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ABSTRACT

Comprehensive field studies were initiated in 2002, to measure emissions of ammonia (NH₃), hydrogen sulfide (H₂S), carbon dioxide (CO₂), methane (CH₄), non-methane hydrocarbons (NMHC), particulate matter less than 10 microns diameter (PM₁₀), and total suspended particulates (TSP) from swine and poultry production buildings in the U.S. This paper focuses on the quasi-continuous gas concentration measurement; at identical locations, in paired barns, in seven states. Documented principles, used in air pollution monitoring at industrial sources, were applied in developing EPA-based quality assurance project plans. Air was sampled from multiple locations with each gas analyzed with one high quality commercial gas analyzer, located in an environmentally-controlled on-site instrument shelter. A nominal 4-L/min gas sampling system was designed and constructed with Teflon wetted surfaces, bypass pumping, and sample line flow and pressure sensors. Three-way solenoids were used to automatically switch between multiple gas sampling lines with 10-min or longer sampling intervals. Inside and outside gas sampling probes were between 10 and 123 m away from the analyzers. Analyzers used chemiluminescence, fluorescence, photoacoustic-infrared, and photoionization detectors for NH₃, H₂S, CO₂, CH₄ and NMHC respectively. Data was collected using PC-based data acquisition hardware and software. This paper discusses the methodology of gas concentration measurements, and the unique challenges that livestock barns pose for achieving desired accuracy and precision, data representativeness, comparability, and completeness, and instrument calibration and maintenance.

Key Words: Sampling, ammonia, hydrogen sulfide, volatile organic compounds, methane, quality control, animal housing, continuous emission monitoring, emission factors

IMPLICATIONS

Air emission is the next major manure management issue that U.S. agriculture faces. Gas pollutants emitted by concentrated animal feeding operations (CAFOs) can create neighborhood nuisance, animal and/or human health concerns, or non-compliance with state and/or federal regulations. Currently, an assessment of the true impact of these pollutants is limited by the lack of reliable emission rate data. Gas emission measurement methods described in this paper were
used to determine baseline emission rates for animal confinement buildings, provide data for the development of process-based models, and test abatement methods.

INTRODUCTION

Air pollutants emitted by livestock and poultry production operations represent potential risks to: the health and well-being of herdsmen and livestock, people in neighboring areas, and the global environment (NRC, 2003). Air pollutants of particular interest are: ammonia (NH₃), hydrogen sulfide (H₂S), volatile organic compounds (VOCs), and particulate matter (PM₁₀ and TSP). Odor contributes to the nuisance experienced in areas surrounding livestock facilities. Additionally, methane (CH₄) and carbon dioxide (CO₂) are considered to be important greenhouse gases.

Two collaborative, multi-state emission studies of air pollutants from animal production began in 2002. A six-state project entitled “Air Pollutant Emissions from Confined Animal Buildings” (APECAB); was designed to quantify and characterize baseline emissions of NH₃, H₂S, odor, PM₁₀, and TSP from four swine barn types and two poultry barn types. A study entitled “Control of Air Pollutant Emissions from Swine Housing” (CAPESH); involved similar measurements at a fifth swine barn type, plus measurements of CH₄ and NMHC. The study included an evaluation of automatic soybean oil sprinkling, misting of essential oils, and fan plume obstruction and vertical deflection curtain.

The APECAB study was a collaboration of land-grant universities in six states (IN, IA, MN, IL, NC, TX) while the CAPESH study was conducted by land-grant universities in two states (IN, MO). Both studies used measurement methodology based on previous work (Heber et al., 2001; Heber et al., 2003). At each measurement site, a mobile on-site instrument shelter (OSIS) was stationed between two identical or nearly identical; mechanically-ventilated, confined animal production buildings. Emission measurements were quasi-continuous. The OSIS housed a gas sampling system (GSS), gas analyzers, environmental instrumentation, PC-based data acquisition system, controller units for real-time PM monitors, standard gas cylinders, and various supplies and equipment. Gas concentrations were extracted and measured at the air inlets and outlets of each barn, while total barn airflow was simultaneously monitored. Odor samples were collected approximately biweekly to determine odor emissions. Emission rates, at any instant, were calculated by multiplying differences in concentration between ventilation inlets and outlets by barn airflow rates (Heber et al., 2001):
\[ E_i = Q_{\text{air}} \times (C_{i,\text{exhaust}} - C_{i,\text{inlet}}) \]  

where: \( E_i \) = emission rate for gas “i”, \( Q_{\text{air}} \) = ventilation rate through a barn, \( C_{i,\text{exhaust}} \) = concentration of gas “i” in the ventilation outlet, and \( C_{i,\text{inlet}} \) = concentration of gas “i” in the ventilation inlet.

Greater emphasis was placed on data quality; to maximize confidence, credibility and comparability of these measurements, as compared with previous studies (Heber et al., 2001). Testing over 15 months allowed annual emission factors to be fully characterized. Long-term measurements allow for the assessment of variations caused by seasonal effects, animal growth cycles, diurnal variations, and waste management practices. The purpose of this four-part series is to describe how well-established principles of quality assurance and quality control (QAQC) were applied to emission measurements at livestock barns in order to develop a common protocol for both studies. Parts 1-4 address gas concentrations, particulate matter concentrations, odor concentrations, and barn airflow rate, respectively.

