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SOLID SEPARATION EQUIPMENT VERIFICATION TESTING PROTOCOL

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ABSTRACT

This paper describes a protocol for the evaluation of solids separation technology for the U.S. Environmental Protection Agency's Environmental Technology Verification (ETV) Program. Verification activities for solids separation technologies shall be conducted under the ETV Water Quality Protection Center, which is overseen by the U.S. Environmental Protection Agency (USEPA) and NSF International (NSF), with technical assistance and oversight from North Carolina State University (NCSU).

EPA instituted the ETV Program to verify the performance characteristics of commercial-ready environmental technologies through the evaluation of objective and quality-assured data. The general test plan described here is designed to determine the effectiveness of a solid separation device in separating solids from liquid swine waste from the NCSU farm with between approximately 0.5% and 1.0% total solids over a period of four weeks. The verification process is therefore limited in scope to waste from a specific farm and of limited solids content. While the four week verification period allows observation of some operational problems, it is expected that not all maintenance problems will manifest themselves. Within these limits, the verification process provides a means to evaluate various solid separation systems under similar conditions.

Although the primary purpose of solid separators is to remove and recover solid material, the implementation of these systems will have an impact on the entire waste management system. It is therefore necessary to quantify the effect this equipment has on the partitioning of other waste constituents of interest such as nitrogen, phosphorus, potassium, copper, and zinc. Technical professionals will need this information to determine the value of the separated material as well as to design subsequent waste treatment and land application operations.

The overriding principle of this test is an accounting of the mass of each waste stream and its constituents. The critical requirement is measurement of both volume and concentration for each component of interest. Therefore, tests will be run using a fixed volume of wastewater from a

mixing tank. In this way, the partitioning of the constituents between the liquid and solid phases can be determined. Twelve samples will be collected over a four-week period.

KEYWORDS. Swine, solid separation, nutrient partitioning

INTRODUCTION

Animal waste is becoming a major concern with respect to ground and surface water quality throughout the country. This is evident from the recent proposed rules developed by the U.S. Environmental Protection Agency (USEPA) on managing manure nutrients (USEPA, 2002). Although waste from all confined animal species is a concern, swine waste presents unique challenges because of the volume of liquid used in many production systems. Many alternative swine waste treatment systems are being developed and tested around the country and many of these systems include separation of manure solids from flushed waste. Solid separation not only reduces the organic load to the treatment system but also represents an opportunity for recovery of nutrients and materials. Many swine producers have been contacted by vendors of solid separation technologies claiming to remove large amounts of nutrients and organic matter. One reason producers have been reluctant to adopt these technologies is uncertainty concerning these performance claims.

USEPA instituted the ETV Program to verify the performance characteristics of commercial-ready environmental technologies through the evaluation of objective and quality-assured data. Managed by USEPA's Office of Research and Development, ETV was created to substantially accelerate the entrance of innovative environmental technologies into the domestic and international marketplaces. ETV provides purchasers and permitters of technologies with an independent and credible assessment of the technology they are purchasing or permitting, thereby reducing financial risk associated with the selection of technologies for specific applications and facilitating technology authorization. Technology manufacturers benefit from increased acceptance of their products arising from a USEPA-reviewed, independent report supporting their claims. Participation on the part of technology manufacturers is strictly voluntary.

The Water Quality Protection (WQP) Center was created by the USEPA to evaluate the performance of technologies designed to protect ground and surface waters. Operated by NSF International, the WQP Center will reduce the financial risk to technology purchasers, and provide reliable data to assist consulting engineers in specifying a technology and regulatory agencies in approving use of a technology. Solids separation for flushed swine waste is one of the key technology areas undertaken by the Center. A test protocol has been developed for these technologies (Environmental Technology Verification Water Quality Protection Center, 2002). The testing is being conducted at North Carolina State University, and test results are posted on the USEPA and NSF web sites. This paper presents details of the testing protocol and the testing facilities at North Carolina State University.

TESTING LOCATION

Verification testing shall be conducted at the North Carolina State University Lake Wheeler Road Field Laboratory Swine Educational Unit. This farm is designed and operated as a research and teaching facility. The farm capacity is 250 sows for farrow to wean and can finish approximately half of the pigs weaned each year. Flush waste routinely flows to an anaerobic lagoon for treatment and flush water is recycled from the lagoon. Wastewater can be diverted to a 9,460 L (2,500 gal) glass-lined tank with a sloped bottom and bottom drain. The tank is 3.7 m (12 ft) in diameter and 3.0 m (10 ft) deep. This tank is equipped with a 3.7 kW (5 hp) mixer with a 0.61 m (2 ft) diameter impeller. The size and mixing speed were selected to keep solids suspended with minimum turbulence so aeration and physical changes to the manure are minimized. The system is also designed to provide sufficient wastewater for testing with characteristic concentrations that are typical of those encountered in the industry. Waste will be left uncollected in the underfloor pits for two days before flushing. By using less flush water with more waste, the concentration of waste constituents will be increased. To eliminate problems and errors associated with flow measurement and sampling, the entire quantity of waste generated over this two-day period will be collected in the glass-lined tank. This entire volume of wastewater will be sent to the test unit. In the same manner, the entire quantity of liquid that is discharged from the test unit will be collected in the 11.4 m^3 (3,000 gal) effluent tank and the entire quantity of separated solids will be collected on the adjacent concrete pad. Figure 1 shows the influent and effluent tanks used at the test site.

