TEST PLAN FOR THE VERIFICATION OF TECHNOLOGIES FOR SEPARATION OF MANURE SOLIDS FROM FLUSHED SWINE WASTE

Prepared for:
NSF International
P. O. Box 130140
Ann Arbor, MI 48113-0140
734-769-8010
800-673-6275

with support from the
U.S. Environmental Protection Agency
Environmental Technology Verification Program

Prepared by:
North Carolina State University
Animal and Poultry Waste Management Center
Department of Biological and Agricultural Engineering
Raleigh, NC 27695

Verification Test Plan

Triton Systems Solid Bowl Centrifuge, Model TS-5000
for Separation of Manure Solids from Flushed Swine Waste

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SECTION 1 PROJECT DESCRIPTION AND OBJECTIVES

1.1 Executive Summary

This document is a test plan for the evaluation of the Triton Systems Solid Bowl Centrifuge, Model TS-5000, hereinafter referred to as the Triton Systems Solid Bowl Centrifuge. Verification activities for the Triton Systems Solid Bowl Centrifuge shall be conducted over a four-week period under the guidance of the U.S. Environmental Protection Agency's Environmental Technology Verification (ETV) Program. In particular, the evaluation will be overseen by the ETV Source Water Protection Pilot, which is cooperatively managed by the U.S. Environmental Protection Agency (USEPA) and NSF International (NSF), formerly the National Sanitation Foundation. Technical assistance during the testing process shall be provided by North Carolina State University (NCSU).

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 an USEPA-reviewed, independent report supporting their claims. Participation on the part of technology manufacturers is strictly voluntary.

The ETV Source Water Protection Pilot, one of twelve pilots initiated under the program, is guided by the expertise of stakeholder groups. Stakeholder groups consist of representatives of key customer groups for the particular technology sector, including buyers and users of technology, developers and vendors, state and federal regulatory personnel, and consulting engineers. All technology verification activities are based on testing and quality assurance protocols that have been developed with input from the major stakeholder/customer groups.

NSF International is an independent, not-for-profit organization, dedicated to public health, safety, and protection of the environment. NSF develops standards, provides educational services, and offers superior third-party conformity assessment services, while representing the interests of all stakeholders. In addition to well-established standards-development and certification programs, NSF specifically responds to and manages research projects, one-time evaluations and special studies.

NSF is the verification partner organization for three ETV programs: Drinking Water Treatment Systems, Wet Weather Flow Technologies, and Source Water Protection Technologies. The goal of the ETV Source Water Protection Pilot is to verify the performance of commercial-ready technologies used to protect ground and surface waters from contamination. Testing conducted under the ETV program using this test plan does not constitute NSF or USEPA Certification of the product tested. Rather, it recognizes that the performance of the equipment has been determined using an objective, quality-assured process. ETV reports are signed by USEPA and NSF prior to being prepared for final distribution.

This test plan is designed to determine the effectiveness of the Triton Systems Solid Bowl Centrifuge in separating solids from liquid swine waste with between 0.5% and 1.0% total solids. Separation processes partition a waste stream into two streams, a liquid phase and a solid phase. As no process is totally efficient, the liquid phase contains some solid material and the solid phase contains some amount of water. The chemical constituents in the waste will also partition between the two phases based on the physical and chemical conditions imposed by the system. Because subsequent treatment operations to process both the liquid and solid waste streams must be based on their respective characteristics, the performance of this equipment shall be determined in terms of waste constituent partitioning.

The overriding principle of this test is an accounting of all of the mass of the waste 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.

After setup by the vendor, with assistance from NCSU staff, the equipment will be run for a short time to ensure proper operation (see section 4.2.1, Start-Up Period). Verification testing will take place according to the evaluation procedures described throughout this document three times per week over a four-week period.

1.2 General Description of the Equipment

The Triton Systems Solid Bowl Centrifuge consists of a solid bowl basket centrifuge with associated control systems. The following is a summary of the characteristics of the Triton Systems Solid Bowl Centrifuge:

Size	48” Diameter x 30” Deep
Maximum RPM	1400
Maximum G’s	1300
Bowl Capacity	Sixteen (16) cubic feet
Air Pressure	80-100 psi (1 CFM required with an instantaneous surge of 20 CFM for 2 seconds to operate controls)
Type	Bottom Discharge
Weight (with drive motor)	7700 to 9560 pounds

The critical design parameters for scale-up and scale-down of the technology are the horsepower input and the surface area of the bowl.