**BACKGROUND**

Field measurements of gas concentrations and emissions at commercial livestock barns have been previously addressed (Phillips et al., 2000). A ranking exercise was conducted on all the possible approaches to measuring ammonia. The approaches were ranked according to: quick turn-around, usefulness to barns and animal waste storage, meteorological flexibility, procedural ease, operating costs, capital costs, repeatability, bias, time-resolution, required operator skill, and range of detection, to reflect scientific, practical, and financial considerations. The desired accuracy is ±5% for concentration and ±5% for airflow. The selection of sampling time and location for livestock barns is not straightforward, but is important for obtaining representative gas concentrations for emission determinations. Ventilation exhaust is the most appropriate sampling location for emission measurements in negative-pressure, mechanically-ventilated barns. Relatively accurate gas emission rates can be estimated, since the ventilation rate can also be measured at the exhaust(s) (Heber et al., 2001; Berckmans and Ni, 1993; Hartung et al., 2001). Sampling time and frequency should be designed to cover diurnal and seasonal swings in concentration (Ni and Heber, 2001). Sampling frequency with automatic multipoint systems, depends on the number of locations and the response-time of the measurement system.
Measurement of gas concentrations at livestock barns is subject to a number of potential errors (Table 1). However, little information about QAQC (calibration of measurement systems, assessment of precision and bias) has been provided in most studies of livestock barn air quality (Ni and Heber, 2001), as a consequence of the lack of attention in data quality. Primarily, this is due to the deficiency of QAQC requirements by research sponsors, and therefore by researchers themselves. However, this scenario is changing with greater funding of emission studies by sponsors that require QAQC documentation.

A 12-month field test of emissions at 8 swine finishing barns was conducted using: one set of unattended continuous analyzers per barn pair (measure gas concentrations in multiple air sample streams) that were pneumatically-switched, under PC control, on 10 to 15-min sampling intervals through Teflon sampling tubes (Heber et al., 2001). Multiple filtered air streams (sample location group) were combined into single air streams that were pumped to the analyzers to measure concentration. The maximum residence time in the 6-L/min gas sampling system was 45 s. Analyzers in the environmentally-controlled OSIS were calibrated weekly by full-time field engineers. Some of the 62 barn-months of emission data collected was published (Heber et al., 1997; Heber et al., 1998; Heber et al., 2000; Lim et al., 1999; Ni et al., 2000; Ni et al., 2002; Ni et al., 2000).

Recommendations to improve gas emission data in future studies were as follows (Heber et al., 2001): develop a detailed quality assurance project plan, maintain air sampling tubes 3ºC above the sampled air temperature, replace gas line filters on fixed schedules, design equipment against damage and corrosion, monitor space-heater operation, select low-maintenance equipment and sensors, and provide remote online access to real-time data.

EXPERIMENTAL METHODS

Description of Barns

Some characteristics of the barns monitored in the APECAB and CAPESH studies are described in Table 2. The Missouri and Indiana sites are discussed in this paper in greater detail, to illustrate housing examples in swine and poultry production respectively. Each swine barn at the Missouri site (Figure 1) had two rows of 24 pens and a center alley. Manure was collected in four shallow gutters. Each gutter was flushed four times per day with recycled lagoon liquid.
Ventilation air typically entered the attic through the eaves and into the room through passive, gravity-operated ceiling air inlets. The barns were tunnel-ventilated during warm weather in the summer. Air entered through openings in the sidewall curtains near the east-end of the barn. The operation of the flushing valve and space-heater were monitored as process control variables that affect emissions.

The Indiana site consisted of two caged-hen layer barns in a 16-barn complex. Each barn had ten, 177-m long rows of cages, in the 3.3-m high second floor. Manure was scraped off boards under the cages into the 3.2-m high first floor. Twenty-five 918-mm dia auxiliary-circulation fans enhanced manure drying on the first floor. Incoming air flowed through 2.7-m high, evaporative cooling pads in the roof of the attic. Fresh air then entered the second floor through temperature-adjusted baffled ceiling air inlets. The number of belted exhaust fans operated in stages 1-8 were: 5, 10, 18, 26, 35, 42, 56, and 75 respectively. In stage 9, the water-flow of the evaporative cooling system was activated, and the 19 fans turned on for stage 8 were shut off.

Figure 2 displays the schematic of the monitoring plan for the two barns. Each exhaust location was sampled individually; via one tube, whose end was located about 0.5 m directly in front of the fan, at the same height as the fan hub. Each air inlet and animal exposure SLG consisted of three sampling lines (or laterals) connected in parallel to a mixing manifold. Each lateral sampled from a location in the middle of each of three lengths of the barn. The end of each lateral tube, for the air inlet SLG, was located in the attic about 10 cm above the baffled ceiling opening. The end of each lateral, for the animal exposure SLG, was located in an emptied cage that was about 0.75 m above the 15-cm wide manure slot. Ventilation air entered the pit through the slots.

The Texas site consisted of two swine finishing barns at a 5-barn complex. The pigs were confined to 54 pens per barn, located on both sides of a narrow central walkway. The typical growth cycle was approximately 20 wks long. The barns were 72.1 m long and 12.6 m wide. Waste was collected in a shallow pit, drained weekly by discharging to an on-site earthen basin. The pit-bottom was recharged with fresh water or water from the lagoon, depending on the basin level. Barns were tunnel-ventilated in the summer. The inlet ventilation air entered through two side curtains at the short side of the barn. In the winter, air entered via the attic and the 20 ceiling diffusers (uniformly distributed lengthwise in the barn over the pens). Cold weather heating was
aided by two natural gas heaters per barn. Hot weather cooling was aided by a misting system; consisting of approximately 60 nozzles, evenly distributed on both side walls. Five fans exhausted air from the barn. Sampling lines were located at the inlet curtain, inlet diffusers, exhaust fans (2), and in pens that were located at 1/3 and 2/3 the length of each barn. The thermocouples at the 1/3 length also monitored the operation of space heaters. The Texas, Missouri and Iowa sites were very similar, except for waste handling. For all sites (Table 2), the collaborating producer recorded mortalities, animal inventory and weight, water and nutrient consumption, and the occurrence of special activities (e.g., generator tests, manure removals or agitation, changes in diet and animal health, temperature set points, ventilation interventions, barn cleaning, and power failures).

