

## Measuring Concentrations of Ammonia in Ambient Air or Exhaust Air Stream using Acid Traps

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Strong acid solutions have been widely used in acid traps to determine concentrations of ammonia in ambient air or exhaust air stream. A literature survey indicates the method has a long history and a wide variation in use. Through a series of studies, this paper examines several factors including volume of the acid, depth of the acid, and airflow rate; that might affect the efficiency of sulfuric acid traps and recommends steps researchers and other users may take to ensure reliable results from this method. The results from these series of studies indicate: (i) an inverse relationship between the efficiency of the acid traps and the amount of ammonia to be trapped even when the capacity of the acid trap is excessively greater than the maximum theoretical stoichiometric capacity needed to dissolve all of the ammonia, (ii) for the same volume of acid, the efficiency of the acid trap increased with the acid depth but overall, the efficiency at any given acid depth decreased as the amount of ammonia through the trap increased, and (iii) at the two airflow rates examined in this study (0.5 and 1.0 L/min) the efficiency of the acid traps decreased at similar rates as the concentration of ammonia in the sample air increased but the efficiency of the trap was significantly higher at the lower airflow rate. To obtain reliable measurements from this method, therefore, multi-point calibrations within the entire range of target measurements is recommended to provide accurate corrections of the measurements.

To determine ammonia in a stream of air or concentrations in ambient air, a sample of air is pulled through an acid solution that traps the ammonia. Most of the ammonia traps use either boric acid, sulfuric acid, or some other strong acid. Boric acid is chosen if titration is used to determine amount of ammonia trapped while sulfuric acid is used when colorimetric methods are used (APHA, 1998). The choice of either method (titration or colorimetric) can also depend on the concentration of ammonia in solution. In general, colorimetric methods are used to determine lower concentrations of ammonia in solutions (down to 2 mg/L) than titration methods, although interferences may be a problem in some cases with respect to colorimetric methods.

Ammonia absorption in the acid is a neutralization reaction behind the titration method. This understanding helps to estimate the amount of acid solution to trap most of the ammonia in the air sample. If this principle is not well understood or is not used to make these estimations, the amount of acid could be overwhelmed by the amount of ammonia in the air sample. Such situations results in poor trapping of the ammonia and hence underestimation of ammonia concentration in the air sample in question. The other extreme is the use of excessive amount of the acid in the trap. Although this situation does not really amount to a significant expense (because acid is inexpensive) the resulting ammonia concentration in the trap acid solution may be below detection limit.

A review of the use of acid traps using dilute sulfuric acid solution indicates a wide variation and lack of consistency in the use of this method. Todd et al. (2006) describes an acid trap system to evaluate effect of crude protein in the diets of beef cattle (*Bos taurus*) on ammonia emissions from beef cattle facilities. This system was based on the earlier work of both Shi et al. (2001) and Cole et al. (2005) in which both had used 100 mL of 0.9 mol/L sulfuric acid traps at airflow rates of 3 L/min. Cole et al. (2005) study was also for evaluating influence of dietary crude protein on ammonia emissions from beef cattle facilities. On the other hand, Shi et al. (2001) had used their system to evaluate the efficacy of surface amendments in mitigation of ammonia emission from beef cattle feedlots. There is neither indication of pre-estimation of the amount of acid necessary based on estimated ammonia in the air stream nor attempt to calibrate (i.e., determine trapping efficiencies) in either of these systems.

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Misselbrook et al. (2005a) used 75 mL of 0.02 mol/L orthophosphoric acid with an airflow rate of 4 L/min to evaluate effect of dietary manipulation in dairy cattle. Misselbrook et al. (2005a) conducted a one point calibration and reported a mean recovery of 97% for this single point calibration. Cabrera et al. (2001) describes another similar system used for determining ammonia loss from soil surfaces that used 150 to 650 mL of 0.05 mol/L sulfuric acid. Similarly, this study also neither indicates any attempt to rationalize the volume of the acid used nor calibration over a range of ammonia concentrations.

