A Quality Assurance Project Plan for Monitoring Gaseous and Particulate Matter Emissions from Broiler Housing Lara B. Moody, Hong Li, Robert T. Burns, Hongwei Xin, Richard S. Gates, Steven J. Hoff, Doug Overhults. 2008. Appendices J-T, pp. 199-258 in A Quality Assurance Project Plan for Monitoring Gaseous and Particulate Matter Emissions. Copyright © American Society of Agricultural and Biological Engineers. ASABE # 913C0708e.

Appendix J: SOP of Reporting and Calculation of Contaminant Concentrations, Ventilation and Emissions

This SOP gives instructions for calculating and reporting gas (ammonia, carbon dioxide, hydrogen sulfide, non-methane hydrocarbon) and dust ($PM_{2.5}$, PM_{10} , TSP) concentrations and barn emission rates.

Concentration Defined

Concentration is broadly defined as the fraction of a constituent of interest contained within a sample. Concentration of gaseous contaminants are generally reported in volumetric, massbased, or mixed units (see next section). Concentrations of particulates can be provided as number of particles in a sample or mass of particulates in a certain size range in a mass of sample gas.

Gaseous Contaminants—Units of Measurement²

Concentration of gaseous contaminants may be expressed in volumetric, mass-based, or mixed units fractions. The first two are dimensionless except for any scale factor, whereas mixed units fractions are typically expressed as mass of contaminant per unit volume of sample mixture, e.g. $mg m^{-3}$.

Volumetric concentration is generally expressed in parts per million (ppm) or parts per billion (ppb), defined as the parts of contaminant per million or billion parts of air by volume (1000 ppb = 1ppm).

Mass-based concentration is expressed in either mg kg⁻¹ or μ g kg⁻¹, with the numerator referring to the mass of contaminant and the denominator to the mass of sample air.

Mixed-unit concentration is most convenient for this work, and is generally expressed as either mg m⁻³ or μ g m⁻³. While this is technically a mixed-unit expression for concentration, it is often called "mass concentration", and we adopt this naming convention in this SOP. It is convenient because when multiplied by volumetric flow rate of air, an emission rate is directly determined.

Conversion between volumetric $[C]_V$ (ppm) and mass $[C]_M$ (mg m⁻³) concentrations, assuming the mixture behaves as an ideal gas, is¹:

$$[C]_{V} = 8.309T/(Mp) [C]_{M}$$
(1)

$$[C]_{M} = 0.1204 [C]_{V} Mp/T$$
(2)

where: p = sample mixture pressure, kPa

² Air Contaminants. Chapter 12. ASHRAE HOF. 2005. American Society of Heating Ventilation and Air-Conditioning Engineers, Atlanta GA.

T = sample mixture temperature, K M = relative molar mass of contaminant, dimensionless

Gaseous Contaminants—Standard Conditions

The relation between $[C]_M$ and $[C]_V$ for standard conditions of 25°C and 101.325 kPa, is given by:

$$[C]_{M} = (M/24.45) [C]_{V}$$
(3)

For ammonia, carbon dioxide, hydrogen sulfide, and methane; the relative molecular masses are 17.03, 44.01, 34.08, and 16.04, respectively. From equation (3) the relation between standard concentration on mass and volumetric basis are:

$$\begin{split} [C]_{M,NH3} &= 0.6965 \, [C]_{V,NH3} \\ [C]_{M,CO2} &= 1.8 \, [C]_{V,CO2} \\ [C]_{M,H2S} &= 1.3939 \, [C]_{V,H2S} \\ [C]_{M,CH4} &= 0.6560 [C]_{V,CH4} \end{split}$$

Gaseous Contaminants—Effects of Temperature and Pressure

A volumetric concentration measurement (ppm) taken at non-standard conditions may be converted to mass concentration (mg m^{-3}) by equation (2) for a given pressure, p, and absolute temperature, T.

Particulate Contaminants—Units of Measurement

Concentration of particulate contaminants may be expressed in mixed units (mass of particulate in a unit volume of sample air), or as a particle count per unit volume of sample air. If the latter is used, the particle count is understood to be within a particle size range, e.g. up to 2.5 μ m or 10 μ m (PM_{2.5} and PM₁₀). Total suspended solids may be expressed as either particle count or particle mass per unit volume of sample air. In this SOP, we shall always use mixed units for particulate concentrations, i.e., mg m⁻³.

Ventilation Rates—Units of Measurement

Fan ventilation rates (standard $m^3 s^{-1}$, or $sm^3 s^{-1}$) for each running fan are determined from building static pressure difference (SP, Pa) and the calibration equation for the fan, as follows:

$$Q_{FAN} = a + b SP$$

where the parameters a,b are different for each fan and are obtained from regression of the FANS calibration data explained elsewhere in this SOP.

Building ventilation rate, Q'_e, is determined by summing all fans that are running at any time. In the event that large spatial variations are noted, building ventilation rate can be broken into

representative amounts near each sampling location, typically two values in the broiler housing, e.g. Q'e₁ and Q'e₂.

The relations between actual (Q), moist standard (Q'), and dry standard (Q'') volumetric flow rates are:

$$Q'' = (1-W)Q' = (1-W)Q(p/p')(T'/T)$$
(4)

where:	W	=	humidity ratio of air, kg H2O per kg dry air
		=	0.62198 p / (p-pv)
	$\mathbf{P}_{\mathbf{w}}$	=	water vapor pressure (kPa)
		=	(rh/100)p _{v,sat}
	rh	=	air relative humidity,%
	p_{ws}	=	saturation water vapor pressure
	р	=	actual pressure (kPa)
	p'	=	standard pressure, 101.325 kPa
	Т	=	actual absolute temperature, K
	T'	=	standard absolute temperature, K
	Q	=	actual (moist) volumetric flow, m ³ s ⁻¹
	Q'	=	moist standard volumetric flow, sm ³ s ⁻¹
	Q"	=	dry standard volumetric flow, dsm ³ s ⁻¹

Emission Rates—Units of Measurement

Emission rate (ER) of a gaseous or particulate contaminant is expressed as a mass flow per unit time, e.g. g NH_3 s⁻¹. It may be further refined on a per live-weight, per animal or per animal-unit basis. Consideration should be given to avoid expressing ER on a time interval that is shorter than the minimum sampling frequency used to obtain the data. Thus, for example, if several minutes are required to acquire concentration data then it is unrealistic to report ER on a mass per second basis.

Emission Rates—Calculations

Emission rate (ER) is the difference in mass flow between all inlets and outlets for the contaminant of interest. Mass flow of contaminant entering a building can be expressed as the background volumetric concentration $[C]_{Vi}$ multiplied by the incoming volumetric flow rate, Q_i . Similarly, the exhaust mass flow of contaminant is the product of exhaust volumetric concentration $[C]_{Ve}$ multiplied by the exhaust volumetric flow rate, Q_e . Note that volumetric flow rates Q_i and Q_e are typically not equal, since air density differences usually exist between inlet and outlets. Mass flow rate of ventilation air, M_i and M_o , are equal. Equation (5) expresses the general definition of emission rate:

$$E = Q'_{e}[C]_{Me} - Q'_{i}[C]_{Mi}$$
(5)

In equation (5), the volumetric flow rates Q'_e and Q'_i may be either actual flow rate, or standardized flow rates (either moist standard = sm³ s⁻¹ or dry standard = dsm³ s⁻¹). Moist

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standard conditions are most appropriate. The mass concentrations of outside and inside samples should be adjusted to standard temperature and pressure, as per equation (4).

In equation (5), the volume units used for volumetric flow rate (denominator) and mass concentration (denominator) must match. For example, non-standard moist air, standard moist air, or standard dry air would be the consistent units to use. Further, typically Q'_e is measured (see SOP for airflow measurement), but Q'_i must be computed from continuity:

$$Q_i / \upsilon_i = Q_e / \upsilon_e$$

$$Q'i = (\upsilon_i / \upsilon_e) Q'_e$$
(6)

or,

where v_i , v_o are inside air and outside air specific volumes, m^3 moist air per kg dry air. Specific volume may be calculated from air density (kg moist air per m³ dry air, or kg ma per kg da) and humidity ratio W (kg H₂O per kg da), by:

$$\upsilon = (1 + W) / \rho \tag{7}$$

Summary of Measurements and Calculations for Emission Rate

Measurements required to compute ER include:

- 1. Interior and exterior air state points: dry-bulb temperature, relative humidity
- 2. Interior and exterior mass volumetric concentrations
- 3. Building static pressure differences, and atmospheric pressure

Intermediate calculations to compute ER include:

- 1. Interior and exterior saturation and partial water vapor pressure, humidity ratio, air density, specific volume (from equations 4 and 7)
- 2. Exhaust standard ventilation rate, Q'e (from FANS calibration and measured static pressure)
- 3. Incoming standard volumetric ventilation rate, Q'_i (from equation 6)

Combining equations (5)-(7), compute emission rate ER from:

$$ER = Q'_e ([C]_{Me} - (v_i / v_e) [C]_{Mi}) \quad (mg \text{ contaminant } s^{-1})$$
(8)

Adjust ER time basis to reflect issues described in next section.

Concentration Data Extraction, Averaging and Interpolation

Gas Concentration Extraction and Averaging

When analyzing each of the house air samples, four 30-second measurement cycles by the ammonia analyzer will be performed to ensure attainment of 97% or better of the expected concentration value. If fans at all three sampling locations are running, the time interval of a complete sampling cycle will be $120 \times x = 360$ seconds. If SW3 and/or TF1 are not running,

their sample analysis will be skipped, and the sampling will go back to SW1 or fresh/background air. Airflow rates corresponding to the measured concentrations will be used in the calculation of the overall house emission rate. Since compositions of the background air are much more stable than the house air, it will be sampled only once every 2 hours. As a result of the larger step change in ammonia concentration between the house air and background air, a longer sampling time (i.e., 5 minutes) will be used to allow full stabilization of the analyzer readings. Only the concentration readings at the end of the sampling cycle will be considered as valid measurements.

Gas Concentration Interpolation

When there is only 1 valid reading per location during a 360 sec sampling cycle. To account for potential concentration changes during this period, linear interpolation between the two adjacent readings of the same location will be performed to determine the concentrations in between.

Complete Data Sets

To avoid errors introduced into calculated average values as a result of partial data days that result in biased time weights, only complete-data days (CDD) that include over 75% valid data should be used for calculating average daily means (ADM). Similarly, hourly averages should be reported only if over 75% of the data during that hour is valid and monthly averages are reported only if over 75% of the days are valid.

Quantity Names and Terminology

Reading: One data point read by LabVIEW. It is not necessarily recorded by LabVIEW.

Data record: A single data point recorded in data files by the LabView data acquisition program. A data record consists of an average of 1-s readings collected over the data recording interval. LabVIEW takes a reading every second and writes a data file. LabVIEW also records an average of 30 readings every 30 s and records that in another data file.

Daily mean: Average of all valid and interpolated data records during CDD.

Average daily means (ADM): Average of valid daily means. It can be obtained over a month, a season, or a year.

Overall test mean: Average of all valid data over the entire test.

Appendix K: SOP of Model 101E UV Fluorescence H₂S Analyzer

Introduction

The M101E UV Fluorescence H_2S Analyzer is a microprocessor controlled analyzer that determines the concentration of hydrogen sulfide (H_2S) in a sample gas drawn through the instrument. It requires that sample and calibration gases be supplied at ambient atmospheric pressure in order to establish a constant gas flow through the sample chamber. The H_2S in the sample gas is converted into SO₂ which is then exposed to ultraviolet light causing the SO₂ to become excited (SO₂*). As these SO₂* molecules decay back into SO₂ they fluoresce. The instrument measures the amount of fluorescence to determine the amount of SO₂ present in the sample chamber and by inference the amount of H_2S present in the sample gas.