**Approach to Gas Concentration Measurements**

Sampling location groups (SLGs) consist of multiple probes that bring air into a mixing manifold from multiple sampling points (Heber et al., 2001). Samples from multiple locations were acquired via one gas sampling line, by making composites with a mixing manifold (e.g. four pit exhaust fans, five end wall tunnel fans, etc.) (Heber et al., 2001). However, there are several reasons for having individual sampling points, compared with a mixed stream from several points. One reason is that all data need not be invalidated upon failure of one or more of the exhaust fans. In other cases, all exhaust points were not at continuously-operating fans, and multiple fans may have different capacities. Furthermore, the individual concentrations can be averaged; while having the benefit of additional exhaust point characteristics. If mixed, only the mean value is known, and the contributions of each point become unknown. In these studies, there were 1 to 4 exhaust SLGs, 1 to 2 ventilation inlet SLGs, and an animal exposure SLG (Figures 1 and 2).

The allocation of the SLGs at each barn depended on site-specific barn configurations. The number of inlet or background SLGs ranged from one for both barns (CAPESH site) to two per barn (APECAB site). By reason of spatial variation, multiple exhaust locations were used to enhance the representativeness of the exhaust concentration for emission calculations. Since there were multiple exhaust points in a barn, it was not advisable to use the concentration measured at one fan, and the airflow of another fan, to calculate the emission rate of both fans. This is
because the fans could be separated by a large distance, or, one fan might be in a manure pit, while the other is in the wall. If the fans are grouped together (tunnel ventilation), a single point can be representative of air exhausting from any of the fans (e.g., Figure 1). The number of exhaust SLGs ranged from 2 to 4 among APECAB barns, whereas the CAPESH barns had only one SLG at the exhaust.

A gas sampling system (GSS, Figure 3) in the environmentally-controlled OSIS, drew continuous gas samples from each SLG. Air from each SLG was sampled and measured continuously for one sampling period, before switching to the next SLG. The use of a 10-min sampling period, for 6 to 12 locations, resulted in 12 to 24 sampling periods per day for each location. The sampling sequence should be randomized to minimize systematic bias, due to residue from the previous sample (Hartung, 2002). The first 7 to 9 min of gas concentration data are ignored to allow all gas analyzer outputs to stabilize. The response time of the system was tested by attaching a 50-L bag of calibration gas at the end of the longest sampling tube. This helped determine that a 7 to 9-min purge achieved a 90% minimum response to a step input. Hourly sampling of exhaust air was sufficient to capture diurnal variations in emissions (Hartung, 2002). Although the APECAB project used a 1- to 2-hr sampling cycle, there were 2 to 4 exhaust locations in each barn. Therefore, exhaust concentrations in each barn were sampled 24 to 96 times per day.

Hourly sampling of exhaust air was necessary in the CAPESH study. After initially using a 10-min sampling period, sudden peaks in H$_2$S and CH$_4$ were observed, which corresponded to the 2-min flushes that occurred in the manure pit every 30-min. Based on the need to capture at least one complete rise and fall of flush-induced gas concentration, the sampling period was increased to 60 min for the exhaust SLG. The sampling period for ambient air was increased to 20 min, due to the observed slow stabilization of the NH$_3$ analyzer output to relatively low ambient concentrations.

The duration of samples at a given SLG can be calculated as: the total number of samples multiplied by the number of readings per sample. Sampling time ranged from $24 \times 3 \text{ min} = 72 \text{ min (1/20 of the day)}$ to $12 \times 1 \text{ min} = 12 \text{ min (1/120th of the day)}$. Although this seems like a very small data capture percentage, the frequency of sampling, compared with the frequency of the measured variable is important, not the total duration of sampling. The twelve, 60-sec samples were distributed throughout the day, thus capturing the diurnal variations of emissions.
GAS SAMPLING

Gas Sampling System

Purdue University designed and constructed the gas sampling systems (GSS, Figure 3) that were used for each project. An array of 12, 3-way solenoid valves (S1-S12) (Part #648T032, Neptune Research, West Caldwell, NJ) in the GSS, facilitated automatic sequential gas sampling from multiple locations. The samples were drawn through 10 to 123 m long FEP Teflon tubes (6.4 mm ID) at 4-5 L/min. A 47-mm dia., in-line Teflon PFA filter holder (Part 6-47-6, Savillex Corporation, Minnetonka, MN) housing a 47-mm dia., Teflon PTFE-laminated polypropylene membrane filter (Part U-02916-64, Cole-Parmer, Vernon Hills, IL), with 0.45- to 1.0-µm pore size, was installed at the sampling probe to remove airborne particulates from sampled air. The filters were replaced at least biweekly.

The selected gas stream flowed sequentially from the sample probe, via tubes, through a 3-way solenoid valve, manifold (M2), Teflon-lined diaphragm pump (P2) (Part 107CAB18-TFEL, Combined Fluid Products, Lake Zurich, IL), mass flow meter, flow restrictor, and sampling manifold (M3). There, internal or external pumps of the gas analyzers drew air through a short (<3 m) 3.2 mm I.D., 6.4 mm O.D. tube. Manifold M2 and the solenoids were connected together with 22 to 38 cm long, 6.4 mm I.D., 9.5 mm O.D. tubes. All tubes, solenoids and fittings were Teflon. Odor samples were collected through an odor sampling port. Bypass pump P1 drew air from all inactive (unsampled) sampling tubes via 3-way solenoid valves and manifold M1 at approximately 1-L/min per tube. Bypass pumping reduced the response time of gas analysis, by at least the residence time in the tubes, e.g. 38 sec for a 100 m long tube at 5 L/min. The GSS unit (Figure 3) allowed for automated calibration of all analyzers using an optional standard gas manifold module. The operation of solenoid valves in this calibration module could be activated remotely using Labview software.

A 0-6895 Pa differential static pressure sensor (Model 2301001PD2F11B, SETRA, Inc., Boxborough, MA) and a stainless-steel lined 0-10 L/min mass flow meter (Model 50S-10, McMillan Company, Georgetown, TX) provided a permanent QA record of negative pressure and volumetric flow in sampling manifold M2 for each SLG. The resulting real-time display of sampling airflow and pressure facilitated troubleshooting the GSS. Low negative pressure in
manifold M2 could have indicated sampling pump failure or deterioration, air leaks in the system, or ice formation in GSS exhaust hoses outside the OSIS. High negative pressure may result from overloading filters with PM or condensate in the sampling lines. The control system was capable of being programmed to shut off the gas sampling pump in case of abnormal readings. The mass flow meter and the manifold pressure sensor were used to assure proper GSS operation (Figure 11). Abnormal M2 pressure indicated leaks following a filter change (low vacuum) and blockage due to condensation in gas tubing (high vacuum).