Batches of wastewater are introduced through the curb cover to the bottom of the bowl by means of a tangential feed tube. The material should be fed into the bowl in the same direction as the bowl rotation (clockwise when viewed from above) and while the bowl is accelerating. During the separation process, the clarified liquid will overflow from the lip ring at the top of the bowl, strike the curb and flow down the curb to the discharge pipe at the bottom of the curb. As long as the feed cycle continues, the clarified liquid will continue to flow over the lip ring. A baffle is present at the top of the bowl to trap floating solids.

Some supernatant liquid and lightweight solids will be removed by the skimmer assembly, which operates at a speed equal to the feed speed. The skimmer is a combined air motor and adjustable "Hydro-Check" unit (located on the curb cover), and a skimmer tube (located inside the bowl). After the skimmer retracts, any supernatant or light solids still present will be unloaded by the plow along with the caked solids.

When solids accumulation reaches a set point, the cake will be removed at a slow bowl speed by the plow and will drop through the opening at the bottom of the bowl. The plow speed is controlled by a combined air motor and "Hydro-Check" unit (located on the curb cover) and is preset during setup.

Available support equipment for the Triton Systems Solid Bowl Centrifuge includes: 1) an optional chemical addition systems for polymer addition to improve solids separation, and 2) an optional oxygen addition system for odor control. No support equipment will be included in this verification test. The Operations Manual is included as Attachment A.

1.3 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 2,500 gal glass-lined tank of 12 ft diameter and 10 ft depth. This tank is equipped with a 5 hp mixer with a 2 ft diameter impeller. This system is designed 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. To eliminate problems and errors associated with flow measurement and sampling, the entire quantity of waste generated over a 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 effluent tank and the entire quantity of separated solids will be collected on the adjacent concrete pad.

1.4 Statement of Verification Objectives

The Triton Systems Solid Bowl Centrifuge provides solid-liquid separation for swine manure and claims to reduce nutrient levels in the effluent. Qualitative operation and maintenance requirements of the Triton Systems Solid Bowl Centrifuge will be recorded. Operation and maintenance parameters to be measured include, but are not limited to, ease of cleaning, frequency of operational problems during testing, and extent of required operator oversight. Details of the type of operating and maintenance data/observations that will be collected are listed in Section 4.1. Because the test period lasts only four weeks, the verification process does not indicate what long term operational problems are likely to occur for the technology. Power consumption shall also be verified as an important component of equipment performance.

Although the primary purpose of this equipment 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, zinc, and others. 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.

SECTION 2 PROJECT ORGANIZATION

This section defines the participants in this technology verification and their roles and responsibilities. The key organizations in the technology verification include the Verification Organization (NSF), the Testing Organization (NCSU), the Vendor (Triton Systems, LLC), and USEPA.

The technology verification shall be conducted by the Biological & Agricultural Engineering Department at NCSU, through the Animal & Poultry Waste Management Center. The Environmental Analysis Laboratory in NCSU's Biological & Agricultural Engineering Department shall conduct all component analyses.

The main NSF and USEPA contacts are:

Tom Stevens, Pilot Manager
NSF International
P.O. Box 130140
Ann Arbor, MI 48113-0140
v. 734-769-5347 f. 734-769-5195

Maren H. J. Roush, Project Coordinator
NSF International
P.O. Box 130140
Ann Arbor, MI 48113-0140
v. 734-827-6821 f. 734-769-5195

Ray Frederick, Project Officer
United States Environmental
Protection Agency
2890 Woodbridge Ave. (MS-104)
Edison, NJ 08837-3679
v. 732-321-6627 f. 732-321-6640

The main NCSU contacts are:

J.J. Classen, Associate Professor
Biological & Agricultural Engineering
Campus Box 7625
v: 919-515-6800 f: 919-515-7760

F.J. Humenik, Coordinator
Animal Waste Management Programs, CALS
Campus Box 7927
v: 919-515-6767 f: 919-513-1023

C.M. Williams, Director
Animal & Poultry Waste Management Center
Campus Box 7608
v: 919-515-5387 f: 919-513-1762

Craig Baird
Biological & Agricultural Engineering
v: 919-513-2515 f: 919-513-1023