The M101E H₂S analyzer is basically a SO₂ analyzer with an H₂S \rightarrow SO₂ conversion stage inserted into the gas stream before the sample gas enters the sample chamber. The H₂S to SO₂ converter receives sample gas from which the SO₂ has been removed by a scrubber. Once the naturally occurring SO₂ is removed from the sample gas, the special converter changes the H₂S in the sample stream to SO₂ using a high-temperature catalytic oxidation. The chemical process is:

$$2H_2S + 3O_2 \longrightarrow 2H_2O + 2SO_2$$

The physical principle of the M101E's measurement method relies on the fluorescence that occurs when Sulfur dioxide (SO₂) is excited by ultraviolet light with wavelengths in the range of 190 nm - 230 nm. This reaction is a two-step process. The first stage occurs when SO₂ molecules are struck by ultraviolet photons of the appropriate wavelength (19 nm - 230 nm). The SO₂ retains some excess energy that causes one of the electrons of the SO₂ molecule to move to a higher energy orbital state. In the case of the Model 101E, a band pass filter between the source of the UV light and the affected gas limits the wavelength of the UV light to approximately 214 nanometers (nm).

$$SO_2 + hv_{214nm} \xrightarrow{Ia} SO2 *$$

The second stage of this reaction occurs after the SO_2 reaches its excited state (SO_2^*). Because the system will seek the lowest available stable energy state, the SO_2^* molecule quickly returns and the wavelength of this fluoresced light is also in the ultraviolet band but at a longer (lower energy) wavelength centered at 330 nm.

$$SO_2 * \longrightarrow SO_2 + hv_{330nm}$$

The linearity of the 101E was checked. Figure K.1 shows the linearity results for the two analyzer (SN: 965 and 966) accuracy checks. For each check, a linear regression was calculated from API 101E response versus the nominal H_2S gas standard concentration over the range of 0 to 500 ppb.

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For 965, the slope of the regression line was 1.05, with an intercept of -8.58 and r^2 value of 0.9999. For 965, the slope of the regression line was 0.996, with an intercept of -8.23 and r^2 value of 0.9999. Over the range of concentrations tested (0 to 500 ppb H₂S), the API 101E demonstrated a high degree of linearity.

The response time of the analyzer to step changes in gas concentrations was tested (Figure K.2). The analyzer was challenged with two H₂S calibration span gases, 44 ppb and 93 ppb H₂S respectively in a N₂ balance ($\pm 2\%$ accuracy) (Matheson Gas Products, Inc., Montgomeryville, PA). Response time was determined from the amount of time required for the Model 101E to reach 95% of the change in response during the zero air to 44 ppb H₂S span gas, 0 to 93 ppb, and 44 to 93 ppb shown in Figure K.2. The response time was approximately 75 seconds.

Start Up

After electrical and pneumatic connections are made, turn on the power switch on the front panel. The exhaust and PMT cooler fans should start. The display should immediately display a single, horizontal dash in the upper left corner of the display. This will last approximately 30 seconds while the CPU loads the operating system.

Once the CPU has completed this activity it will begin loading the analyzer firmware and configuration data. During this process, a string of messages will appear on the analyzer's front panel display:



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The "fault", red LED will be blinking. Then push the "CLR" key to clear the "SYSTEM RESET" message. The M101E requires about 60 minutes warm-up time before reliable H_2S measurements can be taken. If the warning messages persist after 60 minutes, investigate their cause using the troubleshooting guidelines in Chapter 11 of the 101E User's Manual.



Figure K.2. API response time.

Functional Check

After the analyzer's components have warmed up for at least 30 minutes, verify that the software properly supports any hardware options that were installed. Check to make sure that the analyzer is functioning within allowable operating parameters, using the values listed in the manufactory data sheet with the operation manual. To view the current values of these parameters press the following key sequence on the front panel.



Changing the Sample Particulate Filter

The particulate filter should be inspected often for signs of plugging or excess dirt. It should be replaced according to the service interval in Table 9-1 of manual (every week) even without obvious signs of dirt. Filters with 1 and 5 μ m pore size can clog up while retaining a clean look. Handle the filter and the wetted surfaces of the filter housing with gloves and tweezers. Do not touch any part of the housing, filter element, PTFE retaining ring, glass cover, or O-ring with bare hands.

- 1. Turn OFF the analyzer to prevent drawing debris into the sample line.
- 2. Open the M101E's hinged front panel and unscrew the knurled retaining ring of the filter assembly.
- 3. Carefully remove the retaining ring, glass window, PTFE O-ring, and filter element.
- 4. Replace the filter element, carefully centering it in the bottom of the holder.
- 5. Re-install the PTFE O-ring with the notches facing up, the glass cover, then screw on the hold-down ring and hand-tighten the assembly. Inspect the (visible) seal between the edge of the glass window and the o-ring to assure proper gas tightness.
- 6. Re-start the analyzer.

Calibration Procedure



Step 1: Set/verify the analog output reporting range of the 101E.

Step 2: Set the expected H₂S span gas concentration.



SAMPLE RANGE = 500.0 PPB H2S =XXX.X Set the Display to show the H2S STB test function. This function calculates the < TST TST > CAL SETUP stability of the H₂S x measurement SAMPLE H2S STB=X.XXX PPB H2S =X XXX < TST TST > CAL SETUP ACTION: Allow zero gas to enter the sample port at the rear of the instrument. Wait until H2S STB falls below 0.5 ppb. This may take several minutes. M-P CAL H2S STB=X.XXX PPB SO2 =X.XXX < TST TST > CAL SETUP M-P CAL H2S STB=X.XXX PPB SO2 =X.XXX < TST TST > ZERO CONC EXIT Press ENTR to changes the M-P CAL H2S STB=X.XXX PPB SO2 =X.XXX OFFSET & SLOPE values for the SO₂ measurements. < TST TST > ENTR CONC Press EXIT to leave the calibration unchanged and return to the previous menu. ACTION: Allow span gas to enter the sample port at the rear of the instrument. The value of H2S STB may jump significantly. Wait until it falls back below 0.5 ppb. The SPAN key now This may take several M-P CAL H2S STB=X.XXX PPB H2S =X.XXX appears during the minutes. transition from zero to SPAN < TST TST > CONC EXIT span. You may see both keys. If either the ZERO or Press ENTR to change the offset & SPAN buttons fail to slope values for the H₂S M-P CAL RANGE = 500.0 PPB H2S =X.XXX appear see Section 11 measurements. for troubleshooting tips. Press EXIT to leave the calibration <TST TST > ENTR SPAN CONC unchanged and return to the previous menu. M-P CAL RANGE = 500.0 PPB H2S =X.XXX EXIT returns to the main SAMPLE display < TST TST > ENTR CONC EXIT

Step 3: Perform the zero/span calibration procedure.

Manufacturer Contact Information

Teledyne Advanced Pollution Instrumentation 9480 Carroll Park Drive San Diego, CA 92121-5201 USA Phone: 858-657-9800 800-324-5190 Fax: 858-657-9816 Email: api-sales@teledyne.com Website: http://www.teledyne-api.com/

Reference

Instruction Manual of Model 101E UV Fluorescence H_2S Analyzer, Rev. A2, 26 September, 2005.

Date of Calibration:		Calibrated by:	
Time	Items	Unit =	Notes
	Ambient pressure, (mm Hg)		
: :	Sample airflow (L/min)		
: :	Zero air applied		Cylinder P: psi
: :	H ₂ S Reading		
: :	H_2S (ppm) applied		Cylinder P: psi
: :	H ₂ S Reading		
: :	New Calibration	Yes/ _{No}	
: :	Zero air applied		
	H ₂ S applied		
: :	Finish calibration	^{Yes} / _{No}	
: :	Time switch off		
: :	Connect analyzer back to sampling system.		
Notes:			

Maintenance/Calibration Record Sheet for API 101E H₂S Analyzer

Appendix L: SOP of VIG Model 200 (Methane/Non-Methane/Total Hydrocarbon) Analyzer

Introduction

The methane/non-methane/total hydrocarbon analyzer VIG 200 measures concentrations of a wide variety of hydrocarbons in gas mixtures and in air, and can separate the methane component from the non-methane component by using a GC column. This makes VIG-200 ideal for stack monitoring, laboratory or process control use. The model 200 uses column technology to separate methane and non-methane from total hydrocarbons (THC). When a small sample is fed though a column, the column separates each components of the THC one at a time, lightest first and heaviest last. The lightest hydrocarbon is methane. A small sample is sent through the column, waits for the methane to come out, and measures the methane; the rest of the sample is sent backward through the column, is measured again, and is called non-methane. The measurement is achieved by the use of a FID (flame ionization detector) coupled with a very precise amplifier. The FID injects the gas into a hydrogen-based flame. The flame helps to break the electrons from their orbit, along with a high voltage surrounding the flame. The voltage forces the electrons to jump from the flame on to the electrode giving a measurable medium. The amplifier takes this information and makes it linear and in scale.



Installation

Fuel: Hydrogen (100%), Ultra High Purity Grade @ 18 psi.
Combustion Air: Hydrocarbon free, oil free, dry air @ 18 psi. (From Zero Air Generator)
Zero Air: Zero air Ultra High Purity Grade @ 9-10 psi.
Compressed air: Oil free, dry, Compressed air@ 55 psi. (From Air Compressor)
Calibration gas: Methane/Propane mixture balanced in air @ 9-10 psi.
Carrier gas: Nitrogen Ultra High Purity Grade @ 30 psi.

Start Up

- Connect compressed air to the unit and set the incoming pressure to 55 psi.
- Press "Power" switch. Connect nitrogen to the unit at the carrier gas port and set the carrier gas pressure on the front panel to 22 psi.
- Press any key to continue.
- Find the "Oven" key. If no, push the "More".
- Set the desired oven temperature to 275°F by turning front panel knob.
- Connect all required gases to the proper ports on the rear panel.
- Go the "Burner 1" and Burner 2". Press the "Ignite". Make sure that "Auto" is flashing. If not, press the "Auto" key.
- After the alarm lamp on the front panel goes out, check and adjust the fuel, combustion air, carrier gas, and compressed air to the desired value in the Table 1 on the VIG 200 user manual.

Warming Up the Monitor

A period of 6 hours is suggested to warm up the analyzer before a calibration task is started. This will reduce the time required for calibration.

Calibration

Before calibration, make sure the display and output values are the peak value.

- Press "Main" / "More" / "Menu" until "Modes" is displayed.
- Press "Modes".
- Press "Peak" for Display and Output.
- On the front panel of the Labview program, click the "Manually control" and put all four SVs (solenoid valves) on "OFF".

Zero Gas Calibration

- 1. Close regulator valve on the zero gas cylinder.
- 2. Open main valve on zero gas cylinder.
- 3. Insert the 1/4" ID tubing (from the gas cylinder) into the inlet of the flow meter mounting on the side of instruments rack.
- 4. Adjust regulator valve until vent airflow is about 5 L/min (read from bottom of ball of the vent monitoring flow meter). This provides a little extra zero air to the analyzers and keeps the pressure inside the manifold close to the atmospheric pressure.
- 5. Flow Zero gas for 2 minutes.
- 6. Press "Main" / "More" / "Menu" until "GC" is displayed and Press "GC". Press "Single".
- 7. Wait for about 3 minutes until the CH_4 and Residual analysis is finished.
- 8. If the CH₄ or Residual reading is not in the range of ± 0.1 ppm, the potentiometer needs to be adjusted.
 - i. Press "Setup".
 - ii. Press "Methane" or "Residual" and Press "Zero".

- iii. Turn the front panel knob. If the reading is larger than "0", reduce the potentiometer value. If the reading is smaller than "0", increase the potentiometer value.
- iv. Press Main" / "More" / "Menu" until "GC" is displayed and Press "GC".
- 9. Press "Single".
- 10. Wait for about 3 minutes until the CH₄ and Residual analysis is finished.
- 11. Repeat steps 6-10 for both Methane and Residual until an acceptable zero reading is reached.
- 12. Press "Main" / "More" / "Menu" until "Modes" is displayed.
 - i. Press "Modes".
 - ii. Press "Track" for Display.
 - iii. Press "Main" / "More" / "Menu" until "GC" is displayed.
 - iv. Press "GC" and Press "Setup"
 - v. Press "Total".
 - vi. Press "Zero".
 - vii. Turn the front panel knob until the Total reading in the display window reads 0.0 ppm.
- 13. Close regulator and remove tubing from the zero gas cylinder.
- 14. Change the Modes display setup back to "Peak".