Gas Sampling System Leak Testing

An external system tested leaks in the GSS (Figure 11). To test for leakage, all solenoids were shut off from manifold M2. Pump P2 was turned off. Bypass pump P1 could be either on or off during the test. Pump P4 was operated to produce airflow as recorded by the rotameter, the system was airtight if zero airflow was indicated. The empty jar dampened pump-induced vibrations. Items tested include: pump P2, manifold M2, mass flow meter, flow restrictors, solenoids, and all fittings and tubing that connected these devices. Leaks were often found in the solenoids and fittings, so tests should be done during setup and on a regular basis thereafter, e.g. bimonthly.

Gas Transfer Tubes

Gas sampling tubes transferring relatively warm moist air must be maintained above the dew point temperature of sampled air at all times. Condensation control and prevention is critical, because many of the gases being tested (NH₃, H₂S, and CO₂) partition to water result in measured concentration errors. Thus, if large errors are introduced by condensation then the capability of accurate concentration measurements with expensive analyzers is in vain. Condensation occurs inside tubes at temperatures only a few degrees cooler than air sampled. For example, air at a typical condition of 20°C and 70% humidity will condense at 14.4°C. Sometimes, indoor humidity and temperature are higher, for example 25°C and 80% at which condensation occurs if the sampling line is cooled to less than or equal to 21.3°C. The assumption of a well-mixed ventilated space with uniform inside temperature is invalid in a typical livestock barn. Many planned and unplanned entries of cold outside air into the barn, and
cold inside surfaces, produce a treacherous minefield for unheated sampling lines in these barns. The trouble spots include: air inlets distributed along the walls and ceiling, leaks through fan backdraft shutters, doors, windows, curtains, and through holes in the barn envelope created by rodents and barn damage or deterioration. Raceway entrances into the barn and the OSIS are points of potential condensation, as well as the OSIS itself. A filtered, electric-powered, positive-pressure HVAC system in the OSIS (to keep pollutants out) must maintain inside temperature within instruments’ operating range and above the dew point of sampled air to prevent condensation in the exposed unheated tubes. This is especially true during the winter, when inside relative humidity is high. Cool air from the HVAC system must be directed away from unheated tubing. OSIS temperature should be recorded. The obvious minimum precaution involves heating the bundle of gas tubes in a insulated suspended raceway, between the barn and the OSIS. The most reliable and risk-free protection however, involves heating the entire length of all sampling lines; however, this can be prohibitively expensive when using systems that have many sets of long tubes (Table 2). The strategy used in these studies was to run the lines through the warmest path between inside air sampling locations and the raceway to the OSIS, sufficiently avoiding cold spots to prevent condensation.

The gas sampling plan in the APECAB project consisted of 10- to 115-m long FEP Teflon tubes (6.4 mm I.D. × 9.5 mm O.D.) that extended from the GSS in the OSIS to various sampling locations within the barns (fan exhausts, ventilation inlets, animal zones). Heated sampling tubes were used to prevent condensation where tubes might be exposed to cold ambient air. The APECAB project sites typically used self-regulated heat tape (typically 49 W/m nominal) that was controlled either by PC or thermostat. One option was to power the heat tape continually, since it was self-regulated. However, the CAPESH site used a non-regulated, custom-built, heat tape (39 W/m) wrapped around each individual tube. A capillary tube thermostat protected the circuit from overheating (Figure 5), and a backup thermostat ensured heating in case of PC failure. An autotransformer was also used to reduce the potential heating capacity of an oversized heating system.

**Gas Sampling Time**

Sampling time is critical for obtaining quality data. On the one hand, with multipoint sampling, it is most desirable to move through the cycle quickly, sampling all locations within a short
interval. However, analyzers must equilibrate, and moving too quickly to the next location might create a systematic bias to the data. Because, analyzers do not have sufficient time to output the actual concentration. Tradeoffs with respect to the response times of the analyzers, should be considered when choosing the sampling period, and should be dependent on the slowest analyzer. The average of multiple sequential valid readings (one or more) following equilibrium of the analyzer, within a sampling period, is the concentration for that sampling period. A 10-min sampling period was used in the APECAB study, but sampling period was site specific. For example, the sampling period was changed to 60-min in the CAPESH study, because waste gutters in the barns were flushed every 30-min for 2-min and caused pronounced peaks of H₂S and CH₄. A 60-min cycle would capture at least one full peak whereas a 10-min cycle with a 9-min equilibrium time would have missed most of these events. More time was required for the NH₃ analyzer to decrease to a low reading (e.g. zero), as compared with the time required to increase to a high reading (e.g. span gas). Consequently, a 20-min sampling period was used for the background sample in the CAPESH study and the majority of the APECAB sites. Net emissions are underestimated if ambient samples are biased high due to short sampling periods.

**GAS ANALYZERS AND CONCENTRATION MEASUREMENT**

**Ammonia**

Ammonia was measured real-time with a chemiluminescence-based NH₃ analyzer (Model 17C, Thermal Environmental Instruments (TEI), Franklin, MA). It was a combination NH₃ converter and nitrogen oxides analyzer that is typically used for ambient monitoring, but has a range of 1 ppb to 200 ppm (capable of measuring the typical concentrations inside animal barns³,¹⁵). Besides having an appropriate range for source measurements, the chemiluminescence method was chosen for its stability, reliability, and high precision (0.5% of full scale). The analyzer’s full scale was adjusted between 20-200 ppm, depending on maximum levels expected at the barn. If NO and NO₂ measurements were negligible, the analyzer is operated in the total nitrogen (N₉) mode, to decrease response time and costs of NH₃ scrubber replacements.

