

# Instrumentation for Evaluating Differences in Ammonia Volatilization from Broiler Litter and Cake<sup>1</sup>

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**Primary Audience:** Researchers, Nutritionists, Industry Supervisors

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## SUMMARY

Greater understanding of the mechanisms affecting NH<sub>3</sub> volatilization from reused broiler bedding is needed to determine pathways for mitigating NH<sub>3</sub> emissions. A chamber acid trap (CAT) system was developed to provide an improved laboratory method for determining NH<sub>3</sub> volatilization from litter or cake samples and for assessing treatment technologies to decrease NH<sub>3</sub> losses from poultry litter. The CAT system offers precision control of air flow rate through sample chambers as well as straightforward, precise determination of the amount of N volatilized. This article outlines the basic setup of the CAT system. The system can be utilized and modified for researching specific mechanisms involving physical, chemical, or biological treatments affecting NH<sub>3</sub> volatilization from litter or cake.

**Key words:** ammonia, broiler, cake, litter, method

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## DESCRIPTION OF PROBLEM

Management of potential pollution from animal agricultural activities requires comprehensive strategies to protect land, water, and air quality. Among these, aerial emissions, specifically NH<sub>3</sub> in broiler production, are receiving the most recent scrutiny largely due to more concentrated animal production and urban encroachment. Ammonia emission components

and quantities depend on animal species as well as the type of production system employed [1]. There is a tremendous need to develop new farm management practices and manure treatment technologies to reduce NH<sub>3</sub> losses from broiler houses, but research data and technology transfer information is lacking with respect to NH<sub>3</sub> generation in broiler litter systems [2]. Development of laboratory systems to precisely measure NH<sub>3</sub> volatilization is needed to assess volatiliza-

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<sup>1</sup>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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tion mechanisms, management practices, and treatment technologies.

The fundamental relationships among litter temperature, pH, moisture, and uric acid N with regard to  $\text{NH}_3$  production have been revealed in previous works [3–5]. However, modeling  $\text{NH}_3$  losses using these parameters remains unrealized, partially due to the spatial irregularity of the litter within the house [6]. Groot Koerkamp and Elzing [7] found it difficult to establish the extent that factors other than dry matter influenced  $\text{NH}_3$  release using layer manure. These researchers encouraged qualitative and quantitative assessment of parameters involved in volatilization and degradation reactions.

Laboratory feasibility studies can be used to develop practical solutions to reduce litter emissions within and outside broiler houses. A laboratory acid trap method, similar to the method presented forthwith, has been used to evaluate chemical litter treatments [8, 9]. Among the following treatments,  $\text{Ca}(\text{OH})_2$ ,  $\text{Al}_2(\text{SO}_4)_3 \cdot 18\text{H}_2\text{O}$  (alum), alum +  $\text{CaCO}_3$ ,  $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ , and multi-purpose litter treatment (a commercial product), the alum treatments reduced  $\text{NH}_3$  99% (alum) and 36% (alum +  $\text{CaCO}_3$ ), as well as resulted in greater total and soluble litter N. Kim and Patterson [10] also used an  $\text{NH}_3$  trapping method to show the efficacy of  $\text{ZnSO}_4$  in reducing the number of microorganisms using uric acid, which resulted in increased total N in manure.

The objectives of this work were (1) to communicate the improved flow control components of the laboratory acid trap method for evaluating  $\text{NH}_3$  release from litter and cake samples and (2) demonstrate the utility of the method for use by others. The method, which has been named the chamber acid trap (CAT) system, is a simple, inexpensive method that utilizes  $\text{H}_3\text{BO}_3$  indicator solution to capture  $\text{NH}_3$  and uses subsequent titration with HCl to determine  $\text{NH}_3$  losses.