Span Gas Calibration

- 1. Close regulator valve on the span gas cylinder.
- 2. Open main valve on span gas cylinder.
- 3. Insert the 1/4" ID tubing (from the gas cylinder) into the inlet of the flow meter mounting on the side of instruments rack.
- 4. Adjust regulator valve until vent airflow is about 5 L/min (read from bottom of ball of the vent monitoring flow meter). This provides a little extra span gas to the analyzers and keeps the pressure inside the manifold close to the atmospheric pressure.
- 5. Flow Span gas for 2 minutes.
- 6. Press "Main" / "More" / "Menu" until "GC" is displayed and Press "GC". Press "Single".
- 7. Wait for about 3 minutes until the CH₄ and Residual analysis is finished.
- 8. If the CH₄ or Residual reading is not in the range of span gas concentration ± 0.1 ppm, the potentiometer needs to be adjusted.
 - i. Press "Setup".
 - ii. Press "Methane" or "Residual" and Press "Span".
 - iii. Turn the front panel knob. If the reading is larger than span gas concentration, reduce the potentiometer value. If the reading is smaller than span gas concentration, increase the potentiometer value.
 - iv. Press Main"/ "More" / "Menu" until "GC" is displayed and Press "GC".
- 9. Press "Single".
- 10. Wait for about 3 minutes until the CH₄ and Residual analysis is finished.
- 11. Repeat steps 6-10 for both Methane and Residual until an acceptable zero reading is reached.
- 12. Press "Main" / "More" / "Menu" until "Modes" is displayed.
 - i. Press "Modes".
 - ii. Press "Track" for Display.

- iii. Press "Main" / "More" / "Menu" until "GC" is displayed.
- iv. Press "GC" and Press "Setup"
- v. Press "Total".
- vi. Press "Span".
- vii. Turn the front panel knob until the Total reading in the display window reads 0.0 ppm.
- 13. Close regulator and remove tubing from the zero gas cylinder.
- 14. Change the Modes display setup back to "Peak".
- 15. Press "Main" / "More" / "Menu" until "GC" is displayed and Press "GC". Press "Auto".
- 16. On the front panel of the Labview program, click the "Manually control" and close the manually control.

Manufacturer Contact Information

VIG Industries, Inc. 4051 East La Palma Ave., Suite C Anaheim, California 92807-1751 Toll Free in the US: 800-862-7844 Phone: 714-632-8200 Fax: 714-632-8201 Email: Service@vigindustries.com

Reference

Manual of Model 200 Methane/Non-Methane/Total Hydrocarbon Analyzer

Date of Calibration:			Calibrated by:	
Time	Items		Unit =	Notes
	Ambient pressure, (mm Hg)			
: :	Sample airflow (L/min)			
: :	Zero air applied			Cylinder P: psi
: :	Total Reading			
: :	CH₄ Reading			
: :	Propane Reading			
: :	CH ₄ (ppm) applied			Cylinder P: psi
	CH₄ Reading			
: :	Propane (ppm) appli	ied		Cylinder P: psi
	Propane Reading			
: :	New Calibration		^{Yes} / _{No}	
: :	Zero air applied			
	Total adjustment:	CH ₄ adjustment:	Propa	ane adjustment:
	Total Reading:	CH₄ Reading:	Propa	ine Reading:
: :	CH ₄ applied			
	Total adjustment:	CH ₄ adjustment:	Propa	ane adjustment:
	Total Reading:	CH₄ Reading:	Propa	ine Reading:
: :	Propane applied			
	Total adjustment:	CH ₄ adjustment:	Propa	ane adjustment:
	Total Reading:	CH₄ Reading:	Propa	ine Reading:
: :	Finish calibration		Yes/ _{No}	
: :	Time switch off			
: :	Connect analyzer back to	sampling system.		
Notes:				

Maintenance/Calibration Record Sheet for VIG 200

Appendix M: SOP of Tapered Element Oscillating Microbalance (TEOM) for TSP Measurement

Introduction

The TEOM instrument (TEOM 1400 Ambient Particulate (PM_{10}) Monitor) is a continuous PM monitoring device designated by USEPA as an equivalent method (EPA Designation No. EQPM-1090-079) for PM_{10} ($\leq 10 \mu m$ aerodynamic diameter). The acronym TEOM stands for "Tapered Element Oscillating Microbalance", an inertial measurement technique that operates on changes in the resonant frequency of an oscillating element as a function of increases in particle mass collected on a filter attached to the element. Changes in the element's resonant frequency are sampled electronically in quasi-real time, providing both continuous and time-averaged measures of mass accumulation that are directly proportional to instantaneous and time-averaged mass concentrations in air, respectively. The device operates at an industry-standard, volume-controlled flow rate of 16.7 L/min so that it can be outfitted with a variety of commercially available pre-separator inlets suitable for measuring PM. *The unit is fitted with a TSP inlet head in this application*.

Switching Instrument On/Off

Since the airflow is maintained at a constant volume, corrected for local temperature and barometric pressure, the operation of this monitor requires that the temperature and pressure sensors are connected for proper temperature and pressure readings and flow corrections.

Turn On Unit

Supply power to the instrument by plugging in the power cord to 120 VAC, and pressing the power button on the front panel of the TEOM control unit. The main screen (four-line display) will soon appear after showing the name of the instrument. Turn on the pump to draw the sample streams by plugging in the power cord to 120 VAC. The monitor waits at least 30 min after being powered up to compute the first mass concentration data.

Turn Off Unit

Press the power button on the front panel of the TEOM control unit. The four-line display becomes blank. Turn off the vacuum pump by pressing the power button and disconnect the control unit from 120 VAC by unplugging the power cord.

Status Line on Main Screen

Whenever a status code other then "OK" is shown on the display, the instrument automatically turns on the "Check Status" light on the front of the control unit. The information displayed on the main screen includes status condition, operating mode, A/O 1 mode, RS-232 mode, protection, and time. For example, the screen will show the following line at time XX:XX:



Status condition

- OK Normal operation
- M No frequency signal
- T Temperature(s) outside of operational bounds
- F Flow(s) outside of operational bounds
- X Filter nearing capacity—exchange filter

Operating mode

- 1 Temperature/flow stabilization
- 2 Begin TM computation
- 3 TM computed, begin MR/MC computation
- 4 Normal operating mode
- S Setup mode
- X Stop all mode

A/O 1 mode

- (Blank) Analog output 1 normal definition
- + Analog output 1 used for status watch
- XX% Filter loading (percent)

RS-232 mode

- N None
- P Print online
- R R&P protocol
- A AK protocol
- G German network protocol
- S Storage to printer
- F Fast storage output

Protection

- U Unlocked
- L Low lock
- H High lock

XX:XX Current time (24-hr format)

Using the Keypads and Software

User should refer to Section 4.5 of the operating manual and become familiar with the incorporated menu-driven software and keypads.

Filter Storage and Exchange

The measurements must be conducted with TEOM filter cartridges that are made of Tefloncoated glass fiber filter paper. Filters should be stored inside the sensor unit for easy access and to keep them dry and warm. *Do not handle new filters with your fingers*. Instead, use the filter exchange tool and follow the procedures given in the operating manual. Keep the sample pump running to facilitate filter exchange. Use the two pockets on the right side of the mass transducer (inside) to store the next two new TEOM filters, for pre-conditioning and removal of excessive moisture build-up prior to use.

System Operation after Power Failure

The system resets itself when power is regained, and enters the same mode as before. All operating parameters are maintained in the system's battery backed-up CMOS memory.

Setting Variables Stored in Data Logger

The <Step Screen> key toggles the instrument between the View Storage Screen and Set Storage Screen, or, pressing 09 <Enter> can gain direct access to the Set Storage Screen from any screen. The first eight lines of the Set Storage Screen contain the titles of the variables currently being stored in the data logger (Program Register Codes listed in Appendix A of operating manual). The "Interval" variable defines the time (in seconds) between successive writings of data to the circular buffer.

Code	Variable	Units
008	Mass concentration	μ g/m ³
009	Total mass	μg
035	Pressure drop	%
039	Current main flow	L/min
040	Current auxiliary flow	L/min
041	Status condition	code
057	30-min average mass concentration	μ g /m³
130	Current ambient temperature	°C
131	Current ambient pressure	atm

Table M 1	Some	nonular	nrooram	register	codes
	Some	populai	program	register	coucs.

Setting Analog Outputs

The instrument's three analog output channels are accessible from the identical 15-pin connectors on the front and back panels of the control unit. Details regarding the pin assignments and voltage (VDC) of outputs are given in the operating manual (Section 5.1). To bring up the Set Analog Output Screen, press <A/O>, or select "Set Analog Output" from Menu Screen, or press 04<Enter>.

Setting Sampling Average Time

The <Step Screen> key toggles the instrument to "Set Hardware", then press <Enter>. Press the "Data Stop" to start stetting the sampling average time.

Nomenclature

- TM Total mass
- MR Mass rate
- MC Mass concentration
- A/O Analog output
- atm atmosphere, 746 mBar

Calibration

Procedures are based on routine flow auditing, leak checking, and mass calibration verification. Since the TEOM monitor can be directly mass calibrated, it can be directly quality assured using a mass standard. All QA procedures should be coordinated with routine maintenance procedures to minimize down time.

Flow Audit

A flow audit adapter is provided and the procedures are outlined in the operating manual. Both the sample flow rate and total flow rate may be checked using the flow audit adapter with a capped nut for closing the flow splitter bypass line port. It is recommended that the volumetric flow rates be within $\pm 7\%$ of the set points. The United States Environmental Protection Agency (USEPA) requires a tolerance of $\pm 10\%$ for the total flow through the PM₁₀ inlet. If measured flows differ by more than the stated tolerances, recheck all settings, and perform the test again. Large errors in the flow may indicate other sources of error, such as a malfunctioning flow controller, a system leak, or improper temperature and pressure settings.

Leak Check

The leak check procedures are included in the operating manual (Section 7.6). The leak check should be performed with NO sample filter attached to the mass transducer; this will prevent accidental damage from occurring to the sample filter cartridge when exposed to the high pressure drop (vacuum) in the sample line created by the leak check. Flow rates should indicate less than 0.15 L/min for the main flow and less than 0.65 L/min for the auxiliary flow with the end of the sample line closed, if not, systematically check plumbing for connector leaks.

Mass Calibration Verification

The mass transducer is permanently calibrated and never requires recalibration under normal use. However, the mass measurement accuracy of the instrument may be verified following procedures in the operating manual. R&P offers a mass calibration verification kit to help perform this procedure.

Maintenance

The lifetime of a *TEOM filter cartridge* depends on the nature and concentration of the particulate sampled and the main flow rate setting (1, 2, or 3 L/min). The filter must be exchanged when the filter loading value (as shown on the status line of the main menu)

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approaches 70%. At a flow rate of 3 L/min, 100% filter loading generally corresponds to a total mass accumulation of approximately 3 to 5 mg of particulate. Filter lifetime at a main flow rate of 3 L/min is generally 21 days at an average PM_{10} concentration of 50 µg/m³. Flow splitter adapters for 1 and 2 L/min operation are available for use in areas with higher particulate concentrations.

The factory recommended schedule of periodic maintenance and the schedule to be adopted for use in livestock barns are as follows:

Maintenance item	Factory	This Project
Clean air inlet head	Upon filter exchange	Twice a Week
Replace TEOM filter cartridge	70% load	Weekly
Exchange in-line filters	6 mon. or when loaded	When loaded
Leak test	Annually	Every flock
Analog board calibration	Annually	Every 6 months

The TEOM TSP air inlet requires regular maintenance in livestock barns. The TEOM inlet itself should be cleaned weekly. In order to do this one must first push the <Data Stop> button on the keypad. Remove the dirty TEOM inlet and replace it with a clean inlet. The dirty TSP inlet will be taken back to work station for cleaning and ready for next inlet swap. For cleaning the dirty inlet, please see Manual Appendix G: Inlet Maintenance. Grease as needed to maintain a slight layer of silicon grease. Carefully replace the inlet unit, and return to the control unit of the TEOM system, and press the <Data Stop> button once more. This will prevent skewed data from being recorded. It will automatically begin recording in approximately 30 min.