OBJECTIVES

Although the primary purpose of solid separators is to remove and recover solid material, the use of this equipment will have an impact on the entire waste management system. It is therefore necessary to quantify the effect this equipment has on the partitioning of other waste constituents of interest such as nitrogen, phosphorus, potassium, copper, and zinc. Power consumption shall also be verified as an important component of equipment performance as will qualitative operation and maintenance requirements.

Specific objectives of the verification test are to:

- Determine the separation efficiency with regard to the mass of solids;
- Characterize the separated solids and resulting liquid stream with respect to nutrients, metals, and pathogen indicators;
- Gather qualitative operation and maintenance requirements of the system.

Figure 1. Influent and effluent tanks at the NCSU test site.

EXPERIMENTAL DESIGN

Evaluation Criteria

The mass balance is the basis for the evaluation. The parameters to be measured represent the constituents of interest in solid separation operations. Critical parameters to be determined in liquid streams include pH, conductivity, total organic carbon, total ammoniacal nitrogen, TKN, total phosphorus, soluble phosphorus, potassium, total solids, total suspended solids, volatile solids, chloride, copper, and zinc. The same parameters shall be measured in the recovered solids with the exception of conductivity, total organic carbon, TKN, and total suspended solids. In lieu of total organic carbon and TKN measurements in the recovered solids, total carbon and total nitrogen shall be determined by the combustion method. In addition, bulk density shall be determined. Because chloride is conserved through solid separation operations, it is included as a check on the mass balance. In addition to the parameters for mass balance determination, one sample per week will be taken from the influent, effluent, and recovered solids for the determination of E. coli bacteria as an indication of the pathogen load.

Operation and maintenance requirements of the equipment during the test period shall be evaluated on a qualitative basis. Important considerations such as ease of cleaning, time required for cleaning, ease of performing inspections, frequency of operational problems during testing,

completeness of the Operations & Maintenance Manual, and extent of required operator oversight will be considered.

System Operation

Prior to testing, each separation device shall be operating properly as determined by observation of the vendor. The set up period shall be limited to five days to accommodate scheduling and to ensure the site and the ETV program are not used for additional vendor research and development on the separation device. The schedule during the start-up period will be adjusted to accommodate the installation of the unit and initiation of its operation.

The test period for the verification of solids separation technologies under this test plan is 33 days, consisting of a maximum of five days for technology set up and determination by the vendor of valid operation and 28 days of testing. Sampling and evaluation procedures shall be carried out three days per week (Monday through Friday) for a period of at least four weeks of valid operation. A total of 12 samples of the influent, effluent, and recovered solids shall be collected, one set on each of the twelve sampling days during the verification period. Valid operation means that procedures and equipment are operating correctly (pumps working, hoses intact, waste flowing) but is not an indication of technology performance. Interruption of testing for inclement weather or any other reason shall not automatically invalidate the verification test.

For safety considerations, two NC State personnel shall be present during each testing operation. During the evaluation period, wastewater from the university's farm shall be diverted to the influent-mixing tank. Generally, waste from the same houses shall be used throughout the verification test. Floating solids will be excluded (expanded metal screen) because they are characteristic of sows rather than finishing pigs, which are the source of most of the flushed swine waste in production systems. Exclusion of the floating solids also allows more effective mixing in the influent tank with minimum agitation to minimize parameter changes and provide a homogeneous wastewater for determining mass input to test equipment. The testing shall be done as a batch process. After no more than 30 minutes in the influent-mixing tank, the wastewater shall be pumped to the separator at the vendor-recommended flow rate for the verification test. A 5.6 kW (7.5 hp), 208 volt, centrifugal pump shall be used, capable of passing solids up to 3.8 cm (1.5 in) in diameter. Liquid effluent shall be collected in the effluent collection tank. All of the solids shall be collected below the discharge apparatus of the unit in a separate container of known weight. Measurements shall include total volume of wastewater entering the unit, total volume of wastewater leaving the unit, total weight of solids recovered from the unit, and concentrations of each measured component. The volume entering and leaving the unit shall be based on the dimensions of the mixing tank and the receiving tank. The solids shall be collected in large containers and the total weight of solids shall be determined using appropriate scales at the testing location. Concentrations of the quality parameters shall be determined by laboratory analysis.