Guizou and Beline (2005) reports using 0.1 mol/L sulfuric acid traps to determine concentrations of ammonia in a stream of ventilation air from broiler houses. Details of the rate of air sampling and the volume of the acid used were not given. Rana and Mastroilli (1998) used 250 mL 0.1 mol/L sulfuric at 4 L/min to determine concentration of ammonia in air after field application of manure. Again, no details of calibration were provided. In a similar work, Genermont et al. (1998) used a 0.075 mol/L sulfuric acid trap at an airflow rate of 3 L/min to measure ammonia fluxes after slurry spreading in the field. And yet, in another similar study, McInnes et al. (1985) used 18 mL of 0.05 mol/L sulfuric acid at airflow rates of 3.3 L/min to measure concentrations of ammonia in ambient air to estimate ammonia flux after application of fertilizer.

More rigorous tests on ammonia trapping in acid solutions for measuring ammonia emissions from land applied manure and manure storages were reported by Misselbrook et al. (2005b) and Xue et al. (1998), respectively. In the former study, two traps in series each containing 30 mL orthophosphoric acid solution were tested at three different concentrations (0.1, 0.01, and 0.001 mol/L) and at airflow rates of 2 and 4 L/min. The conclusion was that one flask was adequate while the airflow rate and the acid strength had no significant effects on the ammonia trapping efficiency. In the latter study, Xue et al. (1998) also used two traps in series each containing 1 mol/L sulfuric acid solution. They reported ammonia trapping efficiencies of 96.45 to 99.5% in the first trap and 0.5 to 3.45% in the second trap. This system thus achieved nearly 100% trapping efficiency. The airflow rate in this system was not given.

In a review paper on methods for ammonia concentration in air, Phillips et al. (2001) notes the acid trap method is simple, cheap, and reliable and suitable for low concentrations of ammonia in air but requires a large excess dilute solution of a strong acid. This is rather subjective because there is no guidance on what "excess" acid solution means. In summary, there seems to be no consensus not only on the need to calibrate but also how to calibrate such a system. In addition, there seems to be lack of guidance in deciding what volume of the acid or airflow rate to use for accurate results.

This paper examines several factors that might affect the performance of sulfuric acid traps and recommends steps researchers may take to ensure reliable results from this method. Specifically, this study examined efficiency of an acid trap: (i) over a range of ammonia concentration in an air stream, (ii) at two different amounts of acid, (iii) at two different acid depths, and (iv) at two different airflow rates through the acid trap system.

## Methods and Materials

### System Configuration

An ammonia trap system similar to those in the literature (Shi et al., 2001; Portejoie et al., 2004; Todd et al., 2006; Cole et al., 2005) was used in this study (Fig. 1). This system essentially consisted of a simulated-manure-storage or a simulated-manure-application system, an acid bottle to trap the emitted ammonia, a critical orifice and a variable area flow-meter (Catalogue no. C-32460-42, Cole-Parmer Instrument Company; 5% full-scale accuracy) to regulate flow rate of the sweep-air, and a vacuum pump (Model DOA-P104-AA, Gast MFG Corp.) to pull air through the system. A 4-L completely sealed plastic container was used for the manure storage allowing only the entry and exit of headspace sweep-air. This air carrying emitted ammonia was delivered to the acid traps via equal lengths of 6.35 mm ID Teflon tubing in all acid trap systems to cancel effects of ammonia adsorption on the tubing. Acid bottles were made of graduated Polypropylene cylinders sealed with vinyl stoppers with two short 6.35 mm OD glass tubes. To one end of one of these tubes was connected the ammonia carrier-air tubing while the other end was connected to a short 6.35 mm ID Teflon tubing that delivered carrier-air to the bottom of the trap-bottle at the open end. The other short 6.35 mm OD glass tube was connected to the vacuum end with a Teflon tubing to exhaust the ammonia-scrubbed air out.