Other Settings

Flow rate through sample inlet: 16.7 L/min (1 m³/hr) Main flow rate: 1 L/min Temperature of sample stream: 50°C Particulate concentration: $< 5 \ \mu g/m^3$ to several g/m³ Standard conditions: 1 atm pressure, 20°C

Spare Parts and Consumables

Slow blow 2A, 250 V fuse 2A & 250 V in-line fuse, P/N 04003419 Box of 20 TEOM filter cartridges (TX40 media), P/N 57-000397-0020 Large bypass in-line filter, P/N 57-002758 Flow controller filter, P/N 30-003097

Manufacturer Contact Information

Rupprecht & Patashnick Co., Inc. 25 Corporate Circle Albany, NY 12203 USA Timothy Morphy Assistant Product Manager Phone: 518-452-0065 Fax: 518-.452-0067 tmorphy@rpco.com (email)

Peggy O'Gormon Marketing Associate Phone: ext. 3229 email: pogorman@rpco2.com

Reference

TEOM 1400 Operation manual

Appendix N: SOP of Tapered Element Oscillating Microbalance (TEOM) for PM₁₀

Introduction

The TEOM instrument (TEOM 1400 Ambient Particulate (PM_{10}) Monitor) is a continuous PM monitoring device designated by USEPA as an equivalent method (EPA Designation No. EQPM-1090-079) for PM_{10} ($\leq 10 \mu m$ aerodynamic diameter). The acronym TEOM stands for "Tapered Element Oscillating Microbalance", an inertial measurement technique that operates on changes in the resonant frequency of an oscillating element as a function of increases in particle mass collected on a filter attached to the element. Changes in the element's resonant frequency are sampled electronically in quasi-real time, providing both continuous and time-averaged measures of mass accumulation that are directly proportional to instantaneous and time-averaged mass concentrations in air, respectively. The device operates at an industry-standard, volume-controlled flow rate of 16.7 L/min so that it can be outfitted with a variety of commercially available pre-separator inlets suitable for measuring PM. *The unit is fitted with a PM*₁₀ *inlet head in this application*.

Switching Instrument On/Off

Since the airflow is maintained at a constant volume, corrected for local temperature and barometric pressure, the operation of this monitor requires that the temperature and pressure sensors are connected for proper temperature and pressure readings and flow corrections.

Turn On Unit

Supply power to the instrument by plugging in the power cord to 120 VAC, and pressing the power button on the front panel of the TEOM control unit. The main screen (four-line display) will soon appear after showing the name of the instrument. Turn on the pump to draw the sample streams by plugging in the power cord to 120 VAC. The monitor waits at least 30 min after being powered up to compute the first mass concentration data.

Turn Off Unit

Press the power button on the front panel of the TEOM control unit. The four-line display becomes blank. Turn off the vacuum pump by pressing the power button and disconnect the control unit from 120 VAC by unplugging the power cord.

Status Line on Main Screen

Whenever a status code other then "OK" is shown on the display, the instrument automatically turns on the "Check Status" light on the front of the control unit. The information displayed on the main screen includes status condition, operating mode, A/O 1 mode, RS-232 mode, protection, and time. For example, the screen will show the following line at time XX:XX:



Status Condition

- OK Normal operation
- M No frequency signal
- T Temperature(s) outside of operational bounds
- F Flow(s) outside of operational bounds
- X Filter nearing capacity—exchange filter

Operating Mode

- 1 Temperature/flow stabilization
- 2 Begin TM computation
- 3 TM computed, begin MR/MC computation
- 4 Normal operating mode
- S Setup mode
- X Stop all mode

A/O 1 Mode

- (Blank) Analog output 1 normal definition
- + Analog output 1 used for status watch
- XX% Filter loading (percent)

RS-232 Mode

- N None
- P Print online
- R R&P protocol
- A AK protocol
- G German network protocol
- S Storage to printer
- F Fast storage output

Protection

- U Unlocked
- L Low lock
- H High lock

XX:XX Current time (24-hr format)

Using the Keypads and Software

User should refer to Section 4.5 of the operating manual and become familiar with the incorporated menu-driven software and keypads.

Filter Storage and Exchange

The measurements must be conducted with TEOM filter cartridges that are made of Tefloncoated glass fiber filter paper. Filters should be stored inside the sensor unit for easy access and to keep them dry and warm. *Do not handle new filters with your fingers*. Instead, use the filter exchange tool and follow the procedures given in the operating manual. Keep the sample pump running to facilitate filter exchange. Use the two pockets on the right side of the mass transducer (inside) to store the next two new TEOM filters, for pre-conditioning and removal of excessive moisture build-up prior to use.

System Operation after Power Failure

The system resets itself when power is regained, and enters the same mode as before. All operating parameters are maintained in the system's battery backed-up CMOS memory.

Setting Variables Stored in Data Logger

The <Step Screen> key toggles the instrument between the View Storage Screen and Set Storage Screen, or, pressing 09<Enter> can gain direct access to the Set Storage Screen from any screen. The first eight lines of the Set Storage Screen contain the titles of the variables currently being stored in the data logger (Program Register Codes listed in Appendix A of operating manual). The "Interval" variable defines the time (in seconds) between successive writings of data to the circular buffer.

Code	Variable	Units
008	Mass concentration	μg/m³
009	Total mass	μg
035	Pressure drop	%
039	Current main flow	L/min
040	Current auxiliary flow	L/min
041	Status condition	code
057	30-min average mass concentration	μg/m ³
130	Current ambient temperature	°C
131	Current ambient pressure	atm

Setting Analog Outputs

The instrument's three analog output channels are accessible from the identical 15-pin connectors on the front and back panels of the control unit. Details regarding the pin assignments and voltage (VDC) of outputs are given in the operating manual (Section 5.1). To bring up the Set Analog Output Screen, press <A/O>, or select "Set Analog Output" from Menu Screen, or press 04<Enter>.

Setting Sampling Average Time

The <Step Screen> key toggles the instrument to "Set Hardware", then press <Enter>. Press the "Data Stop" to start stetting the sampling average time.

Nomenclature

- TM Total mass
- MR Mass rate
- MC Mass concentration
- A/O Analog output
- atm atmosphere, 746 mBar

Calibration

Procedures are based on routine flow auditing, leak checking, and mass calibration verification. Since the TEOM monitor can be directly mass calibrated, it can be directly quality assured using a mass standard. All QA procedures should be coordinated with routine maintenance procedures to minimize down time.

Flow Audit

A flow audit adapter is provided and the procedures are outlined in the operating manual. Both the sample flow rate and total flow rate may be checked using the flow audit adapter with a capped nut for closing the flow splitter bypass line port. It is recommended that the volumetric flow rates be within $\pm 7\%$ of the set points. The United States Environmental Protection Agency (USEPA) requires a tolerance of $\pm 10\%$ for the total flow through the PM₁₀ inlet. If measured flows differ by more than the stated tolerances, recheck all settings, and perform the test again. Large errors in the flow may indicate other sources of error, such as a malfunctioning flow controller, a system leak, or improper temperature and pressure settings.

Leak Check

The leak check procedures are included in the operating manual (Section 7.6). The leak check should be performed with NO sample filter attached to the mass transducer; this will prevent accidental damage from occurring to the sample filter cartridge when exposed to the high pressure drop (vacuum) in the sample line created by the leak check. Flow rates should indicate less than 0.15 L/min for the main flow and less than 0.65 L/min for the auxiliary flow with the end of the sample line closed, if not, systematically check plumbing for connector leaks.

Mass Calibration Verification

The mass transducer is permanently calibrated and never requires recalibration under normal use. However, the mass measurement accuracy of the instrument may be verified following procedures in the operating manual. R&P offers a mass calibration verification kit to help perform this procedure.

Maintenance

The lifetime of a TEOM filter cartridge depends on the nature and concentration of the particulate sampled and the main flow rate setting (1, 2, or 3 L/min). The filter must be exchanged when the filter loading value (as shown on the status line of the main menu)

approaches 70%. At a flow rate of 3 L/min, 100% filter loading generally corresponds to a total mass accumulation of approximately 3 to 5 mg of particulate. Filter lifetime at a main flow rate of 3 L/min is generally 21 days at an average PM_{10} concentration of 50 µg/m³. Flow splitter adapters for 1 and 2 L/min operation are available for use in areas with higher particulate concentrations.

The factory recommended schedule of periodic maintenance and the schedule to be adopted for use in livestock barns are as follows:

Maintenance item	<u>Factory</u>	This Project
Clean air inlet head	Upon filter exchange	Twice a week
Replace TEOM filter cartridge	70% load	Weekly
Exchange in-line filters	6 mon. or when loaded	When loaded
Leak test	Annually	Every flock
Analog board calibration	Annually	Every 6 months

The TEOM PM_{10} air inlet requires regular maintenance in livestock barns. The TEOM inlet itself should be cleaned weekly. In order to do this one must first push the <Data Stop> button on the keypad. Remove the dirty TEOM inlet and replace it with a clean inlet. The dirty PM_{10} inlet will be taken back to work station for cleaning and ready for next inlet swap. For cleaning the dirty inlet, please see Manual Appendix G: Inlet Maintenance. Grease as needed to maintain a slight layer of silicon grease. Carefully replace the inlet unit, and return to the control unit of the TEOM system, and press the <Data Stop> button once more. This will prevent skewed data from being recorded. It will automatically begin recording in approximately 30 min.

Other Settings

Flow rate through sample inlet: 16.7 L/min (1 m³/hr) Main flow rate: 1 L/min Temperature of sample stream: 50°C Particulate concentration: $< 5 \ \mu g/m^3$ to several g/m³ Standard Conditions: 1 atm pressure, 20°C

Spare Parts and Consumables

Slow blow 2A, 250 V fuse 2A & 250 V in-line fuse, P/N 04003419 Box of 20 TEOM filter cartridges (TX40 media), P/N 57-000397-0020 Large bypass in-line filter, P/N 57-002758 Flow controller filter, P/N 30-003097

Manufacturer Contact Information

Rupprecht & Patashnick Co., Inc. 25 Corporate Circle Albany, NY 12203 Timothy Morphy Assistant Product Manager 518.452.0065 voice 518.452.0067 fax tmorphy@rpco.com (email)

Peggy O'Gormon Marketing Associate Phone: ext. 3229 email: pogorman@rpco2.com

Reference

TEOM 1400 Operation manual

Appendix O: SOP of Tapered Element Oscillating Microbalance (TEOM) for PM_{2.5}

Introduction

The TEOM instrument (TEOM 1400a Ambient Particulate (PM_{10}) Monitor) is a continuous PM monitoring device designated by USEPA as an equivalent method (EPA Designation No. EQPM-1090-079) for PM_{10} ($\leq 10 \mu m$ aerodynamic diameter). The acronym TEOM stands for "Tapered Element Oscillating Microbalance", an inertial measurement technique that operates on changes in the resonant frequency of an oscillating element as a function of increases in particle mass collected on a filter attached to the element. Changes in the element's resonant frequency are sampled electronically in quasi-real time, providing both continuous and time-averaged measures of mass accumulation that are directly proportional to instantaneous and time-averaged mass concentrations in air, respectively. The device operates at an industry-standard, volume-controlled flow rate of 16.7 L/min so that it can be outfitted with a variety of commercially available pre-separator inlets suitable for measuring PM. *The unit is fitted with a PM_{2.5} inlet head in this application*.

Switching Instrument On/Off

Since the airflow is maintained at a constant volume, corrected for local temperature and barometric pressure, the operation of this monitor requires that the temperature and pressure sensors are connected for proper temperature and pressure readings and flow corrections.