## MATERIALS AND METHODS

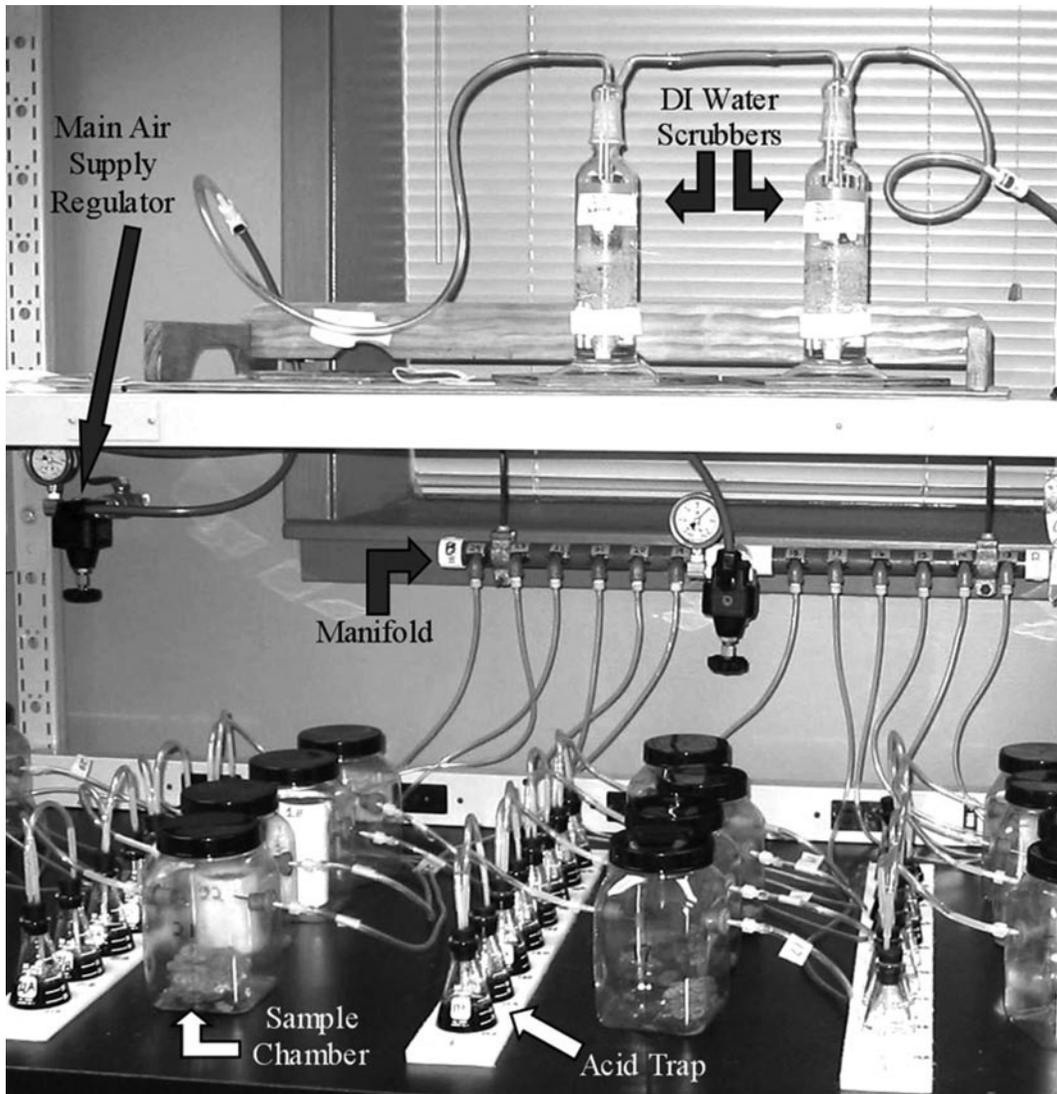
### *CAT System Description*

The CAT system is designed to measure  $\text{NH}_3$  generation from poultry litter or cake over time; however, the method is suitable for other materials where  $\text{NH}_3$  volatilization is the focus of the study. Forty-eight sealed 1-L chambers ac-

commodate litter samples (Figure 1), which are weighed and have a uniform surface area. For friable litter, the area is defined by the chamber footprint ( $93.8 \text{ cm}^2$ ). The system, similar to [6], has been modified to attain precise control of air flow rate. This modification, described in the manifold section below, is the key to the cost-effective nature of the CAT system. Four manifolds supply water-scrubbed air to circulate into each sealed container at approximately 110 mL/min. Exhaust air from each container flows through a series of 2  $\text{H}_3\text{BO}_3$  traps (50-mL Erlenmeyer flasks), each containing 30 mL of  $\text{H}_3\text{BO}_3$  indicator solution. The purpose of the duplicate traps is to ensure that no  $\text{NH}_3$  is lost should the first trap become saturated before titration. The  $\text{H}_3\text{BO}_3$  is used to capture  $\text{NH}_3$  emitted from the sample. The contents from the 2 flasks are combined and titrated with HCl every 24 h for 3 d, or as desired, and then on intermittent days to determine milligrams of N generated in individual containers for each time period. The titration endpoint is based on color change [11]. Cumulative N loss for the litter treatments, which is useful for noting overall emission differences over time, is calculated by summing the milligrams of N generated in each time period.