Figure 6 shows a 24-hr record of NH₃ concentrations measured at the CAPESH site. Concentrations measured at these barns typically range from 5 to 35 ppm in exhaust air, and less than 0.3 ppm in the ambient air. During the 20-min ambient sampling period, concentrations
sometimes decreased, even during the last 10-min of each cycle. The relatively long time to calibrate is common with chemiluminescence-based ammonia analyzers.

**Hydrogen Sulfide**

Hydrogen sulfide was measured in real-time using a pulsed fluorescence-based SO\(_2\) detector (TEI Model 45C) (U.S. EPA. Method EQSA-0486-060) following conversion of the H\(_2\)S to SO\(_2\) with a converter (TEI Model 340). The SO\(_2\) analyzer has a range of 0.01 to 10 ppm, response time of 60 s (10-sec averaging time), sample flow rate of 0.5 L/min, guaranteed precision of 1% of reading or 1 ppb (whichever is greater), and data averaging time of 60 s. Further details are given elsewhere (Heber et al., 2001). Figure 7 provides an example of H\(_2\)S measurement. Sharp peaks in H\(_2\)S, up to 1,100 ppb, were observed during flushing of under-floor manure gutters using anaerobic lagoon effluent. This observation of dynamic changes in concentration is an effect of a process-related event and illustrates the real-time vs. integrated sampling benefits.

**Carbon Dioxide**

Concentrations of CO\(_2\) were measured using 2,000-ppm and 10,000-ppm photo-acoustic infrared-based CO\(_2\) analyzers (Model 3600, Mine Safety Appliances, Pittsburg, PA). The CAPESH site had one 10,000-ppm analyzer, whereas the APECAB study used an additional 2,000-ppm analyzer to increase the sensitivity of CO\(_2\) measurements in the lower concentration range. The sensor utilized dual-frequency photo-acoustic infrared absorption, and was corrected for water vapor content. The guaranteed precision of this analyzer is ±2% of full scale, and the sample flow rate is about 1.0 L/min. A bubbler was needed, in-line from the calibration cylinder, to add the needed moisture for stable analyzer operation during calibration. An example CO\(_2\) concentration record at the CAPESH site on November 8, 2002 is shown in Figure 8.

**Methane and Total Non-Methane Hydrocarbon Analysis**

The CH\(_4\) and the total NMHC concentrations were determined in real-time using a back-flush GC system with a flame ionization detector (TEI Model 55C), in accordance with U.S. EPA
Reference Method 25. It was an automated batch analyzer that repeatedly collected and analyzed of the 0.5 L/min sample stream drawn in by the internal pump. The instrument had a sensitivity of 75.1 \( \mu \text{g/m}^3 \) of NMHC as propane. The \( \text{CH}_4/\text{NMHC} \) analyzer used a two-point calibration that used zero and a span. Zero readings were taken at times when no hydrocarbons were expected to elute from the column during the span gas calibration. The analyzer could be programmed for automatic calibrations, e.g. 7:30 a.m. daily at the CAPESH site, by connecting a span gas to the analyzer’s internal calibration circuit. If the NMHC compound used in calibration was hexane, and other compounds were measured, instrument readings would not be a direct measurement of concentration in ppm. This problem can be addressed by expressing concentrations in ppmc rather than ppm. The concentration in ppmc is calculated by multiplying the concentration in ppm by the number of carbon atoms per molecule in the span gas. A response factor is a linear coefficient that adjusts the instrument reading according to an experimentally determined relationship between calibration gas and the compound being sampled.

Figures 9 and 10 show 24-hr records of \( \text{CH}_4 \) and NMHC concentrations from the CAPESH site respectively. The \( \text{CH}_4 \) concentrations ranged from about 5 to 60 ppm in exhaust air and were highly correlated with flushing events. Ambient concentrations were less than 3 ppm. Relatively low concentrations are expected in a barn with frequent flushing of manure. The NMHC concentrations ranged from 10 to 300 ppbc with an unexplained peak of 1,100 ppbc in Barn 7. Automatic calibration of the analyzer, with 2.03 ppmc hexane, occurred at 7:30 a.m. To avoid possible interference from oxygen, the methane span concentration should not be less than about 1.5 ppm. To obtain good calibration repeatability, the concentration of NMHC concentrations may be above 0.5 ppmc. As a general rule, the instrument is accurate to \( \pm 2\% \) at sample concentrations ranging between 20\% and 120\% of span concentrations.

**Quality Assurance and Quality Control**

Calibration of analyzers assures data quality and provides information about characteristics of the devices such as: response, drift, linearity, stability, and precision. For example, the difference between known NH\(_3\) concentrations and NH\(_3\) analyzer outputs, guides the adjustment of system hardware/software or the correction of concentration data during data processing (Heber et al., 2001; Phillips et al., 2001). During calibrations, gas analyzers operate in their normal sampling mode; calibration gas passes through as much of the sample probe and sample-handling system
as is practicable (U.S. EPA, 1998). It is not always practicable to calibrate the instrument by introducing calibration gases directly into a remote sampling probe (U.S. EPA, 1998). The measurement system in this study included the analyzer, an external GSS, and an array of long sampling lines, with filtered probes on the end of each line. An NH₃ measurement system schematic, Figure 11, showed that calibration gases could be introduced into this measurement system at points A, B, C, or D.

To determine analyzer performance, a multipoint calibration of the gas analyzers was conducted at A, in triplicate, using either a series of gas cylinders; or, with a precision gas mixing and dynamic dilution system (Model 4040, Environics, Inc., Tolland, CT) to provide a series of concentrations that spans the expected range of the target analyte. The calibrations were conducted at the beginning of the project and following long interruptions in service or analyzer malfunction. The maximum gas concentration in the series is preferably between 70 and 120% of expected concentration levels. Alternatively, the calibration gas could be introduced at points B, C, or D for calibrations. However, more calibration gas is required due to the flow rate of the external gas sampling system (4.0 L/min as compared with 0.6 L/min). Multipoint calibration of the NH₃, H₂S, and CO₂ analyzers, and the least squares regression line, were used to convert the analog output (VDC) of the analyzer to engineering units (ppb).