Turn On Unit

Supply power to the instrument by plugging in the power cord to 120 VAC, and pressing the power button on the front panel of the TEOM control unit. The main screen (four-line display) will soon appear after showing the name of the instrument. Turn on the pump to draw the sample streams by plugging in the power cord to 120 VAC. The monitor waits at least 30 min after being powered up to compute the first mass concentration data.

Turn Off Unit

Press the power button on the front panel of the TEOM control unit. The four-line display becomes blank. Turn off the vacuum pump by pressing the power button and disconnect the control unit from 120 VAC by unplugging the power cord.

Status Line on Main Screen

Whenever a status code other then "OK" is shown on the display, the instrument automatically turns on the "Check Status" light on the front of the control unit. The information displayed on the main screen includes status condition, operating mode, A/O 1 mode, RS-232 mode, protection, and time. For example, the screen will show the following line at time XX:XX:



Status Condition

- OK Normal operation
- M No frequency signal
- T Temperature(s) outside of operational bounds
- F Flow(s) outside of operational bounds
- X Filter nearing capacity—exchange filter

Operating Mode

- 1 Temperature/flow stabilization
- 2 Begin TM computation
- 3 TM computed, begin MR/MC computation
- 4 Normal operating mode
- S Setup mode
- X Stop all mode

A/O 1 Mode

(Blank) Analog output 1	normal definition
-------------------------	-------------------

- + Analog output 1 used for status watch
- XX% Filter loading (percent)

RS-232 Mode

- N None
- P Print online
- R R&P protocol
- A AK protocol
- G German network protocol
- S Storage to printer
- F Fast storage output

Protection

- U Unlocked
- L Low lock
- H High lock

XX:XX Current time (24-hr format)

Using the Keypads and Software

User should refer to Section 4.5 of the operating manual and become familiar with the incorporated menu-driven software and keypads.

Filter Storage and Exchange

The measurements must be conducted with TEOM filter cartridges that are made of Tefloncoated glass fiber filter paper. Filters should be stored inside the sensor unit for easy access and to keep them dry and warm. *Do not handle new filters with your fingers*. Instead, use the filter exchange tool and follow the procedures given in the operating manual. Keep the sample pump running to facilitate filter exchange. Use the two pockets on the right side of the mass transducer (inside) to store the next two new TEOM filters, for pre-conditioning and removal of excessive moisture build-up prior to use.

System Operation after Power Failure

The system resets itself when power is regained, and enters the same mode as before. All operating parameters are maintained in the system's battery backed-up CMOS memory.

Setting Variables Stored in Data Logger

The <Step Screen> key toggles the instrument between the View Storage Screen and Set Storage Screen, or, pressing 09<Enter> can gain direct access to the Set Storage Screen from any screen. The first eight lines of the Set Storage Screen contain the titles of the variables currently being stored in the data logger (Program Register Codes listed in Appendix A of operating manual). The "Interval" variable defines the time (in seconds) between successive writings of data to the circular buffer.

Code	Variable	Units
008	Mass concentration	μg/m³
009	Total mass	μg
035	Pressure drop	%
039	Current main flow	L/min
040	Current auxiliary flow	L/min
041	Status condition	code
057	30-min average mass concentration	μg/m³
130	Current ambient temperature	°C
131	Current ambient pressure	atm

Table O.1. Some popular program register codes.

Setting Analog Outputs

The instrument's three analog output channels are accessible from the identical 15-pin connectors on the front and back panels of the control unit. Details regarding the pin assignments and voltage (VDC) of outputs are given in the operating manual (Section 5.1). To bring up the Set Analog Output Screen, press <A/O>, or select "Set Analog Output" from Menu Screen, or press 04<Enter>.

Setting Sampling Average Time

The <Step Screen> key toggles the instrument to "Set Hardware", then press <Enter>. Press the "Data Stop" to start stetting the sampling average time.

Nomenclature

- TM Total mass
- MR Mass rate
- MC Mass concentration
- A/O Analog output
- atm atmosphere, 746 mBar

Calibration

Procedures are based on routine flow auditing, leak checking, and mass calibration verification. Since the TEOM monitor can be directly mass calibrated, it can be directly quality assured using a mass standard. All QA procedures should be coordinated with routine maintenance procedures to minimize down time.

Flow Audit

A flow audit adapter is provided and the procedures are outlined in the operating manual. Both the sample flow rate and total flow rate may be checked using the flow audit adapter with a capped nut for closing the flow splitter bypass line port. It is recommended that the volumetric flow rates be within $\pm 7\%$ of the set points. The United States Environmental Protection Agency (USEPA) requires a tolerance of $\pm 10\%$ for the total flow through the PM₁₀ inlet. If measured flows differ by more than the stated tolerances, recheck all settings, and perform the test again. Large errors in the flow may indicate other sources of error, such as a malfunctioning flow controller, a system leak, or improper temperature and pressure settings.

Leak Check

The leak check procedures are included in the operating manual (Section 7.6). The leak check should be performed with NO sample filter attached to the mass transducer, this will prevent accidental damage from occurring to the sample filter cartridge when exposed to the high pressure drop (vacuum) in the sample line created during leak checks. Flow rates should indicate less than 0.15 L/min for the main flow and less than 0.65 L/min for the auxiliary flow with the end of the sample line closed, if not, systematically check plumbing for connector leaks.

Mass Calibration Verification

The mass transducer is permanently calibrated and never requires recalibration under normal use. However, the mass measurement accuracy of the instrument may be verified following procedures in the operating manual. R&P offers a mass calibration verification kit to help perform this procedure.

Maintenance

The lifetime of a *TEOM filter cartridge* depends on the nature and concentration of the particulate sampled, and the main flow rate setting (1, 2, or 3 L/min). The filter must be exchanged when the filter loading value (as shown on the status line of the main menu) *approaches* 70%. At a flow rate of 3 L/min, 100% filter loading generally corresponds to a total

mass accumulation of approximately 3 to 5 mg of particulate. Filter lifetime at a main flow rate of 3 L/min is generally 21 days at an average PM_{10} concentration of 50 µg/m³. Flow splitter adapters for 1 and 2 L/min operation are available for use in areas with higher particulate concentrations.

The factory recommended schedule of periodic maintenance and the schedule to be adopted for use in livestock barns are as follows:

Maintenance item	<u>Factory</u>	This Project
Clean air inlet head	Upon filter exchange	Twice a week
Replace TEOM filter cartridge	70% load	Weekly
Exchange in-line filters	6 mon. or when loaded	When loaded
Leak test	Annually	Every flock
Analog board calibration	Annually	Every 6 months

The TEOM $PM_{2.5}$ air inlet requires regular maintenance in livestock barns. The TEOM inlet itself should be cleaned weekly. In order to do this one must first push the <Data Stop> button on the keypad. Remove the dirty TEOM inlet and replace it with a clean inlet. The dirty $PM_{2.5}$ inlet will be taken back to work station for cleaning and ready for next inlet swap. For cleaning the dirty inlet, please see Manual Appendix G: Inlet Maintenance. Grease as needed to maintain a slight layer of silicon grease. Carefully replace the inlet unit, and return to the control unit of the TEOM system, and press the <Data Stop> button once more. This will prevent skewed data from being recorded. It will automatically begin recording in approximately 30 min.

Other Settings

Flow rate through sample inlet: 16.7 L/min (1 m³/hr) Main flow rate: 1 L/min Temperature of sample stream: 50°C Particulate concentration: $< 5 \ \mu g/m^3$ to several g/m³ Standard Conditions: 1 atm pressure, 20°C

Spare Parts and Consumables

Slow blow 2A, 250 V fuse 2A & 250 V in-line fuse, P/N 04003419 Box of 20 TEOM filter cartridges (TX40 media), P/N 57-000397-0020 Large bypass in-line filter, P/N 57-002758 Flow controller filter, P/N 30-003097

Manufacturer Contact Information

Rupprecht & Patashnick Co., Inc. 25 Corporate Circle Albany, NY 12203 Timothy Morphy Assistant Product Manager 518.452.0065 voice 518.452.0067 fax tmorphy@rpco.com (email)

Peggy O'Gormon Marketing Associate Phone: ext. 3229 email: pogorman@rpco2.com

Reference

TEOM 1400 Operation manual

Appendix P: SOP of Barometric Pressure Sensor

The purpose of barometric pressure measurements is for correction of the emission calculations.

The sensor has two wires, a red wire for power and the black wire for the output signal. *Warning: Always connect the sensor with the power turned off.*

The barometric pressure sensor may be stored without any special provisions. During storage, place the sensor inside a bag to keep the sensor clean and store it on a shelf or hang it on a wall.

Barometric pressure will be monitored continuously outside of the houses using a WE100 with a range of 80,000 to 110,000 Pa and an accuracy of \pm 300pa. One barometric static pressure sensor is required for each house. The barometric pressure sensor will be compared and calibrated with a calibrated portable pressure sensor at various span pressures.

WE100 barometric pressure transducer specifications:

Output: 4-20mA Range: 800-1100 millibars Accuracy: +1% of full scale Operating Voltage: 10-36VDC Current Draw: Same as sensor output Warm Up Time: 3 seconds minimum Operating Temperature: -40° to +55°C

Calibration

- Calibrate the sensor every year or at a different frequency if specified by the QAPP for a particular project.
- Calibrate the sensor system (sensor and data logger) against a Fortin barometer.
- Follow the documented procedures for reading and adjusting the Fortin barometer.
- Read instantaneous measurements from data logger/sensor system at the time of each Fortin barometer reading.
- Conduct six paired measurements over a two-day period; the average mean of their respective readings is taken out and if they match then the unit is calibrated.
- Record the results of the calibration on the sensor lab notebook form.
- If the mean difference between the Fortin barometer and sensor/data logger measurement is less than or equal to 4 hPa, then the sensor passes calibration and the drift will be back-corrected.
- If the mean difference is > 42 hPa, the barometer needs to be adjusted against a Fortin barometer

Manufacturer Contact Information

Global Water Instrumentation, Inc. 11257 Coloma Road Gold River, CA 95670 USA Phone: 800-876-1172 Fax: 916-638-3270

Appendix Q: SOP for Use of Rotem RSC-2 Scale System

Routine Check

1. Check scales for clearance between scales and litter. Optimum separation is 1 inch.

Routine Download

- 1. Start up the computer notebook with USB-Serial Adapter connected.
- 2. Hookup Serial Communications between notebook and Rotem Serial Cable
- 3. Start up Rotem Scalenet Application
- 4. As soon as software establishes communications with the Rotem Scales, the main screen will appear on the computer screen displaying the current growth day and if the scales are in operation.
- 5. On the application's pull down menu, click on "*Setup*", "*Farm Name*" to change to the current house name (i.e., Tyson 1-5). (This changes only the software data reference, not the data retained in the actual scale's database.)
- 6. On the application's pull down menu, click on "*Setup*", "*Set Flock Number*" to verify current Project Flock Designation Number (i.e., 2). (This changes only the software data reference, not the data retained in the actual scales database.)
- 7. Click on **Collect** icon button to start download of history file for the current flock. Save the download file as both a Rotem (*.his) and Excel (*.xls) file. Designate the files to be saved in the corresponding folder (....\House 1-5\ or\House 3-3\) on the computer notebook (C:\Documents and settings\Administrator\My Documents\Rotem Data\).
- 8. As soon as download is complete, close the Scalenet application, disconnect the communications cables, and turn off the computer.
- 9. Repeat this process during each visit. When downloading, overwrite the previous file.
- 10. At the end of the flock, email the final excel file (*.xls) to ISU for evaluation.

Between Flocks

- 1. Immediately prior to bird catch, remove the scale platform from the load cell, and clean and store it in a safe location.
- 2. Download the final flock weights and email the final excel file (*.xls) to ISU for evaluation.
- 3. Sometime prior to placement of the next flock, check the accuracy of the scales using known weights. Upon verification, remove the platform and store it in a safe location.
- 4. Immediately after placement of the next flock of birds, place the platform back on the load cell. Using the Scalenet application, click on the **Setting** icon button and reset the scales database. A different screen will appear. In the left column, click on "Growth Day" to change the day to 1; click on "Flock Number" to change to the current flock number designation, and click on "Time" to verify the correct time with the computer time.