To avoid the effect of other extraneous factors during a certain test condition, a pair of six parallel ammonia generation and trapping systems were run concurrently. These systems generated and trapped different ammonia concentrations staggered within a range of ammonia emissions between 50 and 300 mg/d. All 12 trap systems were connected to a central sealed container which was in turn connected to a single vacuum pump. The vacuum created in this sealed container pulled the sweep-air through the manure holding containers and into the acid traps. Unless otherwise stated, all experimental runs were conducted over a period of 24 h. Critical orifices and variable area flow-meters regulated the sweep-air flow through each acid trap system. Critical orifices were made from 1-mL pipettes with tips cut to appropriate sizes to meter the desirable airflow. Variable area flow-meters calibrated against a NIST traceable air flow-meter were used to monitor the air flows.

### Study I: Effect of Ammonia Flux on Trap Efficiency

Ammonium chloride solutions were prepared to concentrations ranging between 200 and 2300 mg  $\text{NH}_4^+$ /L to simulate ammonia emissions from manure storage or manure application in the field ranging between 50 and 300 mg/d. To achieve this, 1.0 L/min ambient air was allowed to sweep over the headspace of the emitting surface after injection of 60 mL of 1 mol/L sodium hydroxide (NaOH) solution. The NaOH solution was added to the ammonium chloride solution to raise the pH of the solution and thus change ammonium ( $\text{NH}_4^+$ ) ions in solution to unionized volatile ammonia ( $\text{NH}_3$ ), whose volatilization would be enhanced by the sweep-air in the headspace.

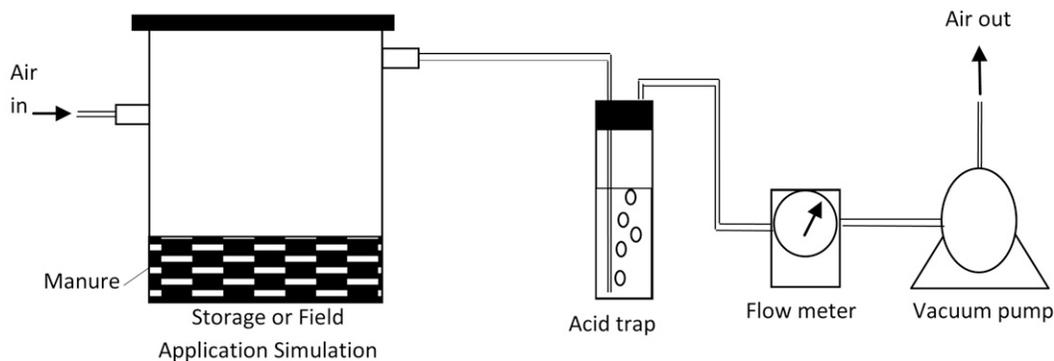
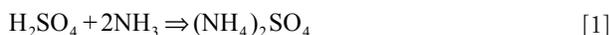


Fig. 1. Schematic of one unit of the acid trap system used in our study.

The following six concentrations of the solution were used in this study: 200, 600, 1000, 1400, 1800, and 2300 mg NH<sub>4</sub><sup>+</sup>/L to provide six different ammonia fluxes.

When ammonia is passed through the acid trapping solution, it neutralizes some of the acid. Adequate acid stoichiometric capacity of the trap system is thus critical to ensure all the ammonia in the sample air is absorbed without complete acid neutralization. For example, when sulfuric acid solution is used in the trap, stoichiometrically one mole of the acid can completely trap two moles of ammonia as shown in Eq. [1].



To evaluate the efficiency of the acid trap in a range of ammonia emissions within the acid solution capacity, 150 mL of 0.2 mol/L H<sub>2</sub>SO<sub>4</sub> (equivalent to 0.03 moles of acid) was used in the trap. The volume of the acid was calculated using Eq. [1] based on the amount of acid necessary to trap 600 mg, which was approximately double the estimated 300 mg (0.0167 moles) maximum amount of ammonia emissions expected from the system. Arithmetically, this volume of acid would thus be more than adequate to dissolve all the ammonia emitted from the entire target emission range (50–300 mg) as well as provide adequate safety capacity (~100%) to trap any over the limit emissions beyond the estimated 300 mg upper limit and up to 600 mg.

## Studies II: Effects of Volume

In subsequent studies to evaluate the effects of the volume of the acid in the trap system, the system was upgraded to hold more than three times (approximately 470 mL) the volume of the acid over the basic system (study I) that held only 150 mL while maintaining the same depth of the acid. A bigger diameter acid bottle was used to achieve this. The ammonia emission sources were also configured as in study I to generate similar ammonia emissions ranging between 50 and 300 mg over a 24-h period at an airflow rate of 1 L/min.