The solids separator vendor for this technology verification is Triton Systems LLC. The Triton Systems LLC contacts for this project are:

Charles Leen, President
Triton Systems, LLC
5355 Royal Vale Lane
Dearborn, MI 48126
v: 313-347-3110 f: 313-963-1707

Kenneth B. Kyte, General Manager
Kyte Centrifuge Sales and Consulting
4901 Morton Rd.
New Bern, NC 28562
v: 252-633-5783 f: 252-633-4826

James W. Ridgway, P.E.
Environmental Consulting & Technology, Inc.
1249 Washington Blvd, Suite 3500
Dearborn, MI 48126
v: 313-963-6600 f: 313-963-1707

2.1 Roles and Responsibilities

The primary roles and responsibilities of each party shall include:

2.1.1 Verification Organization (NSF)

- Coordinate with the Testing Organization to prepare a site-specific test plan;
- Coordinate with technical reviewers and Vendor to review the test plan prior to the initiation of verification testing;
- Coordinate with USEPA and the Vendor to approve the final, site-specific test plan;
- Review the quality systems of the testing organization and subsequently, qualify the testing organization;
- Oversee the technology evaluation and associated laboratory testing;
- Review data generated during verification testing;
- Oversee the development of a verification report and verification statement;
- Print and distribute the verification report and verification statement; and

- Provide quality assurance oversight at all stages of the verification process.

2.1.2 U.S. Environmental Protection Agency (USEPA)

This test plan has been developed with financial and quality assurance assistance from the ETV Program, which is overseen by the USEPA's Office of Research and Development. The verification report generated following this technology verification will be subject to review and approval by USEPA.

2.1.3 Technology Panel

A Technology Panel was formed to assist with the development of technology test plans. The Animal Waste Treatment Technology Panel has ensured that data to be generated during verification testing are relevant and that the method of evaluation for different technologies is fair and consistent. This, and all animal waste treatment test plans, is subject to the Panel's review.

2.1.4 Testing Organization (NCSU)

- Coordinate with the Verification Organization and Vendor relative to preparing and finalizing the Test Plan;
- Conduct the technology verification in accordance with the Test Plan, with oversight by the Verification Organization;
- Analyze all influent and effluent samples collected during the technology verification process, in accordance with the procedures outlined in the Test Plan and attached SOPs;
- Coordinate with and report to the Verification Organization during the technology verification process;
- Provide analytical results of the technology verification to the Verification Organization; and
- If necessary, document changes in plans for testing and analysis, and notify the Verification Organization of any and all such changes before changes are executed.

2.1.5 Vendor – Triton Systems, LLC

- Assist in the preparation of the technology-specific test plan for technology verification and approve the final version of the test plan;
- Provide a complete field-ready version of the technology of the selected capacity for verification and assist the Testing Organization with installation at the test site;
- Provide start-up services and technical support as required during the period prior to the evaluation;
- Provide technical assistance to the Testing Organization during operation and monitoring of the equipment undergoing verification testing as requested;
- Remove equipment associated with the technology following the technology verification; and
- Provide funding for verification testing.

SECTION 3 EQUIPMENT CAPABILITIES AND DESCRIPTION

3.1 Description of Equipment

The Triton Systems Solid Bowl Centrifuge is designed as a simple and efficient method of removing suspended solids from swine and other animal waste slurries. Ancillary equipment provides the ability for chemical addition to aid in the capture of small diameter, neutrally buoyant solids. Recent research suggests that chemical addition is not needed for most applications. Similarly, an optional oxygen delivery system is available to saturate (and /or supersaturate) the liquid fraction of the discharge for both odor control and improved biological reduction. Neither the chemical addition nor oxygen addition systems will be operated during the verification testing.

A photograph of the unit is shown below.

3.2 Engineering and Scientific Concepts on which the Equipment is Based

The Triton Systems Solid Bowl Centrifuge solids separation process relies on an imperforate bowl basket centrifuge operating at up to a maximum of 1300 times the force of gravity. The high "G" force provides rapid separation of suspended solids from the slurry. Although polymer coagulants may be used with the Triton Systems Solid Bowl Centrifuge to improve its efficiency, the vendor claims that chemical addition is not necessary for most applications. No coagulants will be used during testing conducted under the ETV Source Water Protection Pilot. Because the cost of these coagulants adds to the operating costs, chemical addition is only recommended by Triton Systems when there are substantial constraints on the quality of the discharged liquid.