References

Rotem Poultry Scale System RSC-2 User Manual. Scalenet Communications Program for RSC-2 User Manual.

Appendix R: SOP for Fan Current Switch Application

Figure R.1 shows the induction operated current switch (CS) (CR9321, CR Magnetics, St. Louis, MO) application in monitoring fan operational status. The CS is used in a "pigtail" which acts as a short extension cord. The "pigtail" is put in line between the fan motor and its AC power source. Water proof electrical tape is wrapped extensively around the plug connections to protect them from water and dust. The plugs are then "locked" together using multiple plastic ties as a deterrent to being separated. When AC current is present the CS returns a user defined amount of DC current to the MAEMU.



Figure R.1. CS application.

Appendix S: SOP of Litter Sample Analytical Methods

Determination of Manure pH

Safety notes

- Use lab coats, close-toed shoes and hand gloves.
- Mix liquid or semi-liquid manure thoroughly in the fume hood with the front panel pulled down.
- Do not immerse the pH electrode to the bottom of beaker.

Equipment

- pH meter
- 100 ml glass or plastic beakers
- Glass stirring rods
- Distilled water

Calibration

Calibrate the pH meter using standard buffer solutions (pH 4.0 and 7.0), if necessary.

Procedure for Liquid Manure (EPA SW-846, Method 9040)

U.S. Environmental Protection Agency. 1986. Test methods for evaluating solid waste. USEPA report SW-846.

- Thoroughly mix liquid manure (manure should be at room temperature)
- Pour 25 ml of liquid manure into a beaker
- Immerse pH electrode in liquid manure
- Record pH value when the meter has stabilized
- Repeat above steps for replicates

Procedure for Semi-Liquid Manure

Wolf, N. 2003. Determination of manure pH. p. 48-49. *In*: Peters et al. (eds.) Recommended Methods of Manure Analysis. University of Wisconsin System. http://ecommerce.uwex.edu/pdfs/A3769.PDF (accessed on May 23, 2006).

- Thoroughly mix semi-liquid manure (manure should be at room temperature)
- Place 20 ml of semi-solid manure into a beaker
- Add 40 ml of distilled water
- Stir and mix it well with a glass rod
- Allow for 30 minutes to settle solution
- Measure pH by immersing electrode into the supernatant solution
- Record pH value when the meter has stabilized
- Repeat above steps for replicates and report the result as pH (water 1:2)

Procedure for Solid Manure

Wolf, N. 2003. Determination of manure pH. p. 48-49. *In*: Peters et al. (eds.) Recommended Methods of Manure Analysis. University of Wisconsin System. http://cecommerce.uwex.edu/pdfs/A3769.PDF (accessed on May 23, 2006).

- Place 20 g of solid manure into a beaker
- Add 40 ml distilled water
- Stir and mix it well with a glass rod
- Allow for 30 minutes to settle solution
- Measure pH by immersing electrode into the supernatant solution
- Record pH value when the meter has stabilized
- Repeat above steps for replicates and report the result as pH (water 1:2)

Total Solids or Moisture Content Standard Method: 2540 B. Total Solids Dried at 103-105°C

Dish Preparation

ALWAYS handle dishes with gloves on!

- 1. Place clean evaporating dishes in oven at 103-105°C for 1 hour.
- 2. Remove dishes from oven and cool to room temperature in a desiccator. This takes about 2-3 hours.

Testing Liquids

ALWAYS handle dishes with gloves on!

- 1. Measure and record the weight of each measuring dish, using the enclosed balance.
- 2. Place three 10-15 ml of samples (about 2/3 full) into separate evaporating dishes. Measure the samples with a 10 mL pipet.
- 3. Do not get any sample on the outside of the dish. If it happens, wipe it off quickly.
- 4. Measure and record weight of each sample and dish, using the enclosed balance
- 5. Place sample into oven at 103-105°C for 24 hours.
- 6. Remove dishes from oven and cool to room temperature in a desiccator which takes about 2-3 hours.
- 7. Measure and record final weight of samples and dishes, using the enclosed balance.

Testing Solids

ALWAYS handle dishes with gloves on!

- 1. Measure and record the weight of each measuring dish, using the enclosed balance.
- 2. Use about 1 gram of sample for each dish.
- 3. Measure and record the weight of each sample and dish, using the enclosed balance.

- 4. Place sample in 103-105°C oven for 24 hours.
- 5. Remove samples from oven and cool to room temperature in a desiccator which takes about 2-3 hours.
- 6. Measure and record the final weight of the sample and dish, using the enclosed balance.
- 7. Repeat steps 4-6 until the weight is within 4% of the last drying cycle.

Calculations

% total solids =
$$1 - \left(\frac{\text{initial weight} - \text{final weight}}{\text{initial weight}}\right) \times 100$$

% Moisture = 100 - TS

Ammonia, Rapidstill II Standard Procedure Standard Method: 4500-NH₃ B. Preliminary Distillation Step & 4500-NH₃ C. Titrimetric Method

Safety Precautions

When mixing acids, use the fume hood with the front pulled down. Wear a lab coat, safety glasses, and gloves. *Mix small amounts of acid into larger amounts of water, not reverse!* Strong acids will burn holes in your clothing and will burn your skin.

Reagents Required

Label all reagents with reagent name, date of mixing, and your initials.

Sodium Tetraborate (0.025M)

- 1. Measure 9.5 g $Na_2B_4O_7*10 H_2O$ in a weigh boat.
- 2. Dilute to 1 L with distilled water in a 1000 ml volumetric flask.
- 3. Add a stir bar to the flask and mix on a stir plate until all the solid is dissolved, it may take 5 to 10 minutes.

Borate Buffer Solution

- 1. Using a 500 mL volumetric flask, measure 500 mL of 0.025*M* sodium tetraborate and put in a 1000 mL volumetric flask.
- 2. Measure 88 mL 0.1N NaOH by filling a 50 ml volumetric flask with 0.1N NaOH, then measuring 38 mL with a 10mL pipet. Put into the 1000 mL volumetric flask along with the sodium tetraborate.
- 3. Dilute to 1 L with distilled water.
- 4. Add a stir bar and mix well on a stir plate for approximately 5 minutes.

Mixed Indicator Solution—Prepare Monthly

- 1. Measure 20 mg methyl red indicator in a weigh boat and put it into a 100 mL volumetric flask.
- 2. Dissolve in 100 mL 95% ethyl alcohol added to the flask.
- 3. Measure 100 mg methylene blue and put it into a 100 mL volumetric flask.
- 4. Dissolve in 100 mL 95% ethyl alcohol added to the flask.
- 5. Mix the two 100 mL flask together in a 250 volumetric flask, add a stir bar, and mix well on a stir plate for approximately 5 minutes.

Indicating Boric Acid Solution—Prepare Monthly

- 1. Measure 20g H₃BO₃ in a weigh boat and add to a 1000 mL volumetric flask.
- 2. Add approximately 700 mL distilled water to the flask and dissolve the H₃BO₃ with a stir bar and a stir plate; it may take 10 to 15 minutes.
- 3. Add 10 mL mixed indicator solution to the flask.
- 4. Dilute to 1 L with distilled water, add a stir bar and mix well on a stir plate for approximately 5 minutes.

Standard Sulfuric Acid Titrant, 0.02N

- 1. Measure 2.8 mL concentrated sulfuric acid with a 10 mL pipet.
- 2. Dilute to 1 L with distilled water in a 1000 mL volumetric flask to make 0.1N sulfuric acid. Add a stir bar and mix well on a stir plate for 5 to 10 minutes.
- 3. Measure 200 mL 0.1N sulfuric acid with a 200 mL volumetric flask.
- 4. Pour the 200 mL of 0.1N sulfuric acid in a 1000 mL and dilute to 1 L with distilled water.
- 5. Add a stir bar and mix well on a stir plate for approximately 5 minutes.

Sodium Hydroxide (6N)

- 1. Measure 240g NaOH in a large weigh boat.
- 2. Dilute to 1 L with distilled water in a 1000 mL volumetric flask.
- 3. Add a stir bar to the flask and mix well on a stir plate until all the solid is dissolved this could take up to 30 minutes.

Distillation Preparation

- 1. Turn on cooling water by going under the sink and looking way in the back. In the top right corner there is a silver handle. Turn it 45° to the left, allowing water to flow through condenser.
- 2. Depress *Boiler Water* switch, and fill to the top line on the flask (see #10 under Distillation Procedure).
- 3. Turn on the Boiler Heater by pressing the button.
- 4. When water is at a steady boil, the unit is ready for operation.
- 5. Make sure the NaOH inlet tube located on the left side of the Rapidstill is in a 100 mL graduated cylinder that is full of NaOH.

Manure Dilution

20:1 water to manure ratio serial dilution

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- 1. Add 25 mL distilled water to a 50 mL volumetric flask.
- 2. Mix liquid manure for 1 minute at low speed with a drill mixer.
- 3. With a carefully broken 10 mL pipet, fill the flask up to the line with manure sample. to ensure there is exactly 25 mL of manure sample.
- 4. Pour the contents of the 50 mL volumetric flask into a 500 mL volumetric flask.
- 5. Dilute the sample to 500 mL with distilled water.

Sample Preparation

75 mL samples, 10:1 water to dilute-manure ratio

- 1. Mix liquid manure for 1 minute at low speed with drill mixer.
- 2. Extract a 6.815 mL dilute-manure sample, place in a digestion tube.
- 3. Add 68.15 mL distilled water by filling a 50 mL volumetric flask, adding that to the digestion tube, and then adding the rest (18.15 mL) with a 10 mL pipet.
- 4. Add 3.75 mL borate buffer solution with a 10 mL pipet.

Sample Preparation of Known Sample for Calibration

150 mL samples with 100 mg/L NH₃

Manure and 100 mg/L NH₃ Sample

- 1. Extract a 13.7 mL of mixed sample, place in beaker.
- 2. Add 15.0 mL 1000 mg/L NH₃ solution.
- 3. Add 121.3 mL distilled H₂O.
- 4. Add 7.5 mL borate buffer solution.

Distilled H₂O and 100 mg/L NH₃ Sample

- 1. Place 15.0 mL 1000 mg/L NH₃ solution in a digestion tube using a 10 mL pipet.
- 2. Add 135.0 mL distilled H_2O to the digestion tube by filling a 100 mL volumetric. flask first then adding 35 mL with a 10 mL pipet.
- 3. Add 7.5 mL borate buffer solution with a 10 mL pipet.

Preparation of Blank Sample

Carry a blank sample through all steps of procedure to use in final calculations.

- 1. Measure 75 mL distilled H₂0 using a 50 mL volumetric flask and a 10 mL pipet for the rest.
- 2. Add 3.75mL borate buffer solution with a 10 mL pipet.

Distillation Procedure

- 1. Open the protective casing, pull down on the bottom lever piece, and place the digestion tube containing the sample in the clamping device.
- 2. Place a 500 mL Erlenmeyer flask containing 50 mL indicating boric acid solution under the Distillate Outlet tube. Make sure the holes in the tube are completely immersed in liquid.

- 3. Make sure the boiler is full of water. If not, depress the *Boiler Water* Switch to refill the boiler.
- 4. Turn the *Boiler Heater* on.
- 5. Depress *NaOH Addition* button to add 5 mL 6N NaOH solution to the digestion tube according to the graduated cylinder the NaOH is in.
- 6. When the water reaches boiling begin distillation by setting the timer knob to the desired distilling time.
- 7. Distill for 20 minutes or until froth reaches the condenser arm.
- 8. Collect distillate in the 500 mL Erlenmeyer flask.
- 9. Lower the flask so that the end of the outlet tube is free of contact with the liquid and continue distillation during the last minute or two to cleanse condenser and outlet tube.
- 10. When distillation has completed, the unit will return to standby position, with the boiler heater still in operation. DON'T let the boiler stay on, switch the *Boiler Heater* button to the off position.
- 11. Once the boiler is cool, depress *Boiler Water* switch to refill the boiler after each run. If doing additional distillations, turn the boiler heater back on, and the machine is ready when boiling resumes.
- 12. Run samples in duplicate, if numbers are not in agreement (up to 10%), then run a third sample.