The system components [12] are divided into the primary functions of the main air supply, manifolds, and sample housing. A standard air compressor supplies air to the laboratory. The incoming air to the CAT system is regulated at 12 psig and then passes through two 500-mL gas-washing bottles (with fritted cylinders). The bottles contain deionized water that humidifies the air supply. The main air supply is transported via low-density polyethylene 3/8-in. outside diameter tubing to the 4 manifolds where connections use Fast and Tite black polypropylene elbow and T unions as appropriate.

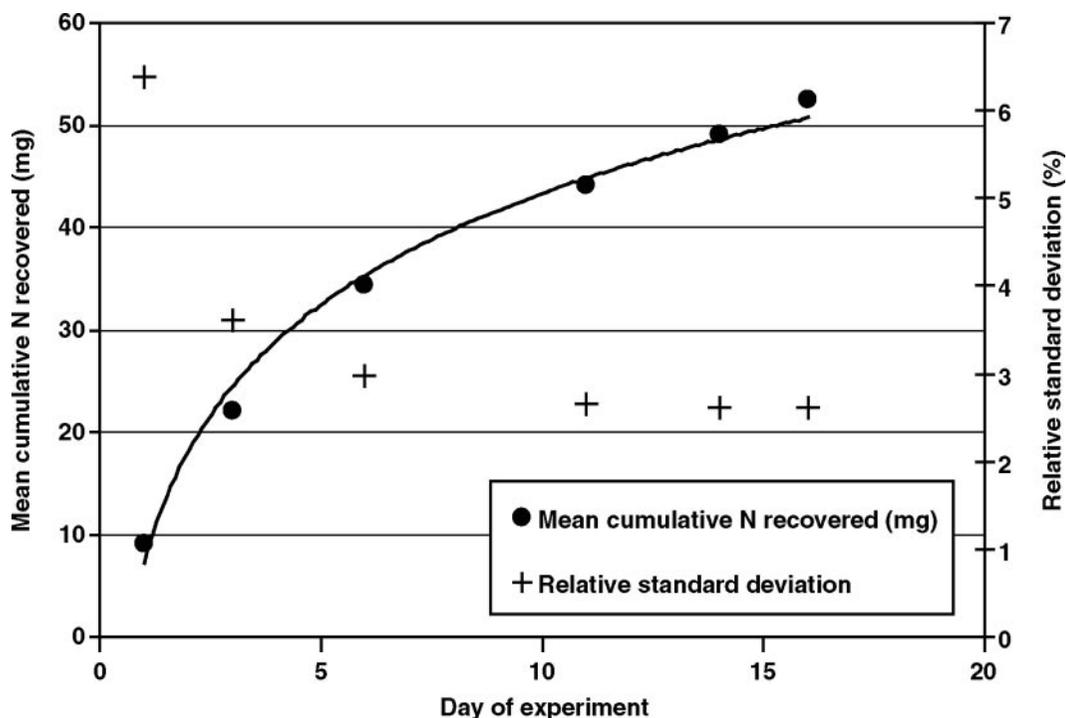
**Manifolds.** Each 3/4-in. polyvinyl chloride manifold is outfitted with a single regulator and pressure gauge controlling air flow to 12 outlet ports. The 110 mL/min flow rate results from setting the manifold pressure at 5 psig. The combination of pressure regulators and restricted flow outlet ports provide the means for precision flow control to the sample chambers on each manifold while eliminating the need for a flow meter associated with each chamber, thus minimizing equipment costs. Each outlet port was created by



**Figure 1.** Partial front view, showing major components, of chamber acid trap system for determining  $\text{NH}_3$  losses from litter or cake samples. DI = deionized water.