Routine zero and span checks were conducted every 1 to 7 d by introducing calibration gases into the probe manifold M2 (point B). The calibration gas introduced at B flowed through the GSS, except for the solenoids that receive the gas from the sampling tubes (Figure 11). In the CAPESH study, a 5.0-L/min, 6-port diluter allowed computer-controlled calibrations and programmable gas concentrations. A 6-port manifold was used to direct the diluter output to point A, B, C, or D. Figure 12 depicts the zero and span checks of gas analyzers of point D at the Missouri site. A bubbler was not used during the calibration shown in Figure 12, which caused a downward drift after equilibrium was attained. Utilizing span CO₂ gas with 2.5% methane serves as an alternative to moisture addition.

An additional calibration technique used in the APECAB project was the introduction of calibration gas into one of the sampling lines (Figure 11). This method required one manual 3-way valve and a bubbler. The manual 3-way valve was placed in a sample line where it enters the OSIS before the solenoid. This 3-way valve either allows flow from either the barn or gas cylinders. The bubbler indicated adequate bypass flow and provided moisture needed for stable
calibration of the CO₂ analyzer. Tubing was manually connected to the required gas cylinder. A bypass vent for excess flow was required in each case. The sample probe itself was the bypass for points C and D. With a diluter flow of 5 L/min and a sampling flow rate of 4 L/min, 1 L/min flowed from the point of injection, out, through the sampling probe. The bypass flow was checked at the probe with a flow meter to confirm sufficient bypass flow. At point B, one of the solenoids was opened to create a bypass, back through the sampling line. A small manifold M4 was used for point A calibrations. A flag indicating calibration time, recorded by the data acquisition program, assisted in data processing. This flag activated when the DAQ software was in calibration mode, either automatically or manually. Bimonthly, in the APECAB project, a bag of calibration span gas and a bag of zero gas were manually introduced into the filtered end of a sampling tube (point D in Figure 11). The results were compared with calibration point B. If the difference in span concentrations between points B and D was more than 5%, then maintenance was needed to correct the problem (U.S. EPA, 1998).

The NH₃ analyzer was challenged with zero air, an NH₃ span gas (dual-certified by NIST-traceable gravimetric formulation and analysis based on vendor reference standard), and a NIST-traceable NO span gas. The NH₃ calibration was conducted every 2 to 7 d, whereas the NO calibration was conducted every 1 to 3 months, as a maintenance check, to calculate converter efficiency. The H₂S analyzer was challenged with zero air, a known concentration of H₂S span gas (weekly), and a known concentration of sulfur dioxide (SO₂) span gas (every 1 to 3 mo). The H₂S and SO₂ calibration gases were certified with NIST-traceable analyses. For the NMHC analysis, selection of an appropriate calibration gas was critical for obtaining an accurate measure of total NMHC. Because the response of the FID is different per unit mass of carbon, the response of the gas used to calibrate it should be as close as possible to the response of the NMHCs being measured. N-hexane (2.0 ppm) was selected as the most appropriate calibration gas for the CAPESH study. The CO₂ analyzer was challenged with zero air with 2.5% methane, and a known concentration of NIST-traceable CO₂ span gas.

Gas Density Measurements

The temperature and humidity of exhaust air, along with barometric pressure, were needed for precise volume correction to standard conditions. Copper-constantan thermocouples (Type T)
were used to sense temperature at the exhaust sampling points. Thermocouples were calibrated before and after the 15-month collection period. Spot checks of each sensor occurred every 3 months using a constant-temperature bath. An electronic RH/temp transmitter (Model HMW61, Vaisala, Woburn, MA) that was housed in a NEMA 4 enclosure monitored temperature and relative humidity at a representative exhaust location in each barn. This RH/temp transmitter used a HUMICAP sensor unit with ±2% accuracy between 0 and 90% RH and ±3% accuracy between 90 and 100% RH.

A water-bath and two precision ASTM mercury-in-glass thermometers (-8 to 32 °C and 25 to 55 °C, 0.1 °C precision) were used for calibration. A salt calibrator kit (Model MK1520000A01000, Vaisala, Woburn, MA) was used to calibrate the capacitance-type rh/temp sensors prior to beginning the study, and every 3 months thereafter. A portable RH/temp probe (Model HMP46, Vaisala, Woburn, MA) with an indicator (Model HM141, Vaisala, Woburn, MA) was used as a NIST-transfer device, to check the RH/temp transmitters and the thermocouples every three months. Atmospheric pressure was monitored with a barometric pressure transducer and compared with that measured at the nearest weather station.

CONCLUSIONS
Gas concentration measurements at animal facilities face many management and technical challenges. A comprehensive measurement system as described in this paper consisted of sampling probes at different locations, an external gas sampling system, various measurement instruments, and a computer-based controller. Operation of such a system, and each of its components, are exposed to many sources of errors. Concentration calculation and conversion also depend on reliable temperature and pressure data. A quality-assured gas concentration measurement is subject to limitations of currently available technologies, materials, and budget. However, careful design of the system, selection of quality materials, and regular and effective maintenance and calibration minimizes the errors. Quality assurance and quality control throughout each stage of research is critical for research management to achieve data quality objectives.
ACKNOWLEDGMENTS
Support from the U.S. Department of Agriculture, the U.S. Environmental Protection Agency, and Premium Standard Farms is acknowledged. The contributions of Rob Dobson, Sustainable Environmental Solutions; Bruce Harris, U.S.E.P.A.; and Cary Secrest, U.S.E.P.A. are acknowledged for their contributions to the development of the quality assurance project plan.