Titration

- 1. Fill a 50 mL buret with 0.02N H2SO4 and add a stirbar to the 500 mL flask containing the distillate.
- 2. Titrate, drop wise, the distillate in the flask.
- 3. Titrate ammonia in distillate with standard sulfuric acid titrant until indicator in distillate turns pale lavender.

Calculation

Liquid samples: mg NH₃-N/L =
$$\frac{(A - B) \times 280}{mL} \times 20$$

Sludge or sediment samples: mg NH₃-N/kg =
$$\frac{(A - B) \times 280}{g \text{ dry wt sample}} \times 20$$

where: $A = volume of H_2SO_4$ titrated for sample, mL B = volume of H_2SO_4 titrated for blank, mL

Rapidstill II Routine Maintenance

Daily Following Use

- 1. Install a clean, empty digestion tube.
- 2. Remove caustic, NaOH supply tubing from vessel containing NaOH and place in vessel of purified water.
- 3. Turn on cooling water and fill boiler. *Turn on boiler heater*.
- 4. Depress *NaOH Addition* until all of the caustic NaOH has been purged out of the system and into digestion tube.
- 5. Remove the digestion tube and dispose of the liquid properly.
- 6. Replace digestion tube and fill half full by depressing *NaOH Addition*.
- 7. When water is at a rolling boil, turn distillation timer to 10 minutes.
- 8. At end of distillation cycle, turn off the boiler heater and cooling water.
- 9. Remove digestion tube and dispose of liquid properly.
- 10. Clean exterior surfaces of machine using a soft cloth and a mild soap and water solution.

Weekly

- 1. Examine boiler for any build up of deposits. Accumulated deposits may be removed by a dilute solution of hydrochloric acid, or by using commercially available descaling solutions that are compatible with glass.
- 2. Examine all plumbing and steam lines, ensuring that the tubing is intact. Examine all tubing connections. Examine the connection stopper for deterioration or cracking.

Total Kjeldahl Nitrogen Standard Procedure Standard Method: 4500-N_{org} D and C

Safety Precautions

- 1. Equipment should not be used until adequate training has been obtained, and the equipment manuals have been read.
- 2. Read the MSDS for sulfuric acid, sodium hydroxide, hydrochloric acid and boric acid. There are strong acids and bases (both heated) being used.
- 3. Wear long pants, lab coat, and close-toed shoes. Safety glasses and various types of gloves should also be worn.
- 4. This analysis should be conducted when there are other personnel in the area.

Reagents Required

Note: Unless otherwise stated, prepare solutions in deionized water. *Label all reagents with reagent name, date of mixing, and your initials.*

Concentrated Sulfuric Acid (90-98%)

1. Store in the cabinet labeled *Corrosives* underneath the fume hood.

2. When using, place the bottle of acid in a spill tray.

Fishertab Kjeldahl Tablets

1. Contains 15 mg selenium in 1.5 gm K₂SO₄.

Indicator Solution

- 1. Measure 225 mg of methyl red with a weigh boat using the enclosed balance.
- 2. Measure 83 mg of methylene blue with a weight boat using the enclosed balance.
- 3. Dissolve the methyl red and methylene blue in a 100 mL volumetric flask with 100 mL of 95% ethanol. This may take 10-20 minutes to dissolve.
- 4. Store the solution in an amber-colored bottle and label.

<u>4% Boric Acid</u>

- 1. Measure 80 gm (500 gm) of powdered boric acid with a large weight boat.
- 2. Slowly add the boric acid to about 1.5 L (10 L) of deionized water and allow the acid to dissolve by using a stir bar and a stir plate (it might take 30 minutes to 1 hour, several hours for greater amounts).
- 3. Bring the volume up to 2 L (12.5 L) with deionized water and continue stirring on the stir plate for 5 to 10 minutes.

30% Sodium Hydroxide

- 1. Measure 750 gm (1800 gm) of dry NaOH using a large weigh boat.
- 2. Slowly add the NaOH to 1.5 to 2 L (~5 L) water and allow the base to dissolved by using a stir bar and a stir plate
- 3. This is an exothermic reaction, container will get hot!
- 4. Bring the volume up to 2.5 L (6 L) with deionized water and continue stirring on a stir plate for 5 to 10 minutes.

Methyl Red Indicator Solution

- 1. Measure 20 mg of methyl red using a weigh boat.
- 2. Dissolve the methyl red in 60 mL absolute ethanol using a 100 mL volumetric flask. This may take 10-15 minutes.
- 3. Bring the volume of the solution up to 100 mL by adding 40 mL distilled water, and continue stirring for 5-10 minutes.

0.1N Hydrochloric Acid: 1:120 Dilution

- 1. Measure out 12.5 mL (125 mL) of concentrated HCl using a 10 mL pipet.
- 2. Put ~1 L (10 L) of deionized water in a container and add to it, slowly, the HCl.
- 3. Add a stir bar to the solution and let it mix for 10-15 minutes on a stir plate.
- 4. Bring the volume up to 1.5 L (15 L) with deionized water and continue stirring.

Standardization of Hydrochloric Acid

- 1. THAM (Tris(hydroxymethyl)aminomethane) is a primary standard base that will be used to standardized the hydrochloric acid.
- 2. Dry THAM in a desiccator (it should not be subjected to heat greater than 100°C.
- 3. Measure about 0.25 gm or less (record exact weight) of THAM using a weigh boat.

- 4. Add the THAM to three 125 mL Erlenmeyer flasks and dissolve in about 30 mL distilled water by swirling the mixture.
- 5. Add two drops of the Methyl Red Indicator (will be a yellow color).
- 6. Titrate the THAM solution to a red color with the HCl solution being standardized using the 25 mL buret.
- 7. Record the volume of acid required for each flask and calculate the normality of the HCl. Average the normality of the three flasks.

Normality of HCl = $(gm THAM \times 1000)/(121.1 \times mL of HCl)$

8. Indicate normality and the date standardized on the label of the 0.1N HCl.

Sample Preparation

Liquids

- 1. Mix liquid manure for 1 minute at low speed with a drill mixer.
- 2. Measure the liquid sample with a pipet. If the sample is not able to be pipetted, treat it as a solid sample.
- 3. At first we will try a range of sample volumes from 0 to 1.00 mL. Prepare duplicates or triplicates of each sample.
- 4. To improve accuracy using a 25 mL buret to titrate the ammonia, more than 15 mL of HCl should be used for the titration of each sample.
- 5. Use at least two blank tubes without sample in a run of 25 digestion tubes. The blanks should contain all reagents, receiving the same treatment as the samples. There will be an indicator color change in the blank. If not, something has gone wrong. Also, if new boric acid or HCl is made during the day, blanks are needed for the new reagents. Blanks *must* be run everyday.
- 6. Samples can be weighed into digestion tubes one or more days before digesting if tubes are covered so they do not become contaminated.
- 7. 0.05-0.06 gm urea or 0.2 gm tryptophan can be used to check the system for complete digestion without loss of nitrogen.

<u>Solids</u>

- 1. Weight representative sample in a tarred weigh boat and record the weight.
- 2. Transfer the sample to a digestion tube making sure that everything gets to the bottom of the tube. Prepared duplicates or triplicates of each sample.
- 3. To improve accuracy using a 25 mL buret to titrate the ammonia, more than 15 mL of HCl should be used for the titration of each sample.
- 4. For solid samples we will first test a range of sample weights from 0 to 1.00 gm.
- 5. Use at least two blank tubes without sample in a run of 25 digestion tubes. The blanks should contain all reagents, receiving the same treatment as the samples. There will be an indicator color change in the blank. If not, something has gone wrong. Also, if new Boric acid or HCl is made during the day, blanks are needed for the new reagents. Blanks *must* be run everyday.
- 6. Samples can be weighed into digestion tubes one or more days before digesting if tubes are covered so they do not become contaminated.

7. 0.05-0.06 gm urea or 0.2 gm tryptophan can be used to check the system for complete digestion without loss of nitrogen.

Digestion Procedure

- 1. Carefully insert the digestion tubes into the holes provided in the flask rack. The flask rack can be used for convenient handling and transportation of the digestion tubes.
- 2. Add 4 Fishertab Kjeldahl tablets to each digestion tube.
- 3. Carefully add 20 mL of concentrated sulfuric acid using a 10 mL pipet to each tube prior to placing the tubes in the digestion block.
- 4. Turn the main power switch located on front of controller to ON position. *The digestion block must be totally contained in a chemical fume hood, and must have as much space between the back of it and the back of the hood as possible*. Set temperature to 410°C using temperature dial on control unit. Allow approximately one hour for unit to reach pre-set temperature (digestor is at pre-set temperature when lights located on front of the digestor are flashing).
- 5. After completing sample and reagent addition to the digestion tubes, carefully elevate the rack by grasping the handles provided and place in position on the digestor unit. As the rack is lifted, each tube will rest on its top rim. Carefully lower the rack so that each tube enters its respective hole and bottoms in the base of the unit.
- 6. Place end plates on the flask rack. These plates completely close the ends of the flask rack and allow the temperature of the upper portion of the tube to elevate providing for proper refluxing and condensation of the sulfuric acid. (A certain amount of foaming will occur at the beginning of the digestion. The refluxing of the acid washes any carbonized material back into the digestion solution. *If foaming is uncontrollable, reduce digestion temperature and/or sample size.*
- 7. Digest samples at 410°C until the acid in the flask is clear and fumes are contained within the digestion tube, and not thick in appearance. A time of two hours is minimal for complete digestion. The "clearness" of the acid (color and absence of particulate matter) and fume density are indicators for completeness of digestion.
- 8. After digestion is complete, remove flask rack containing tubes from digestor and place on heat resistant pad. *Wear heat-protective gloves!* Turn heat settings on the digestion block back to zero and turn off power if there is no more digestion runs for the day.
- 9. With the heat-protective gloves, carefully remove manifolds from the digestion tubs. *Caution:* The manifolds may leak condensed acid and the flask may emit fumes. Carry the manifolds to a sink. Clean manifolds and racks with water and running a lot of water down the drain to dilute the acid.
- 10. After the tubes containing the digest have cooled to room temperature, slowly add 75 mL of deionized water to each tube, swirling to mix. There may be some fume emissions from the tubes so *be careful*. Dilution must be made before a cake is formed from precipitated salts, but not before the digest is cool enough to contain the exothermic reaction.
- 11. Contents in the tubes might turn orange or red-orange. This is common and will not affect results. This distillation should be performed after water is added. If the tubes cannot be distilled, gently vortex the tubes, cover them with aluminum foil and place in the fume hood.

Setup of the Rapidstill Distillation Unit

- 1. Add 70 mL of 4% Boric acid to each of the receiving flasks. Add five drops of Indicator solution (methyl red and methylene blue) to each receiving flask. Swirl to mix. (You can mix the boric acid and indicator solution together prior to putting it into the receiving flask.)
- 2. Turn on cooling tap water by going under the sink and looking way in the back. In the top right corner there is a silver handle. Turn it 45° to the left, allowing water to flow through the condenser. The distillate should be cold as it comes out. The distillation coil should be cold at the bottom and somewhat warm at the top. *Check this flow rate periodically, as the water pressure can vary. If there is inadequate water flow to cool the distillation coil, ammonia will be lost with steam instead of being condensed as a liquid in the receiving flask.*
- 3. Depress *Boiler Water* switch, and fill to the line on the flask (back of the distillation unit).
- 4. Turn on the *Boiler Heater* by pressing the button. When water is at a steady boil, the unit is ready for operation.
- 5. Make sure the NaOH inlet tube located on the left side of the Rapidstill is in a 500 mL graduate cylinder that is full of NaOH.