The daily operation of the verification test will be consistent to the extent possible. At the start of each test day, the separator shall be inspected for unsafe conditions such as loose motor mounts or broken chains. If the daily inspection indicates an unsafe condition, the unit shall not be started until that condition is corrected. Once the inspection is complete, wastewater flow shall be started and the unit shall begin operation.

After the last test is complete the unit shall be thoroughly cleaned with a water hose. The weight of solids removed during this cleaning shall be recorded as will the time required to clean the unit. A narrative description of the cleaning process, including any difficulties, shall be included in the report.

METHODS

Sampling Methods

Triplicate samples shall be taken from the mixing tank for influent samples just before pumping to the separator begins and from the collection tank for liquid effluent samples after mixing for ten minutes after processing by the separator is complete. After mixing of the separated solids for five minutes, triplicate samples shall be taken from the mass of recovered solids. Influent and effluent samples shall be taken using separate sampling containers approximately 0.61 m (2 ft) below the wastewater surface. The sample(s) shall be transferred immediately to a labeled plastic sample bottle provided by the laboratory. Each replicate shall be analyzed as an independent sample and the results averaged. Duplicate analyses for QA/QC purposes shall be taken from the same sample bottle at the laboratory by laboratory staff.

Representative samples from the separated solids shall be produced by dividing the solids into quarter sections and mixing alternate sections. This process will be repeated at least three times. Samples (at least 50 g) shall be taken with a shovel from three different locations within the stacked solids. The mass of the solids samples shall be recorded from the on-site scales.

All samples shall be iced and transported to the laboratory by NCSU staff within one hour after the last sample of a day's test has been collected. For the standard parameters listed above, no preservation methods are necessary if sample analyses commence within twenty-four hours of sample collection. All samples shall be processed within the holding times recommended in *Standard Methods for the Examination of Water and Wastewater (19th ed.)* (Clesceri et al., 1995). Unused samples shall be held in refrigerated storage in the laboratory until the QA/QC checks are completed by the laboratory manager.

Analytical Procedures

The Environmental Analysis Laboratory of the Biological & Agricultural Engineering Department at North Carolina State University shall perform all analyses. Analytical methods used shall be those methods routinely used by the laboratory. These procedures are based on USEPA methods

and *Standard Methods for the Examination of Water and Wastewater (19th ed.)*, as modified by the laboratory to accommodate differences in solids content and flow characteristics between water and animal wastewater. These methods are referenced in Table 1. The analytical methods employed by the Environmental Analysis Laboratory differ from USEPA methods and *Standard Methods* only in the sizes of some pump tubes and dialyzer, and, in the case of TKN, a reduction in the amount of HgO (from 8g to 1g) used to prevent coating of the flow cells. The determination of bulk density of separated manure solids differs from that of soil only in that the manure solids are not dried at 105°C; the bulk density is determined as is.

Table 1. Analytical methods. SM refers to *Standard Methods* procedures; EPA refers to USEPA procedures.

Parameter	Liquid Method Reference	Solid Method Reference	Preservative	Holding Time
TS/Moisture Content	EPA 160.3	EPA 160.3	Refrigerate	7 d
TSS	EPA 160.2		Refrigerate	7 d
VS	EPA 160.4	EPA 160.4	Refrigerate	7 d
E. coli	SM9223 B	SM9223 B	None	30 h
Conductivity	SM2510		None	None
TOC	SM 5310 B		H2SO4 to pH<2	7 d
TC		AOAC 990.03	Refrigerate	7 d
TN		AOAC 990.03	Refrigerate	7 d
pH	EPA 150.1	EPA 150.1	None	2 h
NH3	SM 4500-NH3 G	Methods of Soil Analysis (1982) 84-2 as modified1	Refrigerate	7 d
Cl	SM 4500-Cl- E	Methods of Soil Analysis (1982) 84-2 as modified1	None	28 d
TKN	EPA351.2		Refrigerate	7 d
TP	SM 4500-P BC	Digestion per Soil Sci. Soc. Amer. Proc., V37, 1973. Analysis as liquid	Refrigerate	48 h
OP	SM 4500-P F	Methods of Soil Analysis (1982) 78-4.2.12	Refrigerate	48 h
Cu	SM 3111 B	Methods of Soil Analysis (1982) 78-4.2.12	HNO3 to ph<2	6 mo
Zn	SM 3111 B	Methods of Soil Analysis (1982) 78-4.2.12	HNO3 to pH<2	6 mo
K	SM 3111 B	Methods of Soil Analysis (1982) 78-4.2.12	HNO3 to pH<2	6 mo
Bulk Density		Methods of Soil Analysis (1982) 30-2.1	None	None

1 The extraction for ammonia, nitrite, and nitrate with 1.0 N KCl was modified to use 1.25 N K2SO4. This allows the analysis of chloride in the same extract according to the liquid method.