3.3 Nature, Production Rates, and Handling of Waste Material

The Triton Systems Solid Bowl Centrifuge can process between 20 and 75 gallons per minute, depending on solid loadings and required separation performance. The verification test will be conducted at a flow rate of 35 gallons per minute.

3.4 Limitations of the Equipment

The Triton Systems Solid Bowl Centrifuge is designed to remove the suspended solids fraction of the waste stream. As such, it cannot reduce soluble constituents in the wastewater. The actual removal efficiency for BOD will depend on the ratio of soluble to non-soluble BOD in the influent slurry.

SECTION 4 EXPERIMENTAL DESIGN

4.1 Quantitative and Qualitative Evaluation Criteria

The sampling procedures defined below shall provide the information needed to complete a mass balance on influent and effluent constituents. 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, ammonia, TKN, total phosphorus, soluble phosphorus, total solids, total suspended solids, volatile solids, chloride, potassium, copper, zinc, and E. coli bacteria. The same parameters shall be measured in the recovered solids with the exception of conductivity, total organic carbon, 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. The recovery of chloride represents the maximum recovery of parameters in a given operation or test.

Power consumption by the technology shall be verified, as it is an important component of equipment performance. Current and voltage will be monitored by an Extech, Model 380940 clamp-on power datalogger that also measures voltage. This is a digital device that is calibrated at the factory and does not require field calibration. The time of operation will be determined by start and stop times recorded as part of the daily log. From this information, the total power will be calculated.

In addition, operation and maintenance requirements of the Triton Systems Solid Bowl Centrifuge 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 O&M Manual, and extent of required operator oversight will be considered.

Control system features shall be verified during the test period. The emergency stop pushbutton shall be activated during operation of the Triton Systems Solid Bowl Centrifuge to verify that it disables the drive/motor. Once the emergency stop button is activated, the machine should coast to a stop, according to a May 13, 2002 memo from Specialty Controls, Inc., manufacturer of the control systems for the centrifuge, included as Appendix B.

Power to the Triton Systems Solid Bowl Centrifuge drive system shall be interrupted to verify that the PLC and drive system will subsequently shut down, as designed. It shall also be verified that machine cycling will not resume automatically once power is restored without operator intervention.

The basket cover closed limit switch and unbalance gyration limit, both of which are supervised by a programmable logic controller, shall be verified during the testing phase. Having the cover open is supposed to preclude starting a cycle. Likewise, the centrifuge is designed so that opening the cover while the machine is in operation will stop the cycle and announce a fault. As possible, the NCSU test site staff shall determine whether an out of balance condition will also interrupt the system and annunciate a fault condition.

System alarms that go off during the verification test shall be noted. It shall be verified that where possible, alarm conditions must be corrected (acknowledged/reset by pressing the appropriate function key as indicated on the display) for the text alarm message to disappear.

4.2 Verification Testing Phases

4.2.1 Start-Up Period

Prior to testing, the Triton Systems Solid Bowl Centrifuge 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 ensure the site and program is not used for research and development. Test site personnel shall determine the specific time of day for sampling, considering availability of staff, access to the analytical laboratory, availability of vendor personnel, and the needs of the farm staff. The schedule during the start-up period will be adjusted to accommodate the installation of the unit and initiation of its operation. NCSU personnel will be available on the scheduled arrival date to facilitate set up of the unit and ensure proper connection of piping. Samples for which results are required prior to the start of testing must be transported to the laboratory before the end of business on the second day of set up, as samples taken to the laboratory after that time might not be processed prior to the start of the verification testing.

The rotational speed of the centrifuge during verification testing will be based on data generated from samples collected during the start up phase of the unit and analyzed by the NCSU laboratory.

Addendum: Based on the analytical results of the samples collected during the start-up period, the vendor has indicated that the speed of the bowl should be 1200 rpm (as the unit was set during the set-up period) and that the feed rate shall be 35 gpm.

4.2.2 Operational Phase of Verification Testing

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 and effluent 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.

For safety considerations, two NC State personnel shall be present during each testing operation. During the evaluation period, wastewater from the 250 sow farrow-to-finish unit shall be diverted to the influent-mixing tank. Floating solids shall be excluded because they are not characteristic of most stages of swine production. 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 7.5 hp, 460 volt, centrifugal pump shall be used, capable of passing solids up to 1.5 inches 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 volume, which can be weighed. 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 will 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.