## Study III: Effect of Depth

Two sets each of six acid traps systems were run concurrently to determine the effect of depth of the acid on efficiency of the acid trap. Each of the acid trap in both sets contained 900 mL of the acid solution. However, the acid bottles in one set had a bigger diameter than the acid bottles in the other set

resulting into two different depths of the acid. The acid bottles of the first set of traps held the 900 mL of acid to a depth of 110 mm while the acid bottles of the second set of traps held the same acid volume to a depth of 310 mm. The ammonia emission sources were also configured as in study I to generate similar ammonia emissions ranging between 50 and 300 mg over a 24-h period at an airflow rate of 1 L/min.

## Study IV: Effect of Air Flow-Rate

To evaluate the effect of airflow rate through the acid trap, two airflow rates of 0.5 and 1.0 L/min commonly used in similar systems were selected to carry emitted ammonia. Similarly, each air flow rate was evaluated at six levels of ammonia fluxes using a set of six acid trap systems. The volumes of the acid in all the acid bottles in this experiment were maintained at 900 mL and to a depth of 310 mm. The ammonia emission flux range was set up just as in previous experiments in this study as was the 24-h experimental runs. The acid trap at 0.5 L/min was, however, run for 48 h to obtain a similar range of ammonia emission flux as in the 1.0 L/min acid-trap systems.

## Sample Analysis

In all the experiments, samples of ammonium chloride solution were taken before and after each experimental run to determine the amount of ammonia emitted from the solution. The amounts of ammonia remaining at the end of each experimental run were adjusted for the original addition of NaOH. A sample of the acid-bottle content was also taken at the end of each run to determine the amount of ammonia trapped. Before taking the samples from acid bottles and the ammonia chloride solution storages, the contents were made to the original volumes using distilled water to account for lost water during each test. To avoid loss of ammonia between sampling and analysis, 1 mL of sulfuric acid was added to 9 mL of the samples to acidify the sample. The ammonia concentrations in these samples were analyzed according to standard methods (APHA, 1992). Analyses were done immediately (within 4 h) after sampling taking into account the volume changes from the acidification processes.

## Data Analysis

The efficiencies of the traps were calculated as a percentage of the ratio of the amount of ammonia captured in the trap to

the amount of ammonia that was actually emitted from the ammonia source. For each factor, linear regressions were performed on respective efficiencies against amounts of ammonia emitted. To determine the similarities or differences due to a given factor, an analysis of covariance (ANCOVA) was then conducted on the slopes and intercepts of the respective pair of linear regressions using SAS (SAS Institute, 2003).

The ANCOVA was first conducted to compare the slopes of such a pair of linear regressions. If slopes were significantly different, then no further analysis was conducted because this information was sufficient to conclude that the linear regressions were significantly different. However, if slopes were not significantly different, then another ANCOVA was conducted to determine if the intercepts were either not significantly different (indicating overlapping or nearly overlapping regression lines) or significantly different (indicating parallel regression lines). Unless otherwise stated, statistical analyses and comparisons were conducted at the significant level of  $\alpha = 0.05$  in all cases.

## Results and Discussion

### Studies I and II: Acid Trap Efficiency and Effect of Volume

The variations of the acid trap efficiency with ammonia flux and volume of acid in the trap are shown in Fig. 2 and 3. Ammonia fluxes were determined by the differences in the concentrations of ammonia in the simulated storage before and after each experimental run. The relationships between the efficiency of the trap and the flux of ammonia as well as the volume of the acid in the trap are evident in Fig. 2. In the 150 mL of the acid trap system only 50% of the stoichiometric capacity was used to dissolve the maximum expected ammonia emission during a period of 24 h. The 470 mL acid trap system on the other hand had over six times the stoichiometric capacity of capturing the maximum expected ammonia release during a similar period. In particular, the trapping efficiency of the smaller volume (150 mL) acid trap was more erratic than that of the bigger volume (470 mL) acid trap. The evaluation of the smaller acid trap was, therefore, performed twice at approximately the same conditions and the two sets of data were used in subsequent analyses. In general, the efficiency of the trap evidently decreased as the flux of ammonia increased irrespective of the volume of the acid in the trap.