drilling a single barbed tube fitting (1/8-in. tube inside diameter  $\times$  1/8-in. National Pipe Thread) on the threaded side to accommodate the hub of a 27-gauge stainless dispensing needle. Epoxy is placed on the needle hub before setting it in the fitting, creating an air-tight seal. The needle-fitting assembly threads into a stainless steel street elbow that is also threaded into the manifold. Epoxy is used to ensure no leakage around the threads at the manifold surface. Street elbows were chosen for effectiveness and to minimize the space required for mounting the manifold under laboratory shelving. The male threaded

end of the street elbow could be attached directly to the manifold providing 1 less potential leakage site by eliminating the need for using a pipe nipple if a standard elbow (female connections at each end) had been chosen. There is no reason to suspect that substitution of standard elbows and nipples would affect flow precision or the  $\text{NH}_3$  detection efficiency of the system. Air to individual chambers flows via clear aquarium (vinyl) tubing that is fitted over the needle-fitting assembly and connected to each chamber using a polypropylene male luer lock  $\times$  1/8-in. tube inside diameter barb fitting.



**Figure 2.** Mean cumulative N recovered, representing  $\text{NH}_3$  losses from broiler litter samples (litter used for 18 flocks), and the relative standard deviation in measurements over time in the laboratory chamber acid trap system.

**Sample Housing.** To provide air to the chamber, a 16-gauge needle attaches to the luer lock fitting above and is inserted into a flanged stopper in the side of the sample chamber. Two holes, to accommodate the red flanged stoppers, were drilled in opposing sides of the 1-L sample chambers. The chamber outlet is comprised of a second 16-gauge needle and luer lock fitting that is attached to 4-mm outside diameter, 2.7-mm inside diameter semirigid tubing. The semirigid tubing fits into the 2-hole stoppers in each of the acid traps. The tubing leading from the chamber is immersed in the  $\text{H}_3\text{BO}_3$  so that the  $\text{NH}_3$  in the air exiting the chamber bubbles through the solution and is captured. The opposite hole in the stopper carries tubing to the second acid trap, where it is immersed in  $\text{H}_3\text{BO}_3$  solution. The opposite hole in the stopper on the second trap is open to room air; this hole provides a port for measuring air flow through each chamber. Air flow was measured using a Flow Tracker 1000 [13].

#### Chemicals and Titration

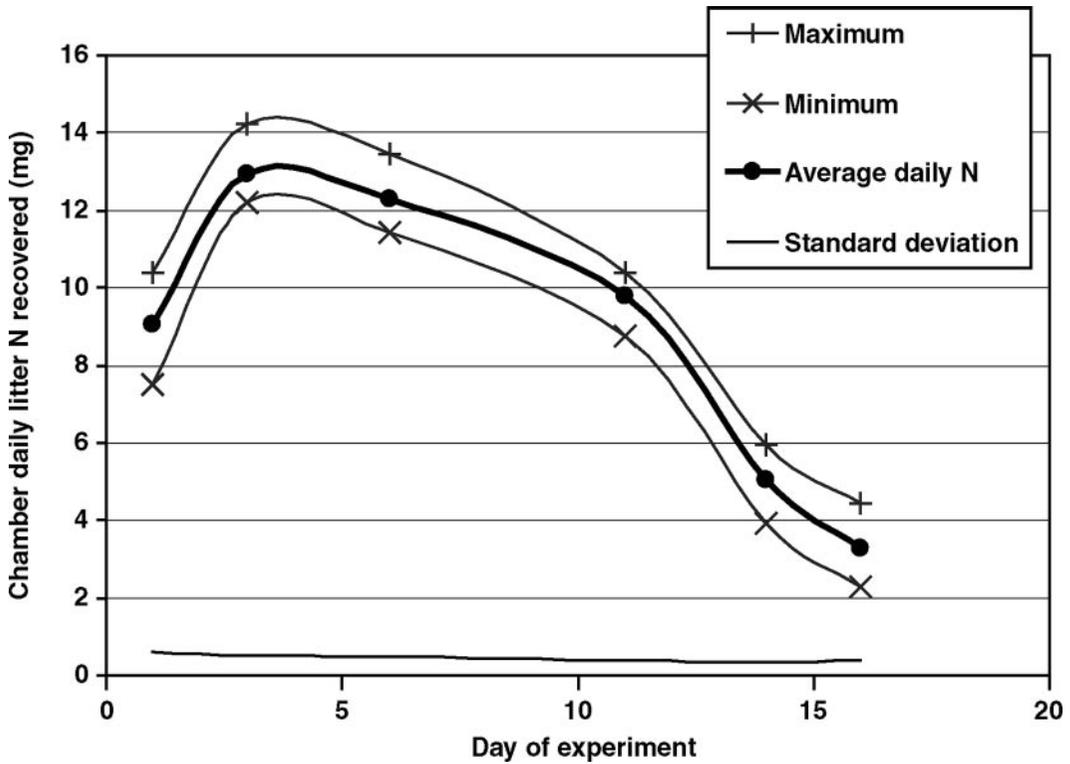
Two chemical solutions are needed to utilize the CAT system, the  $\text{H}_3\text{BO}_3$  indicator solu-