ABBREVIATIONS AND ACRONYMS
APECAB = study entitled “Air Pollutant Emissions from Confined Animal Buildings”
CAPESH = study entitled “Control of Air Pollutant Emissions from Swine Housing”
CEM = continuous emission monitoring
FEP = grade of Teflon
GSS = gas sampling system
NMHCs = non-methane hydrocarbons
OSIS = on-site instrument shelter
PFA = grade of Teflon
QA = quality assurance
QC = quality control
SLG = sampling location group

REFERENCES


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Dr. Albert J. Heber, Dr. Ji-Qin Ni, and Dr. Teng T. Lim are professor, senior research associate, and postdoctoral research associate at Purdue University respectively. Dr. Amy Millmier is an Extension Assistant Professor at the University of Missouri. Dr. Jacek A. Koziel is an assistant professor at the Texas Agricultural Experiment Station. Pei-Chun Tao is a graduate student at Purdue University. Dr. David B. Beasley is a professor at the North Carolina State University.
Dr. Steven Hoff is an associate professor at the Iowa State University. Dr. Larry D. Jacobson is a professor at the University of Minnesota. Dr. Yuanhui Zhang is an associate professor at the University of Illinois at Urbana-Champaign. Dr. Heber serves as the CAPESH project director and is codirector with Dr. Jacobson of the APECAB project. More information about the APECAB project can be found at: http://manure.coafes.umn.edu/apecab/index.html.
### Table 1. Potential gas concentration measurement errors inside livestock barns and possible solutions (Hartung et al., 2001).

<table>
<thead>
<tr>
<th>Sources of errors</th>
<th>Causes or Solutions</th>
</tr>
</thead>
<tbody>
<tr>
<td>Diurnal variation</td>
<td>Repeated measurements over 24 hr</td>
</tr>
<tr>
<td>Seasonal variation</td>
<td>Long-period tests across seasons</td>
</tr>
<tr>
<td>Spatial variation</td>
<td>Sample at multiple locations</td>
</tr>
<tr>
<td>Adsorption and desorption</td>
<td>Reduce wetted surface areas. Use bypass pumping. Use Teflon.</td>
</tr>
<tr>
<td>Condensation in sample lines</td>
<td>Keep sample transfer lines above dew point temperature</td>
</tr>
<tr>
<td>Leaks in sampling system</td>
<td>Conduct leak tests and eliminate leaks</td>
</tr>
<tr>
<td>Dust in sampling system</td>
<td>Filter dust at sampling probe and sampling line inlet</td>
</tr>
<tr>
<td>Human operational errors</td>
<td>Proper training, oversight and standard operating procedures, record keeping</td>
</tr>
<tr>
<td>Calibration gas</td>
<td>Select reliable gas provider, dual analysis, and internal checks</td>
</tr>
<tr>
<td>Calibration procedure</td>
<td>Input gas at sample probe in replicated multipoint calibrations</td>
</tr>
<tr>
<td>Poor sensitivity and precision</td>
<td>Proper calibration and maintenance</td>
</tr>
<tr>
<td>Poor frequency response</td>
<td>Proper selection, calibration and maintenance of analyzers</td>
</tr>
<tr>
<td>Interference of particles</td>
<td>Filtration of particles from gas sampling lines</td>
</tr>
<tr>
<td>Interferences of other gases</td>
<td>Analyzer selection and calibration</td>
</tr>
<tr>
<td>Operation of measurement</td>
<td>Human errors</td>
</tr>
<tr>
<td>Temperature data</td>
<td>Use accurate sensors. Conduct scheduled calibrations</td>
</tr>
<tr>
<td>Atmospheric pressure data</td>
<td>Use accurate sensors. Conduct scheduled calibrations</td>
</tr>
<tr>
<td>Data rounding</td>
<td>Increase resolution of recorded data</td>
</tr>
</tbody>
</table>

Adapted from Ni and Heber, 2001
Table 2. Characteristics of test sites and barns.