If at all possible, verification testing will begin on a Monday. The daily operation of the verification test will be consistent to the extent possible. Testing will take place in the morning hours unless circumstances dictate otherwise. The centrifuge will be inspected according to the daily schedule listed below. The status of the unit will be recorded on the daily log sheet and the unit will be started. If the daily inspection indicates an unsafe condition, the unit will not be started until that condition is corrected. After it has been determined that the daily test can proceed, the wastewater will be collected and mixed.

Daily maintenance and inspections, listed below, will be performed prior to unit start up. Once the inspections are complete, the unit will be started. Because the operational speed is preset in the control panel, no further adjustments are necessary. An indicator light will show when the unit is at the proper speed to begin waste feed. The motor speed will be verified by checking the motor speed indicator each time the speed changes for the next function. Wastewater flow will then be started.

The centrifuge to be verified has a greater capacity for solids accumulation than can be accommodated by the proposed operation of the test site. It will therefore not be possible to operate the solid bowl centrifuge in a continuous mode. During each day of operation, the entire contents of the mixing tank will be pumped into the centrifuge unit operating at the preset feed speed. When wastewater flow stops, the unit will maintain this speed for one hour prior to skimming. The skimming will then be manually initiated. Following skimming, turning a manual switch will slow the centrifuge bowl to the preset plow speed. An indicator light will show when this speed is reached and the plowing will then be initiated manually. The plowing procedure is then repeated. Once all effluent and solids have been removed from the discharge points, the unit will be shutdown. Any remaining solids that fall out of the unit prior to the next test date will be collected and the mass recorded. A sample will be taken to the laboratory for moisture content determination. The times at which the unit is started and the times at which the centrifuge reaches operating/skimming speed and plow speed will be recorded.

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

Inspection and Maintenance Checks	Frequency *
Check speed setting in control panel	Daily
Drain filter reservoir on the "Lubri-Air Control Unit"	Daily
Check air pressure reading	Daily
Check for air leaks at connections	Daily
Adjust lubricator oil feed adjustment screw	Initially
Check alarms/limit switches/meter relays/air pressure cutoffs	Initially

* The term "daily" refers to the days of the week during which technology operation and verification testing take place.

Evaluation of operation and maintenance requirements and control system features of the Triton Systems Solid Bowl Centrifuge shall be conducted during the verification test period, as described in Section 4.1.

4.3 Sampling Methods

Triplicate samples shall be taken from the mixing tank for influent samples just before pumping to the Triton Systems Solid Bowl Separator begins and from the collection tank for liquid effluent samples after mixing for ten minutes (mixing shall be initiated once processing by the Triton Systems Solid Bowl Separator is complete). After mixing of the separated solids for five minutes, triplicate samples shall be taken from the mass of recovered solids. Each replicate shall be analyzed as an independent sample and the results averaged. Influent and effluent samples shall be taken using separate sampling containers of at least 500 mL capacity suspended on a pole, if needed, approximately two feet below the wastewater surface. The sample(s) shall be transferred immediately to a labeled plastic sample bottle provided by the Environmental Analysis Laboratory. 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 Environmental Analysis 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.)*. Unused samples shall be held in refrigerated storage in the Environmental Analysis Laboratory until the QA/QC checks are completed by the laboratory manager. Table 4-1 lists the constituents that will be measured in the influent, effluent, and solid samples collected on each day of operation. Table 4-1 also lists the analytical methods and preservation/holding times for each parameter to be measured.

Each sample container shall be labeled with the vendor name, sample location, date, time, replicate number, and name/initials of the person collecting the sample. Daily sampling records shall also be maintained, recording sample location, date and time of sampling, replicate number, type of sample(s) (influent, effluent, solids), and name/initials of the person collecting the sample. Sampling records shall be forwarded to the Verification Organization (via facsimile or overnight carrier) at the completion of each week of testing.

4.4 Supplemental Analyses

Other parameters considered non-critical may be added at the discretion and expense of the vendor. These may include, but are not limited to, iron, magnesium, calcium, alkalinity, COD, and sulfate. There will be no supplemental analyses performed for the evaluation of the Triton Systems Solid Bowl Centrifuge.