tion [14] and the titration (HCl) acid [15]. The titration of the trap solution with HCl is used to determine the amount of  $\text{NH}_3$  as milligrams of N that was lost during the sample exposure to regulated air flow in the system. The N loss (mg of N) is calculated from the following formula using the volume of HCl (mL) consumed in the titration of the  $\text{H}_3\text{BO}_3$ :

$$\text{mg of N} = (\text{mL of HCl}) \times (\text{HCl molarity}) \times \text{MW}_N,$$

where the molarity of the HCl (mol/L) has been determined by standardization with  $\text{Na}_2\text{CO}_3$  and  $\text{MW}_N$  = the molecular weight of N, 14.01 g/mol.

The system has near limitless possibilities for modification to measure gas products from poultry waste and similar materials. Smaller or larger samples can be accommodated based on changes in titration schedule and other factors. Sample size and titrant normality were originally defined by Moore et al. [8]. The volumes referenced [14, 15] are for preparing the chemicals based on using the 48 chambers in a 2- to 3-wk test period. Note [14] provides the necessary chemicals and catalog numbers.



**Figure 3.** For 50-g litter samples after 18 flocks, the average daily N recovered in the chamber acid trap system, the standard deviation among the 48 samples, and the daily maximum and minimum milligrams of N recovered among the samples.

**Titration.** The pair of stoppers connected with the semirigid tubing are removed from the acid traps and set aside; their contents are poured into a single 250-mL beaker. A magnetic stir bar is placed in the beaker, which is put on a stir plate. While stirring, a 50-mL buret with 0.1-mL intervals, containing the HCl and situated above the stir plate, is used to titrate the trap solution. As the endpoint is nearing, the trap solution changes from green to dark purple. Acid flow should cease immediately when the color bursts to reddish pink. The volume for each titration is recorded. Only a minimal amount of training is required to recognize the color change that indicates the endpoint of the titration.

To continue the experiment and prepare the CAT system for the next titration, each 50-mL Erlenmeyer flask (acid trap) should be rinsed with distilled deionized (DDI) water and shaken to remove excess water. The acid traps are refilled by pipette with 30 mL of  $H_3BO_3$  indicator

solution into each trap. The pair of stoppers is placed back on top of the traps. Bubbles should appear to indicate that air flow resumed.

### System Validation

To test the system, broiler litter was collected from a commercial farm in Mississippi during the fall season after 18 flocks had been grown on the litter (originally pine shavings bedding). The bulk litter sample was obtained from a 61 × 91cm (24 × 36 in.) area in the center-nonbrood end of the house. The collection depth was 7.6 cm (3 in.). One week before sampling, the litter had been disked, decaked, and tilled, giving it a very consistent appearance. The grower had selected these management options to attempt to release some litter moisture before the next flock. No chemical litter treatment had been applied. After transport to the laboratory, the gross sample was further mixed to ensure homogeneity and assigned randomly in 50-g allocations

to the chambers in the laboratory CAT system (Figure 1). For the original and postexperiment samples, litter moisture was determined by loss in weight (65°C for 48 h) and pH by using a DDI water:sample ratio of 5:1.