From the ANCOVA results, the slopes of the two linear regressions lines (Fig. 2) were not significantly different ( $P$  value = 0.93; denoted by  $P$  hereafter) indicating efficiencies of the two acid traps decreased at about the same rate as the flux of ammonia increased. The implication of this observation with respect to a one point calibration that is fairly common in the literature is that, if the calibration point falls within the measurement range then any correction made on the measured data using this calibration point will tend to overestimate emissions below the calibration emission while underestimating those measurements above this point. This kind of error in the correction of the measured data may end up reducing the differences in the performance among treatments, which may result in wrong conclusions or recommendations.

The intercepts of the linear regression lines of the efficiency against the flux of ammonia at the two acid volumes (Fig. 2) also were not statistically different ( $P = 0.42$ ) indicating that even the absolute efficiency of the trap did not significantly increase with the threefold increase in the amount of the acid solution in the trap-bottle. The results of these studies thus do not provide any evidence to justify use of more than double the required stoichiometric capacity of the acid in the trap to enhance efficiency of the trap. This inference is further supported by the calibrations of the acid trap at both of the two volumes of the acid (Fig. 3). The linear regression lines of the multi-point calibrations indicate good fits to the measured data, which is manifested in relatively high coefficients of determination of 0.967 for the 150 mL acid trap and 0.985 for the 470 mL acid trap. Multi-point calibration of the acid trap system following this approach thus provides a more accurate correction of the actual measurements: (i) against the expected inability of the acid trap to trap all the ammonia, and (ii) against the change in the trapping ability due to the fluctuation of the ammonia flux. The latter factor is definitely not addressed by single-point calibration procedures although it seems critical for accurate calibration of acid trap systems.

In summary, to improve the reliability of this method, besides ensuring the acid trap has adequate stoichiometric capacity to trap the maximum estimated ammonia in the air stream, a multi-point calibration of the acid trap system within the estimated range of actual measurements is essential. In addition, this calibration procedure is still critical for the reliability of this method even if the stoichiometric capacity of the acid was excessively beyond the maximum estimated concentration of ammonia in the air stream because the efficiency of the trap seems to decrease with increase in the concentrations of the ammonia in the air sample irrespective of the amount of the acid used.

### Study III: Effect of Depth

The effect of the depth of the acid on the performance of the acid trap is presented in Fig. 4. The two traps used for this study both contained 900 mL at depths of 110 and 310 mm in their respective acid-bottles. Again, the efficiency of the trap in either case decreased with increase in the ammonia concentration in the air irrespective of the depth of the acid in the trap-bottle. The slopes of the two regression lines were not significantly different ( $P = 0.91$ ) demonstrating the efficiencies of the acid trap at both the two depths decreased at the same rates with increase of the ammonia flux.

The intercepts of the two respective regression lines (at the two acid depths) on the other hand were significantly different ( $P = 0.04$ ) indicating the efficiency of the trap was higher at the greater depth of the acid. There was approximately 8% average increase in the trap-efficiency achieved by using a smaller diameter acid-bottle that increased the depth of the acid from 110 to 310 mm. The multi-point linear calibrations of the two traps shown in Fig. 5 in addition also indicate good fits to the measured vs. the predicted data as indicated by the high coefficients of determinations of 0.945 at a depth of 110 mm and 0.981 at a depth of 310 mm. The two acid traps thus calibrated very well and as long as either is calibrated over the

