions was observed between the four sites along the pen transect. A similar amount of variability in relative VOC flux was also observed. It is interesting to note that although all sites appeared similar, one site produced a very different pattern of emissions (high NH₃ and VOC, and low CO₂) compared to the other three sites. Spatial variation on a small scale may prove very important when feedlot-scale fluxes are estimated, and additional data collection and analysis will clarify this relationship.

It is widely accepted that emission values measured using covered chambers, similar to the dynamic flux chamber described here, are not the best measures of actual system emissions (Sommer et al., 2004). Actual emissions are more confidently measured using wind tunnel or other non-invasive techniques such as micrometeorology (Harper et al., 1999; Meisinger et al., 2001; Arogo et al., 2003). Some of the potential drawbacks with using covered chambers include the small sample surface area relative to the pen surface spatial heterogeneity, and modified gas exchange between the surface and the atmosphere due to the restricted airflow imposed by the chamber. However, a large sample surface area can mask small-scale heterogeneity and the identification of emission “hot spots” (Sommer et al., 2004). The application of numerous inexpensive chambers would have this advantage over a single large emission chamber. Furthermore, a more appropriate use of flux chambers is for evaluating relative emission differences among treatments, hence their continued usefulness as a field survey instrument is evident (Reichman and Rolston, 2002; Sommer et al., 2004; DeSutter and Ham, 2005).

CONCLUSIONS

An inexpensive (<\$400), portable dynamic flux chamber was developed for use in laboratory and field studies for

measuring relative fluxes of a variety of cattle feedlot emissions. The hemispherical stainless steel chamber was constructed with an internal gas mixing fan. A port was fixed at the top of the chamber that could accommodate SPME gas samplers and headspace gas sampling for greenhouse gases. Furthermore, an in-line bubbler was attached for trapping and sampling ammonia. The chamber was tested to determine flow characteristics, and performed as an ideal continuous-flow stirred reactor with near 100% recovery of methane tracer, with no difference between RT_C and RT_E. As a result, concentrations measured at the sampling port are indicative of concentrations anywhere in the headspace. Application of the inexpensive chambers demonstrated that they were easy to use and required little operator input to quickly obtain multiple samples for comparing emissions in laboratory and field applications. Initial tests within cattle feedlot pens indicated that the emissions of NH₃, VOC, and CO₂ were quite variable at very small spatial scales.

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