4.5 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. The methods are referenced in Table 4-1. SM refers to *Standard Methods* procedures; EPA refers to USEPA procedures. Detailed operating procedures are maintained by the testing organization.

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^{o}C; the bulk density is determined as is. A 50 mL beaker is filled to the top with the separated solids without packing and leveled. The total weight is recorded. The tare weight of the beaker is subtracted from the total weight and divided by 50 mL. The determination is made three times and the average recorded. Results are expressed as g/mL.

Table 4-1 Analytical Methods

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		H_2SO_4 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
NH_3	SM 4500-NH_3 G	Methods of Soil Analysis (1982) 84-2 as modified[1]	Refrigerate	7 d
Cl	SM 4500-Cl^- E	Methods of Soil Analysis (1982) 84-2 as modified[1]	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) 78-4.2.1[2]	Refrigerate	48 h
Cu	SM 3111 B	Methods of Soil Analysis (1982) 78-4.2.1[2]	HNO_3 to ph<2	6 mo
Zn	SM 3111 B	Methods of Soil Analysis (1982) 78-4.2.1[2]	HNO_3 to pH<2	6 mo
K	SM 3111 B	Methods of Soil Analysis (1982) 78-4.2.1[2]	HNO_3 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 K_2SO_4. 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.

4.6 Mass Balance 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 and by the average bulk density of the solids to give a total mass of each parameter removed in the separated solids (Eq. 2).

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 validity 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).

$$M_i^{I,L} = V^{I,L} \times \overline{C}_i^{I,L} \qquad \text{(Equation 1)}$$

$$M_i^S = M_s \times \overline{C}_i^S \qquad \text{(Equation 2)}$$

$$R_i = \frac{M_i^I - (M_i^L + M_i^S)}{M_i^I} \times 100 \qquad \text{(Equation 3)}$$

Where:

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

M_s = Mass of recovered 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.

$\overline{\rho}_B$ = Average bulk density of separated solids.

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

SECTION 5 QUALITY ASSURANCE PROJECT PLAN

The Quality Assurance Project Plan for the ETV consists of the following components:

- Measurement of precision and accuracy
- Methodology for use of blanks
- Performance evaluation samples
- Duplicate samples
- Data correctness
- Calculation of data quality indicators
- Data reporting
- Corrective action plan
- An on-site audit may be conducted during the evaluation.

The project plan QA manager is Dr. John J. Classen. The manager of the Environmental Analysis Laboratory is Ms. Rachel Huie. The sampling manager is Mr. Craig Baird.

5.1 Reporting Requirements

It should be noted that since the Environmental Analysis Laboratory processes many samples during the workday, all samples requiring the same analysis are processed together. However, required QA/QC data will be obtained using samples submitted by this project.

Table 5-1 summarizes the reporting requirements for each parameter analysis performed during this Environmental Technology Verification testing period.

Table 5-1 Reporting Requirements

Parameter	Matrix	Units	Reporting Limits
Total Solids/Moisture Content	Liquid, Solid	mg/L, µg/g	1 mg/L, 1 µg/g
Total Suspended Solids	Liquid	mg/L	1 mg/L, 1 µg/g
Total Volatile Solids	Liquid, Solid	mg/L, µg/g	1 mg/L, 1 µg/g
E.coli Bacteria	Liquid, Solid	MPN/ 100 mL, MPN/g	2 MPN/100 mL, 2 MPN/g
Conductivity	Liquid	mg/L	1 mg/L
Total Organic Carbon	Liquid	mg/L	1 mg/L
Total Carbon	Solid	%	0.1%
Total Nitrogen	Solid	%	0.1 %

Parameter	**Matrix**	**Units**	**Reporting Limits**
pH	Liquid, Solid	pH units	0.1 pH unit
Ammonia	Liquid, Solid	mg/L, µg/g	1 mg/L, 1 µg/g
Chloride	Liquid, Solid	mg/L, µg/g	1 mg/L, 1 µg/g
Total Kjeldahl Nitrogen	Liquid	mg/L	1 mg/L
Total Phosphorus	Liquid, Solid	mg/L, µg/g	1 mg/L, 1 µg/g
Ortho Phosphorus / Available Phosphorus	Liquid, Solid	mg/L, µg/g	1 mg/L, 1 µg/g
Copper	Liquid, Solid	mg/L, µg/g	1 mg/L, 1 µg/g
Zinc	Liquid, Solid	mg/L, µg/g	1 mg/L, 1 µg/g
Potassium	Liquid, Solid	mg/L, µg/g	1 mg/L, 1 µg/g
Bulk Density	Solid	g/mL	1 g/mL

5.2 Methodology for Use of Blanks

Quality control practices at the laboratory include duplicate samples, spiked samples, and blank 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.