Cumulative NH<sub>3</sub> volatilization from the litter was assessed using the CAT method described above with 48 individual litter samples, each placed in a 1,000-mL chamber. Each chamber received humidified air measured at  $111.6 \pm 3.5$  mL/min, the average result of the exhaust flow [13] from each chamber measured at the start of the experiment and on d 2, 8, and 16. Ammonia loss was determined on d 1, 3, 7, 11, 14, and 16. For each chamber, the pair of acid trap contents was combined and titrated with HCl to determine milligrams of N generated by the individual litter samples for each time period. For each titration, the resulting milligrams of N lost was added to the previous total to determine the cumulative milligrams of N recovered by the trapping system. Figure 2 presents the cumulative milligrams of N recovered and the relative standard deviation, the standard deviation-mean as a percentage, obtained using the CAT system. Figure 3 reports the average daily milligrams of N recovered for the chambers, along with the standard deviation and the titrated maximum and minimum N for each day. A portion of these results have been reported previously in an abstract contrasting flux estimates derived from the CAT system vs. an instantaneous flux method [16].

Gas flux from litter is reported as mass emitted per area per time (such as mg/m<sup>2</sup> per h) and could potentially be used to develop emission factors for animal facilities. Litter flux methods have been used to evaluate treatment differences in broiler pen research trials, but the results may be confounded by differences in litter surface characteristics and by use of atypical bedding, which does not parallel that found in commercial houses. The comparative study [16] showed a range of similar estimates (29 to 40 mg/m<sup>2</sup> per h) occurred in the early stages of the CAT experiment but later in the instantaneous flux measurement. The results demonstrated the importance of time selection on the reported flux value and, thus, the caution required in using flux estimates for developing emission data.

## RESULTS AND DISCUSSION

For the validity test, exposure to the air flow in the system caused the litter samples to dry. Litter moisture for the original bulk sample was 23.1%, which decreased to 13.6% after the 16-d CAT experiment. Similarly, the pH dropped between the pre- and postexperiment measurements. The pH originally averaged 8.67 but was 8.22 at the end of the experiment.

Figure 2 depicts the quantitative litter NH<sub>3</sub> release (mg of N recovered) in the CAT system, which can be predicted by the following equation: (mg of N recovered) =  $15.7 \ln(d) + 7.1636$ , where  $R^2 = 0.99$ . The mean cumulative N collected/chamber during the experiment ranged from 9.05 mg of N on d 1 to 52.4 mg of N on d 16. Similar acid trap data reported as cumulative recovery has been useful for determining chemical treatment differences [9], but the utility for studying how litter physical and biological parameters affect NH<sub>3</sub> emissions has yet to be explored. For the current validation study, the relative standard deviation, also shown in Figure 2, was initially 6.4% and decreased to d 7, changing little beyond that day. Between d 7 and 16, the relative standard deviation remained at 2.6%.

Regarding the daily N recovery, Figure 3 shows that the NH<sub>3</sub> release increased between d 1 and 3 but then decreased with each titration through the end of the experiment, probably due to the sample drying over time. In a broiler house, renewed inputs of manure and water spillage, in addition to bird activity disrupting the litter surface, would likely maintain continuous and variable NH<sub>3</sub> emissions. The largest mean N recovered, 13 mg, occurred on d 3. The daily standard deviation of the N released from the samples remained relatively constant during the experiment, ranging from 0.6 mg of N initially to 0.4 mg of N in the latter part of the experiment (Figure 3). Considering the difficulty in determining litter emissions with an acceptable degree of accuracy and that the minimum N loss possible for the system to report is 0.13 mg of N [calculated from the titration equation given previously as the product of the least increment on the buret (0.1 mL), the HCl molarity (0.0906 mol/L), and the the molecular weight of N (14.01 g/mol)], this range of standard devia-

tion in the litter samples seems reasonable for system substantiation.

The system has been used in several preliminary investigations of litter  $\text{NH}_3$  research. An earlier version of the CAT system was employed in a study investigating the differences among litter and cake emission potential [17]. Knowing that moisture is one of the drivers for  $\text{NH}_3$  volatilization in litter systems, initial studies were conducted to ascertain the differences among litter and cake samples when considering the breakup of cake, exposing moisture-rich surfaces [17]. Using 50-g samples, the study reported cumulative losses of just under 100 mg of N for litter and greater emission for cake ( $P = 0.0002$ ), just over 250 mg of N, in a 24-d experiment.