Distillation Procedure

- 1. Open the safety screen, pull down on the bottom lever piece, and place the digestion tube containing the digestor sample in the clamping device. Close the safety screen.
- 2. Place the 500 mL Erlenmeyer receiving flask containing the 70 mL 4% boric acid and 5 drop Indicator Solution under the Distillate Outlet tube on the front right part of the Rapidstill. Make sure the holes in the tube are completely immersed in liquid.
- 3. Make sure the boiler is full of water. If not, depress the *Boiler Water* switch to refill the boiler.
- 4. Turn the *Boiler Heater* on.
- 5. Depress the *NaOH Addition* button to add 85-90 mL of NaOH per 20 mL of acid used in the digestion. Measure this value according to the graduate cylinder the NaOH is contained in.
- 6. When the water is boiling nicely, being distillation by setting the timer knob to the desired distillation time.
- 7. Distill for 20 minutes or until froth reaches the condenser arm. The distillate is collected in the 500 mL Erlenmeyer flask.
- 8. Lower the flask so that the end of the outlet tube is free of contact with the liquid and continue distillation during the last minute or two to cleanse condenser and outlet tube.
- 9. When distillation is complete, the Rapidstill will return to standby position, with the boiler heater still in operation. *Don't* let the boiler stay on, switch the *Boiler Heater* button to the off position.
- 10. Once the boiler is cool, depress *Boiler Water* switch to refill the boiler after each run. If doing additional distillations, turn the boiler heater back on, and the machine is ready when boiling resumes.

11. There may be a few mL of sample left in the tube. Pour the remaining residue into waste container designated for selenium waste. When the container is full or the container has been in the lab for 90 days, the liquid needs to be disposed of through EH&S.

Titration

- 1. Before titrating a sample, completely fill the buret with 0.1N HCl and drain to waste enough so the initial reading of the buret can be made. Record initial reading.
- 2. Place a small stir bar in the receiving flask and place on a stir plate beneath of the buret. Stir gently.
- 3. Add HCl from the buret and allow the color to change from emerald green to a gray to light purple.
- 4. Titrate to the graph to light purple endpoint. *Note:* It is easy to overshoot the endpoint. When it takes longer for the green color to reappear, slow the addition of the acid down to one drop at a time. If too much acid is added (darker purple) the sample has been over titrated and is lost.
- 5. Refill the buret for the next sample.

Calculations

% Nitrogen in sample = $\frac{\text{Normality of acid} \times (\text{mL HCl added} - \text{mL from blank}) \times 1.4}{\text{sample size}}$

Use an average of the replicates of each sample for the final value.

Routine Maintenance and Cleaning

Digestion

Clean with a detergent and water solution when the unit is cool.

- 1. Condensed sulfuric acid will accumulate in the digestion manifold. If acid drops on the digestion block, digestion tubes, support rack, or counter top in the hood, the acid should immediately be wiped up with a wet paper towel.
- 2. Wipe down outer surfaces of the digestion block. Wipe down hood bench if acid has spilled. Wipe out glass attachments at the end of the aspiration tubes. (Use wet paper towels.)
- 3. Wipe off metal tops of the racks, and the racks themselves using wet paper towels.

Calibration

The controller has been factory calibrated and should not require re-calibration. In the event recalibration is required the following procedure should be followed.

- 1. Insert calibration resistor into the 2-pin sensor connector located on the rear of the control unit.
- 2. Place ON-OFF switch, located on the controller, to the ON position.
- 3. Adjust set-point indicator dial to a position where the indicator lights, located on front panel of digestor, are illuminated 50% of the time.

- 4. Loosen setscrew on set-point indicator dial and align calibration mark on dial with pointer label.
- 5. The digestor is now calibrated to give digestion temperature indicated on set-point dial.

Distillation

Daily following use:

- 1. Wash digestion/distillation tubes in hot water (no detergent). Use a scrub brush to clean the outside and inside. Rinse 2 or 3 times with hot water. Rinse twice with distilled water. Place in drying oven or on a rack so they will dry.
- 2. Rinse receiving flasks three times with hot water, and twice with distilled water. Place in dry oven or on a rack so they will dry.
- 3. Install a clean, empty distillation tube.
- 4. Remove caustic, *NaOH* supply tubing from vessel containing *NaOH* and place in vessel of purified water.
- 5. Turn on cooling water and fill boiler. *Turn on boiler heater*.
- 6. Depress NaOH Addition until all of the caustic NaOH has been purged out of the system and into the digestion tube.
- 7. Remove the digestion tube and dispose of liquid properly.
- 8. Replace digestion tube and fill half full by depressing NaOH Addition.
- 9. When water is at a rolling boil, turn distillation timer to 10 minutes
- 10. At the end of the distillation cycle, turn off the boiler heater and cooling water. Remove the digestion tube and dispose of liquid properly.
- 11. Clean exterior surfaces of machine using a soft cloth and a mild soap and water solution.

Weekly:

- 1. Examine boiler for any build up of deposits. Accumulated deposits may be removed by a dilute solution of hydrochloric acid, or by using commercially available descaling solutions that are compatible with glass.
- 2. Examine all plumbing and steam lines, ensuring that the tubing is intact. Examine all tubing connections. Examine the connection stopper for deterioration or cracking.

	Appendix T: Maintenance Checklists								
	Southeastern Broiler Emissions Project Between Flock Requirements Checklist								
Hous	e I.D Date Incoming Flock No								
	Clean screens and replace filters on the gas sample line intakes								
	Check air flow rate in sample lines and solenoid valves								
	Replace TEOM filters and exchange TEOM inlet heads and 2.5 micron cut cyclones								
	Leak test the TEOMs after all other maintenance has been performed on them								
	Instrument Calibration/Verification INNOVA for NH ₃ API for H ₂ S VIG for NMH Polytron I								
	Check all solenoid valves (ON/OFF)								
	Perform a system check on fan operational status and monitoring								
	Check airflow rate at 20% of the exhaust fans (randomly selected)								
	Replace any critical spare parts utilized during the previous flock								
	Collect copies of calibration and event log sheets from previous flock and submit to Lara Moody for archiving.								
	Collect litter samples and deliver to ISU for analysis								

Item	Quantity	In stock
Inclined manometer (red liquid)	1	
Setra 264 sensor (new)	1	
Humidity sensor (new and calibrator)	2	
24 VDC power supply	1	
Barometric sensor (calibrated)	1	
4-20 mA HOBO cable	2	
4-Channel HOBO logger	1	
Thermometer (-8 to 50°C) (NIST)	1	
Cooler (1-2 Gal)	2	
Pump (10 LPM)	1	
Tubing 1/8" ID	10 feet	
Tee (1/4" OD)	1	
Valve (ball)	1	
Cardboard box with an adjustable opening for Setra	1	
Salt (LiCl and NaCl)	500 gm	
Distilled water	1	
Clean syringe (0-10 mL)	2	
Wide mouth bottle (changeable cover)	2B + 4 C	
Ruler (for TEOM)	1	
Fitting box (screwdrivers, hex drivers)	1	
Electrical box	1	
Coverall and sprinkler (disinfect)	1	
30 ft power extension cord	4	
CFP and crossover cable	1	
Dilutor and manual	1	
H ₂ S gas and regulator	1	
Manuals and checklist (TEOM, INNOVA)		
Power tool and drill bits	1	

Checklists of Needed Items for Between Flock Audits

☐ Thermocouple check (unit: °C)										
Time	Location	Temp 1/Standard	Temp 2/Standard	Temp 3/Standard						

Check air flow rate in sample lines and solenoid valve

Time	Temp., °C	Location	Flow rate, LPM	Valve
		SW1		
		SW3		
		Tunnel		
		Ambient		

Gal-gas injection from sampling port

Time	Temp., °C	Location	Flow rate, LPM	NH ₃ Concentration, ppm
		SW1		
		SW3		
		Tunnel		
		Ambient		

Leak test the TEOMs

Time	Temp., °C	Location	TEOM SN	Main Flow rate, LPM	Aux Flow rate, LPM

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API 10	1E check		
Time	Items	Unit =	Notes
	Ambient pressure, (mm Hg)		
: :	Sample airflow (L/min)		
: :	Zero air applied		Cylinder P: psi
: :	H ₂ S reading		
: :	H ₂ S (ppb) applied		Cylinder P: psi
: :	H ₂ S reading		
: :	New calibration	Yes/ _{No}	
: :	Zero air applied		
: :	H ₂ S applied (ppb)		
: :	Finish calibration	Yes/ _{No}	
: :	Time switch off		
: :	Connect analyzer back to sampling system		
Note:			

D Polytro I calibration

Time	Items	Unit =	Notes
	Ambient pressure, (mm Hg)		
: :	Sample airflow (L/min)		
: :	Zero air applied		Cylinder P: psi
: :	H₂S reading		Calibration
: :	H ₂ S (ppb) applied		Cylinder P: psi
: :	H₂S reading		Calibration
: :	New calibration	Yes/ _{No}	Calibration
: :	Finish calibration	Yes/ _{No}	
: :	Time switch off		
: :	Connect analyzer back to sampling system		
Note:			

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Innov:	a check		
Time	Items	Unit =	Notes
	Ambient pressure, (mm Hg)		
: :	Sample airflow (L/min)		
: :	Zero air applied		Cylinder P: psi
: :	NH ₃ reading		
: :	CO ₂ reading		
	N ₂ O reading		
	CH₄ reading		
	Propane reading		
: :	CO ₂ (ppm) applied (with Nafion tubing)		Cylinder P: psi
: :	CO ₂ reading		
: :	NH ₃ (ppm) applied		Cylinder P: psi
: :	NH ₃ reading		
: :	CH ₄ (ppm) applied		Cylinder P: psi
	CH₄ reading		
: :	Propane (ppm) applied		Cylinder P: psi
	Propane reading		
: :	N ₂ O (ppm) applied		Cylinder P: psi
	N ₂ O reading		
: :	New calibration	^{Yes} / _{No}	
: :	Zero air applied		
: :	Zero humidity air applied		
: :	NH ₃ applied		
: :	CO ₂ applied (with Nafion tubing)		
: :	Propane applied		
: :	CH₄ applied		
: :	N ₂ O applied		
: :	Download to bank	Yes/ _{No}	
: :	Time switch off		
: :	Connect analyzer back to sampling system		
Note:			

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VIG 2	00 check			
Time	Items		Unit =	Notes
	Ambient pressure, (mm Hg)			
: :	Sample airflow (L/min)			
: :	Zero air applied			Cylinder P: psi
: :	Total reading			
: :	CH₄ reading			
: :	Propane reading			
: :	CH ₄ (ppm) applied			Cylinder P: psi
	CH₄ reading			
: :	Propane (ppm) appli	ed		Cylinder P: psi
	Propane reading			
: :	New calibration		Yes/ _{No}	
: :	Zero air applied			
	Total adjustment:	CH ₄ adjustment:	Propa	ane adjustment:
	Total reading:	CH ₄ Reading:	Propa	ane reading:
: :	CH ₄ applied			
	Total adjustment:	CH ₄ adjustment:	Propa	ane adjustment:
	Total reading:	CH ₄ reading:	Propa	ne reading:
: :	Propane applied			
	Total adjustment:	CH ₄ adjustment:	Propa	ane adjustment:
	Total reading:	CH ₄ reading:	Propa	ne reading:
: :	Finish calibration		^{Yes} / _{No}	
: :	Time switch off			
::	Connect analyzer back to	sampling system		
Note:				

Check all solenoid valves (On/Off)										
SV#	NO1	NC1	NO2	NC2	NO3	NC3	NO4	NC4		

Fan current switch check

Fan #	SW1	SW2	SW3	SW4	T1	Т2	Т3	Т4	Т5	Т6	Т7	Т8	Т9	T10

Check airflow rate at 20% of the exhaust fans (randomly selected)

	Flow rate, cfm (previous calibration)				Flow rate, cfm				
Fan #	0.04"	0.08"	0.12"	0.16"	0.04"	0.08"	0.12"	0.16"	Diff,%

C Replace any critical spare parts utilized during the previous flock

Part	Description	Note

Collect copies of calibration and event log sheets from previous flock and submit to Lara Moody for archiving

Collect litter samples and deliver to ISU for analysis