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. See Appendix A.

5.3 Measurement of Accuracy in a Matrix Spike Sample

Matrix spike samples will be prepared from samples obtained during this ETV project. The 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 is 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 will be conducted without spiked samples.

5.4 Measurement of Precision

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 each other. If the values exceed 25% difference, the sampling method and analytical method shall be evaluated to assess ways to obtain more representative or consistent samples.

5.5 External Quality Control Checks

After initial calibration, the laboratory shall analyze an external quality control check. This check shall be prepared form a source other than that from which their initial calibration standards were prepared.

5.6 Data Correctness

Data correctness refers to data quality, for which there are four indicators:

- Representativeness
- Completeness
- Accuracy
- Precision

5.6.1 Representativeness

As specified by NSF, representativeness of samples for the ETV shall be ensured by executing consistent sample collection procedures, including sample locations, timing of sample collection, sampling procedures, sample preservation, sample packaging, and sample transport. Each of these items was addressed above in the experimental procedures.

5.6.2 Completeness

Completeness refers to the amount of data collected from a measurement process compared to the amount that was expected to be obtained. For this ETV test plan, completeness refers to the proportion of valid, acceptable data generated using each method. The completeness objective for data generated through this test plan is 85%, meaning that at a minimum, 85% of the data identified in Section 4 shall be reported at the conclusion of testing.

5.6.3 Accuracy

Accuracy of test parameters is addressed in Sections 5.3 and 5.7.3.

5.6.4 Precision

Precision of test parameters is addressed in Sections 5.4 and 5.7.1.

5.7 Calculation of Data Quality Indicators

5.7.1 Precision

As specified in *Standard Methods* (Method 1030 C), precision is specified by the standard deviation of the results of replicate analyses. The overall precision of a study includes the random errors involved in sampling as well as the errors in sample preparation and analysis.

$$\text{Standard Deviation} = \sqrt{\frac{\sum (x_i - \bar{x})^2}{n-1}} \qquad \text{(Equation 4)}$$

Where:

$\bar{x}$ = sample mean

x_i = ith data point

n = number of data points

5.7.2 Relative Percent Deviation

For this pilot, duplicate samples shall be analyzed to determine the overall precision of an analysis using relative percent deviation. All parameters measured in the Environmental Analysis Laboratory shall be subjected to duplicate analysis as described in Section 5.4.

$$Relative\ Percent\ Deviation = \left[\frac{|x_1 - x_2|}{\bar{x}}\right] \times 100 \qquad \text{(Equation 5)}$$

Divide the absolute standard deviation by the value of the mean and then multiply by 100.

5.7.3 Accuracy

Accuracy is quantified as the percent recovery of a parameter in a sample to which a known quantity of that parameter was added. An example of an accuracy determination in this pilot is the analysis of ammonia in a sample to which a standard quantity has been added.

$$Percent\ Recovery = \left[\frac{(x_{known} - x_{measured})}{x_{known}}\right] \times 100 \qquad \text{(Equation 6)}$$

5.7.4 Completeness

$$Completeness = \left(\frac{n_{valid\ and\ acceptable}}{n_{total}}\right) \times 100 \qquad \text{(Equation 7)}$$

5.8 Data Reporting

Data reports shall be prepared by the testing organization, North Carolina State University. Reports shall include date and time of analysis for each sample, including quality assurance samples, daily results of mass balance calculations, percent removal of each parameter of interest, the time of day of each sample event, and any observations by NCSU personnel related to the operation of the technology, i.e. power consumption, chemical use, and maintenance issues. Data reports and laboratory bench sheets shall be copied and faxed to NSF International weekly.

5.8.1 Project Reports

A project report shall be prepared by Dr. John Classen within thirty (30) days of completion of testing. Upon satisfactory review and revision by other NCSU staff, this report shall be sent to NSF and Triton Systems, LLC. Data presented to the Vendor in the project report prepared by

NCSU is not considered ETV verification data until NSF and USEPA have reviewed the data and approved the final verification report and verification statement.