Using the current system, a subsequent investigation of cake comparing 2 poultry houses on separate farms where one had reared 8 flocks and another had reared 18 flocks [18] showed that the farms were similar ( $P = 0.8895$ ) for cumulative  $\text{NH}_3$  emissions. Interestingly, the pH (8.97 and 9.04) and moisture (40.9 and 40.7%) were very similar for the midhouse cake samples on both farms. Other results showed greater emission from broken up samples of the same mass ( $P = 0.0411$ ) and greater emission from 50-g vs. 25-g samples ( $P = 0.0023$ ) [18]. Both studies indicate that cake removal from broiler houses is a sound management practice in litter reuse systems.

Ultimately, new technologies and strategies for emission abatement are needed for both animal health and environmental protection. A recent study employed columns filled with activated carbon made from broiler litter to capture  $\text{NH}_3$  from litter samples in the CAT system [19]. The activated carbon made from broiler litter initially adsorbed 25% of the  $\text{NH}_3$  emitted. Column design improvements, product economic evaluation, and further preliminary investigations are needed, but the study presented prospective solution-oriented litter utilization. Also, the insertion of the carbon columns into the system demonstrates how the system can easily be modified to accommodate various research interests.

The function of this CAT system has been demonstrated in this paper by characterizing the results for a homogeneous litter sample allocated to all chambers. The CAT system provides a low-cost alternative for assessing treatment

technologies by improving flow control in laboratory acid trap methods that have been used in previous studies [8, 9].

## CONCLUSIONS AND APPLICATIONS

1. The CAT system offers a simple system for quantifying  $\text{NH}_3$  losses for laboratory feasibility and mechanistic litter studies. The outlet port design economically provides precision air flow in a manner not previously reported.
2. When reporting mean cumulative  $\text{NH}_3$  released from the homogeneous litter samples, the data followed a log trend and also revealed a relative standard deviation between 6.3 and 2.6%, decreasing with time.
3. Daily  $\text{NH}_3$  losses increased through d 3 of the study and then decreased, which may be attributed to the sample drying within the chamber. The standard deviation of the litter  $\text{NH}_3$  released at each measurement day ranged from 0.6 to 0.4 mg of N.

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12. A detailed materials list and schematic can be obtained from the author of correspondence by request.
13. Flow Tracker 1000, Agilent Technologies, Santa Clara, CA.
14. Boric Acid Indicator Solution. The instructions below are given for preparing a 6-L volume of 0.32 *M* H<sub>3</sub>BO<sub>3</sub> solution for filling the traps at the test start and at each titration. This quantity provides enough solution for 2 titrations so that two 6-L batches are made about every other day for the first few days of an experiment. (1) Weigh out 120 g of granular H<sub>3</sub>BO<sub>3</sub>. (2) Add 5.5 L of DDI water to a 6-L Erlenmeyer flask. Add H<sub>3</sub>BO<sub>3</sub> from step 1. (3) Add 120 mL of ethanol indicator solution to the same 6-L flask. (A 250-mL volumetric flask is used to make the indicator by combining 0.2475 g of bromocresol green, 0.1650 g of methyl red, and bringing to 250 mL with 95% ethanol.) (4) Add 5 mL of 0.1 *N* NaOH. [Using a 100-mL volumetric flask, fill with 60 mL of DDI H<sub>2</sub>O then add 1 mL of NaOH (10 *N*). Bring to 100 mL with DDI H<sub>2</sub>O, inverting several times to mix.] (5) Bring to 6 L with DDI water. Insert large magnetic stir bar; stir until all granules are dissolved. Final color should be dark red. Fisher Scientific (Waltham, MA) catalog numbers follow the chemical names of solution components: boric acid indicator [boric acid crystal, A78-10; bromocresol green, B-383; ethanol (CDA 19 denatured), A406P; methyl red (acid-free), M-296; sodium hydroxide 10*N*, SS255-1]; titration acid [hydrochloric acid (trace metal grade), A508SK-212]; acid standardization [sodium carbonate (anhydrous/powder, certified American Chemical Society), S263–500].
15. Hydrochloric acid. In a 1,000-mL volumetric flask, add approximately 900 mL of DDI H<sub>2</sub>O. Add 8.3 mL of concentrated HCl, cover, and invert to mix. Bring to 1,000 mL with DDI H<sub>2</sub>O, cover, and invert several times to thoroughly mix.
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