2 This method was modified according to North Carolina Department of Agriculture Methods. The extract is then analyzed according to the liquid method.

Calculations

The mass balance of each parameter shall be calculated. The mass of each parameter pumped into and out of the system shall be calculated as the volume of wastewater multiplied by the average concentration determined from the triplicate samples (Eq 1). For the separated solids, the concentration of each parameter shall be multiplied by the total mass of solids collected to give a total mass of each parameter removed in the separated solids (Eq. 2). The results of the verification test of a separator will be reported in terms of the percent of each parameter that is found in the solid and liquid phases.

The mass of each parameter into the system should equal the mass of that parameter that leaves the system in both the solid and liquid form. The reliability of the mass balance shall be determined as the magnitude of the difference between what goes into the system and what comes out of the system expressed as a percent (Eq. 3). Generally this lost mass will be less than ±10%.

$$M_i^{I,L} = V^{I,L} \times \overline{C}_i^{I,L}$$

(Equation 1)

$$M_i^{S} = M_s \times \overline{C}_i^{S}$$

(Equation 2)

$$R_i = \frac{M_i^I - (M_i^L + M_i^S)}{M_i^I} \times 100$$

(Equation 3)

Where:

$M_i^{I,L,S}$ = Mass of component i in the influent, liquid effluent, or separated solids.

M_s = Mass of separated solids.

$\overline{C}_i^{I,L,S}$ = Average concentration of component i in the influent, liquid effluent, or separated solids.

$V^{I,L,S}$ = Volume of influent, liquid effluent, or separated solids.

R_i = Percent of component i not recovered in liquid effluent or separated solids.

QUALITY ASSURANCE PROJECT PLAN

Reporting Requirements

Quality assurance / quality control (QA/QC) data will be collected using samples submitted by this project even though the Environmental Analysis Laboratory processes many samples during the workday and all samples requiring the same analysis are processed together. Quality control practices at the laboratory include blank samples, duplicate samples, and spiked samples. Water blanks (distilled/deionized) are run every 6 samples, duplicate samples are run every 10 samples, and spiked samples are run every 10 samples.

Use of Blanks

Trip blanks of lab grade distilled water shall be made using field containers for 5% of the experimental samples. A trip blank is a sample of lab grade distilled water, which is obtained during the course of collecting experimental samples, subjected to the same collection, processing, preservation, transportation, and laboratory handling procedures as an environmental sample. Blanks shall be considered acceptable if values are less than the method detection limit (MDL) or 10% of the median of all sample analysis values. If values exceed 10%, possible sources of contamination including sample containers, handling procedures, and distilled water shall be isolated and evaluated.

Measurement of Accuracy and Precision

Matrix spikes shall be prepared and analyzed every 10 samples. Recoveries between 85 and 115% of true value shall be considered acceptable. If values are outside the acceptable range, a duplicate sample shall be spiked and analyzed. If this spiked sample recovery is still outside of range, and the rest of the quality control parameters are within acceptance criteria, the spiked sample may be

labeled as having possible matrix effect. It is not suitable to spike samples for all parameters, however. Measurements of pH, conductivity, solids, and E. coli shall be conducted without spiked samples.

A duplicate sample is an aliquot of an already collected, processed, and preserved field sample, which is used as a measure of laboratory analytical precision of various constituents of the sample matrix. Duplicate samples shall be considered acceptable if the values are within 25% of the average value. If the difference exceeds 25%, the sampling method and analytical method shall be evaluated to assess ways to obtain more representative or consistent samples.

On-Site Audits

QA inspections conducted by NSF shall be formally documented in an Audit Report and submitted to the USEPA Pilot Manager, USEPA Pilot Quality Manager, and NSF Partner Manager for review.

The NSF Partner Manager, Project Coordinator, QA Director, or other qualified NSF designee shall conduct a technical system audit and a performance evaluation audit of measurement systems used in testing at least once during the verification testing period for a given technology. In addition to the daily quality control checks on the analytical data, the NCSU QA manager shall check all technical systems and measurement performance at least once during the verification testing period for each technology.

CONCLUSION

This protocol allows describes specific rigorous procedures by which the performance of solid separation equipment can be verified for use on flushed swine waste. Before a specific technology can undergo testing under the ETV program, a specific test plan document will be prepared that is based on these procedures but includes information and instructions unique to the equipment to be tested. The value of the program is in the level of detail of the test procedures, the quality control program of the laboratory, and the extensive review of data by NSF International and the USEPA. Acknowledged limitations of the program are the single species used to produce the waste, the limited solids content of the wastewater, and the lack of a long term test duration.

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