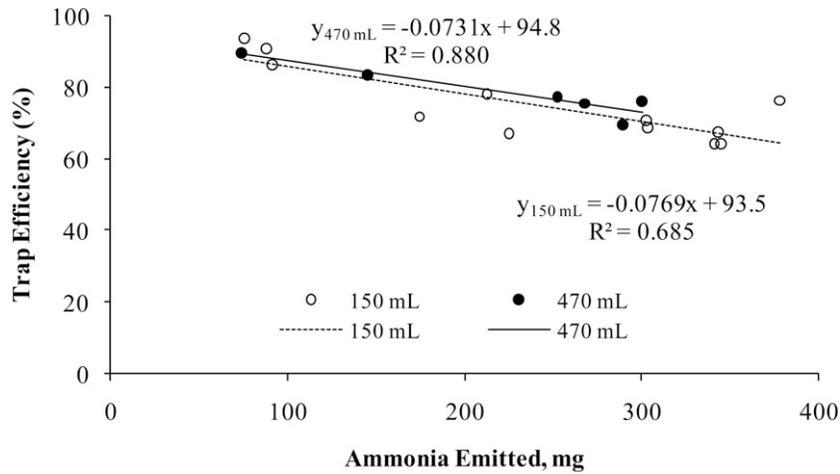


Fig. 2. Efficiency of the ammonia trap as a function of ammonia flux at two different acid volumes (150 and 470 mL) maintained at a depth of 110 mm.

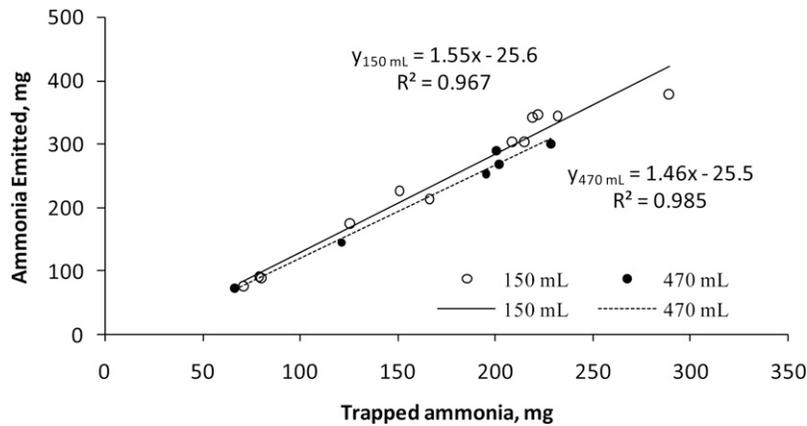


Fig. 3. Prediction of actual ammonia emitted based on ammonia trapped in the 150- and 470-mL acid trap systems both maintained at 110 mm acid depth.

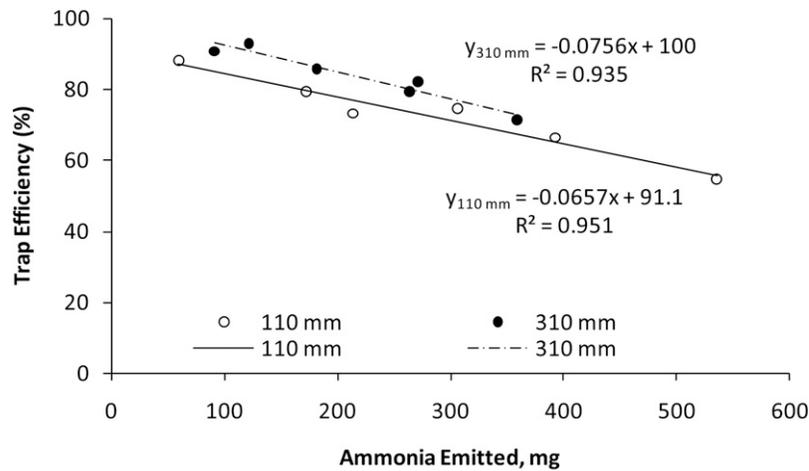


Fig. 4. Efficiency of the 900-mL acid trap as a function of ammonia flux at two different depths (110 and 310 mm).

measurements range, the corrected measurements should be reliable irrespective of the depth of acid in the trap bottle.