<table>
<thead>
<tr>
<th>Location by state</th>
<th>NC</th>
<th>IN</th>
<th>MN</th>
<th>IL</th>
<th>IA</th>
<th>TX</th>
<th>MO</th>
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</thead>
<tbody>
<tr>
<td>Livestock type</td>
<td>Broiler chickens</td>
<td>Layer hens</td>
<td>Swine gestation</td>
<td>Swine farrowing</td>
<td>Swine finishing</td>
<td>Swine finishing</td>
<td>Swine finishing</td>
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<tr>
<td>Inventory†</td>
<td>22,000</td>
<td>250,000</td>
<td>929/512</td>
<td>56</td>
<td>960</td>
<td>1,080</td>
<td>1,100</td>
</tr>
<tr>
<td>Average mass, kg</td>
<td>1.0</td>
<td>1.8</td>
<td>200</td>
<td>200</td>
<td>68</td>
<td>68</td>
<td>68</td>
</tr>
<tr>
<td>Animal occupation, d</td>
<td>63</td>
<td>365</td>
<td>1 wk</td>
<td>21</td>
<td>140</td>
<td>140</td>
<td>140</td>
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<tr>
<td># barns at site</td>
<td>4</td>
<td>16</td>
<td>2</td>
<td>2</td>
<td>4</td>
<td>5</td>
<td>8</td>
</tr>
<tr>
<td>Barn type</td>
<td>litter</td>
<td>HR</td>
<td>PPR</td>
<td>PP</td>
<td>DP</td>
<td>PPR</td>
<td>Flush</td>
</tr>
<tr>
<td>Orientation</td>
<td>NE-SW</td>
<td>N-S</td>
<td>N-S</td>
<td>N-S</td>
<td>E-W</td>
<td>E-W</td>
<td>E-W</td>
</tr>
<tr>
<td>Distance to site, km</td>
<td>120</td>
<td>69</td>
<td>160</td>
<td>96</td>
<td>29</td>
<td>160</td>
<td>94</td>
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<tr>
<td>Shower in/out†?</td>
<td>N</td>
<td>N</td>
<td>Y</td>
<td>Y</td>
<td>N</td>
<td>Y</td>
<td>Y</td>
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<tr>
<td>Barn width, m</td>
<td>12.8</td>
<td>30.5</td>
<td>14.6</td>
<td>18.0</td>
<td>12.5</td>
<td>12.7</td>
<td>13.2</td>
</tr>
<tr>
<td>Barn length, m</td>
<td>152.4</td>
<td>181.4</td>
<td>77.4</td>
<td>22.5</td>
<td>58.5</td>
<td>72.0</td>
<td>61.0</td>
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<tr>
<td>Barn area, m²</td>
<td>1,952</td>
<td>5,613</td>
<td>1,133</td>
<td>405</td>
<td>2,400</td>
<td>910</td>
<td>806</td>
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<tr>
<td>Ridge height, m</td>
<td>3.2</td>
<td>11.6</td>
<td>4.9</td>
<td>7.6</td>
<td>4.6</td>
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<tr>
<td>Sidewall height, m</td>
<td>2.20</td>
<td>6.40</td>
<td>2.29</td>
<td>3.05</td>
<td>2.44</td>
<td>2.44</td>
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<tr>
<td>Barn spacing, m</td>
<td>18.3</td>
<td>22.9</td>
<td>9.2</td>
<td>0</td>
<td>18.3</td>
<td>15.2</td>
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<tr>
<td>Manure storage, d</td>
<td>730</td>
<td>730</td>
<td>400</td>
<td>21</td>
<td>365</td>
<td>7</td>
<td>0.25</td>
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<tr>
<td>Outdoor storage</td>
<td>none</td>
<td>none</td>
<td>basin</td>
<td>none*</td>
<td>none</td>
<td>lagoon</td>
<td>lagoon</td>
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<tr>
<td>Number air inlets†</td>
<td>48</td>
<td>10</td>
<td>14</td>
<td>8</td>
<td>9</td>
<td>20</td>
<td>20</td>
</tr>
<tr>
<td>Inlet type</td>
<td>slot/EP</td>
<td>slot</td>
<td>CCB</td>
<td>CCB</td>
<td>CCB</td>
<td>CCB</td>
<td>CCB</td>
</tr>
<tr>
<td>Inlet control method</td>
<td>∆p</td>
<td>∆p</td>
<td>∆p</td>
<td>gravity</td>
<td>∆p</td>
<td>∆p</td>
<td>gravity</td>
</tr>
<tr>
<td>Controls vendor</td>
<td>HH</td>
<td>AE</td>
<td>AV</td>
<td>MF</td>
<td>VF</td>
<td>AS</td>
<td>JC</td>
</tr>
<tr>
<td>Number of fans†</td>
<td>13</td>
<td>75</td>
<td>6</td>
<td>4</td>
<td>8</td>
<td>5</td>
<td>5</td>
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<tr>
<td># variable speed fans</td>
<td>0</td>
<td>0</td>
<td>1</td>
<td>2</td>
<td>4</td>
<td>2</td>
<td>1</td>
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<tr>
<td>Largest fan dia., cm</td>
<td>122</td>
<td>122</td>
<td>122</td>
<td>122</td>
<td>122</td>
<td>122</td>
<td>122</td>
</tr>
<tr>
<td>Smallest fan dia., cm</td>
<td>91</td>
<td>122</td>
<td>91</td>
<td>46</td>
<td>46</td>
<td>91</td>
<td>91</td>
</tr>
<tr>
<td>Fan manufacturer</td>
<td>HH/DA</td>
<td>AT</td>
<td>AV</td>
<td>MF</td>
<td>MF</td>
<td>AS</td>
<td>AS</td>
</tr>
<tr>
<td># ventilation stages‡</td>
<td>7</td>
<td>9</td>
<td>6</td>
<td>4</td>
<td>7</td>
<td>5</td>
<td>5</td>
</tr>
<tr>
<td># temperature sensors†</td>
<td>3</td>
<td>15</td>
<td>1</td>
<td>1</td>
<td>2</td>
<td>2</td>
<td>2</td>
</tr>
<tr>
<td>Artificial heating</td>
<td>Y</td>
<td>N</td>
<td>Y</td>
<td>Y</td>
<td>Y</td>
<td>Y</td>
<td>Y</td>
</tr>
<tr>
<td>Number of inlet SLG†</td>
<td>1/2</td>
<td>0.5††</td>
<td>2</td>
<td>2</td>
<td>2</td>
<td>2</td>
<td>0.5††</td>
</tr>
<tr>
<td>Number exhaust SLG†</td>
<td>4/5</td>
<td>4</td>
<td>2</td>
<td>2</td>
<td>3</td>
<td>2</td>
<td>1</td>
</tr>
<tr>
<td>No. emission streams</td>
<td>1/4</td>
<td>4</td>
<td>1</td>
<td>2</td>
<td>3</td>
<td>2</td>
<td>1</td>
</tr>
<tr>
<td>Internet service type</td>
<td>phone</td>
<td>wireless</td>
<td>WDSL</td>
<td>phone</td>
<td>phone</td>
<td>satellite</td>
<td>DSL</td>
</tr>
<tr>
<td>Start date in 2002</td>
<td>4/1</td>
<td>12/1</td>
<td>8/28</td>
<td>11/15</td>
<td>8/15</td>
<td>10/10</td>
<td>8/28</td>
</tr>
</tbody>
</table>

*manure stored in deep-pit of adjacent barn
†per barn or room
‡includes continuous winter fans as the first stage
††one sampling probe located between the barns represents inlet air for both barns
AE= Automated Environments, AS= Airstream, AT=Aerotech, AV=Aerovent, CCB=center-ceiling baffled inlet, DP=deep pit, EP=evaporative pad, Flush = shallow-pit with recycle flush, HR=high rise, HH = Hired Hand, MF=Multifan, PP=Pull-plug manure pit, PPR=Pull-plug manure pit with recharge, SLG=sample location groups, SK=sprinkler system, tun=tunnel ventilation, VF=Varifan, WDSL = wireless DSL.
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