Based on these results, we inferred that the depth of the acid is indeed an important factor to consider when setting up a trap and given a choice between a smaller diameter (more depth) and

bigger diameter (less depth) acid-bottle for the same acid amount, a smaller diameter acid-bottle improves the absolute efficiency of the trap. This observation can intuitively be arrived at because the residence time of the air in the trap increases with the increase in depth of the acid in the trap-bottle. The increased residence

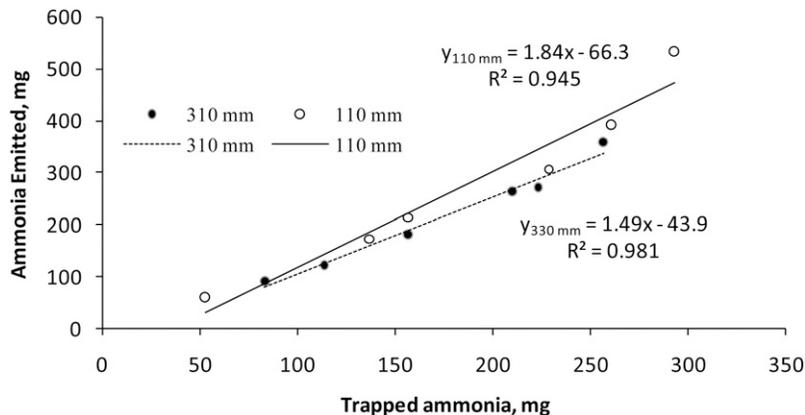


Fig. 5. Prediction of ammonia emitted from trapped ammonia in a 900-mL acid trap at two different depths (110 and 330 mm).

time provides more time for the ammonia to be absorbed in the acid solution. However, the increased depth achieved from using a smaller diameter acid-bottle in the trap did not translate into more reliability of the trap system as clearly illustrated by the multi-point calibrations (Fig. 5) at either of the acid depths.

#### Study IV: Effect of Airflow Rate

The effect of airflow rate was studied at two airflow rates (0.5 and 1.0 L/min) and the results are presented in Fig. 6 and 7. In general, the efficiency of the acid traps at both airflow rates decreased with increase in the ammonia flux (Fig. 6). The major difference was the absolute amounts of ammonia carried from the ammonia source at these two airflow rates. At an airflow rate of 0.5 L/min (meaning lower air velocity sweep through the ammonia source) the experiment had to be run for 48 h to achieve the same range of ammonia emissions as at the airflow rate of 1.0 L/min in 24 h.

Statistically, the slopes of the two regression lines of efficiency against ammonia flux at the two airflow rates (Fig. 6) were not significantly different ( $P = 0.90$ ). We thus inferred that the efficiency of the acid trap decreased with increase in the concentrations of the ammonia in the air at either of the two airflow rates and the rates of decrease of the efficiencies were not significantly different. However, the intercepts were significantly different ( $P$  value = 0.02) indicating some significant differences in the absolute efficiency of the acid trap at the two different airflow rates. This result is not in agreement with that of earlier work of Misselbrook et al. (2005b). These researchers studied the effects of two airflow rates (2 and 4 L/min) on the efficiency of two acid-traps in series each containing 30 mL orthophosphoric acid solution at three different concentrations (0.1, 0.01, and 0.001 mol/L). Their conclusions were that the airflow rate and the acid strength had no significant effect on the efficiency of the trap. In this study, lowering the airflow rate from 1.0 to 0.5 L/min increased the efficiency of the trap by an average of 4%. The results of our studies, however, seem to be logical from theoretical standpoint. Lower air-flow rates may translate into more residence time of the carrier-air in the trapping solution and thus improve the trapping efficiency.

The multi-point calibrations of the acid traps at both airflow rates are presented in Fig. 7. Again, the acid traps linear regressions for the multi-point calibrations lines are statistically good fits to the

measured and predicted data as indicated by the high coefficients of determination of 0.980 at the 1.0 L/min and 0.967 at 0.5 L/min. As in the previous two factors (amount and depth of the acid in the traps), airflow rates do not seem to significantly affect the multi-point calibrations even though in absolute terms, the lower airflow rate was slightly more efficient at trapping the ammonia.

#### Conclusions

The efficiency of the traps decreases with the amount of ammonia to be trapped even when the stoichiometric capacity of the acid trap greatly exceeds the maximum required acid capacity. The absolute efficiency of the trap did not significantly increase with the threefold increase in the amount of the acid solution in the trap-bottle maintained at the same depth. This observation thus did not provide any evidence to justify use of more than double the required stoichiometric capacity of the acid in the trap to enhance efficiency of the trap.

For the same acid amount, the efficiency of the acid trap was higher at the larger depth compared to the smaller depth. However, the rates of decrease of the efficiencies with increasing ammonia flux at different acid depths were statistically not different. When the volume of the acid provides adequate stoichiometric capacity for trapping all the ammonia estimated in a given air sample, the depth of acid in the trap should be maximized. Increasing the depth of the acid by about three times (from 110–310 mm) using a smaller diameter acid-bottle increased the absolute efficiency by an average of 8% in this study.

The efficiency of the acid trap decreased at the same rate with the increase in the ammonia flux at the two different airflow rates (0.5 and 1.0 L/min in this study) through the trap system. However, efficiency of the trap significantly increased with decrease in the airflow rate. Lowering the airflow rate twofolds from 1.0 to 0.5 L/min increased the efficiency of the trap by average of 4%.

For the acid trap method to provide the most reliable results of ammonia flux, a multi-point calibration of the acid trap system covering the entire actual measurement range is necessary to provide accurate correction factors for the actual measurements. The results of this study collectively suggest that unless the range of measurement is known beforehand the system should be calibrated after the actual measurements. This proce-

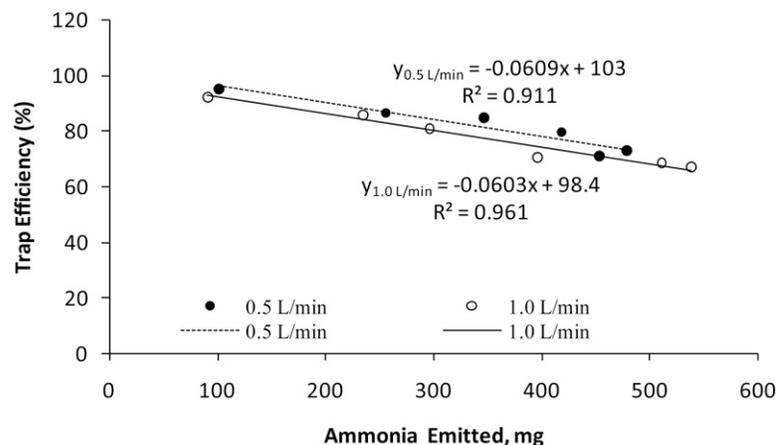


Fig. 6. Efficiency of the 900-mL acid trap system as a function of ammonia flux at two airflow rates (0.5 and 1.0 L/min) through the ammonia trap.

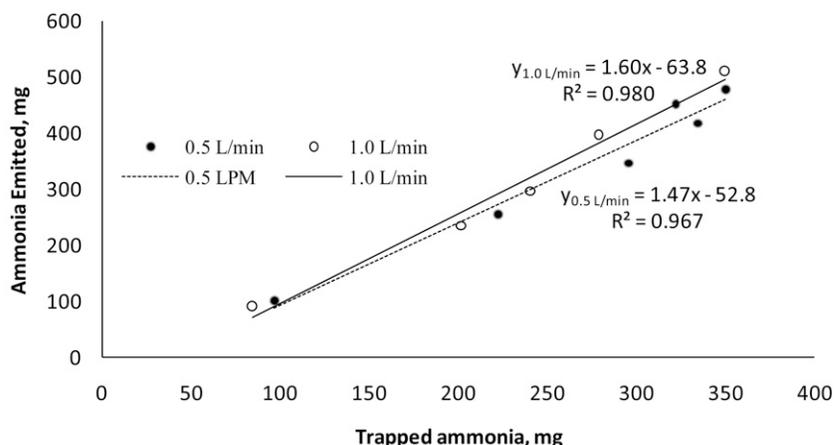


Fig. 7. Prediction of actual ammonia emitted from trapped ammonia trapped at two different airflow rates (0.5 and 1.0 L/min) in a 900-mL acid trap system.

ture ensures that the system is calibrated throughout the entire range of the actual measurements.

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