5.8.2 Audit Reports

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 a given technology.

5.9 Corrective Action Plan

Table 5-2 Corrective Action Plan

Parameter	Acceptance Criteria	Sequence of Steps for Corrective Action
Any duplicate analysis	RelativePercent Deviation ≤ 25%	• Re-sample duplicates • Check instrument calibration and recalibrate according to lab procedures
Any blank sample	Below method detection limits	• Perform procedures specific to each analysis to produce stable baseline
Any matrix spike sample	Within recovery limits of 85 to 115%	• Check and verify all steps in sample collection and analysis • Repeat performance evaluation sampling and analysis; or send to another approved laboratory for verification. If still out and laboratory fortified blank or external QC check is in, then it's caused by matrix interference.
Any external QC check or continuing calibration standard	Within recovery limits of 90-110%	• Stop the run • Check instrument optimization • Check preparation of calibration standards or the preparation of the external qc check • Check calibration and recalibrate if necessary • Rerun all affected samples

SECTION 6 DATA REPORTING, DATA REDUCTION AND DATA VALIDATION

Field personnel from NCSU are responsible for measurement and reporting of liquid volume and solid mass determinations. The Environmental Analysis Laboratory is responsible for all other analytical measurements. Results of analytical tests as well as QA/QC tests shall be delivered to the main contact at NCSU, Dr. John J. Classen, who shall be responsible for calculating the mass flow of each parameter. Dr. Classen is also responsible for conducting and reporting all statistical calculations and results. Data summary sheets, including all raw and reduced data, shall be submitted to NSF International along with the final report. The Environmental Analysis Laboratory shall maintain hard and electronic copies of raw data for at least five (5) years. NCSU shall have no responsibility for data storage after that time. Permanent data and report storage shall be the responsibility of NSF International or USEPA.

The Verification Report shall be a comprehensive document containing all raw and analyzed data, all QA/QC data sheets, a description of all types of data collected, a detailed description of the testing procedure and methods, results and QA/QC results.

A recommended Table of Contents for the Verification Report is as follows.

- Preface
- Glossary
- Acknowledgements
- Executive Summary
- Introduction and Background
- Procedures and Methods Used In Testing (summarizing essential information from the Test Plan)
- Results and Discussion
- Limitations
- Conclusions
- Recommendations
- References
- Appendices (including raw data)

The exact details of the Verification Report shall be agreed upon by NSF and the party(ies) responsible for developing the Verification Report.

SECTION 7 SAFETY CONSIDERATIONS

This section describes safety considerations appropriate for the equipment being tested. The key safety considerations for this pilot are electrical hazards, mechanical hazards, and pathogenic organisms associated with wastewater. Precautions shall be taken based on standard practices for these hazards: power shall be disconnected prior to servicing equipment, all shafts and bearings shall be covered or enclosed, and care shall be taken in sample collection to protect the health of NCSU staff. Details of safety precautions are included in the safety plan for the Lake Wheeler Road Field Laboratory Swine Educational Unit and are available at the main office of the Unit. Safety measures specific to the Triton Systems Solid Bowl Separator are described in the separator's O & M manual, included in Appendix A. All personnel involved in this project shall review and sign the safety plan prior to working on the site. To ensure personal safety, two NC State employees will be present during testing of each technology as designated by the principal investigator.

All applicable safety rules of the University shall be enforced by the Testing Organization for the duration of the verification test. In particular, laboratory operations shall be in accordance with the safety plan (01-09-353) approved by the department of Environmental Safety and Health at North Carolina State University.

SECTION 8 REFERENCES

Clesceri, L. S., A. E. Greenberg, et al. (1995). Standard Methods for the Examination of Water and Wastewater., 19th ed, APHA, AWWA, WPCF.

Page, A.L., ed. Methods of Soil Analysis, 1982. American Society of Agronomy, Inc.; Soil Science Society of America, Inc., Madison, Wisconsin.

Journal of the Association of Official Analytical Chemists, 1989 Vo 72, p770, Method 990.03, Protein (crude) in Animal Feed, Combustion Method

Environmental Analysis Laboratory
Local Quality Assurance / Quality Control Procedures

The value of laboratory data is directly related to the confidence in which the investigator places the results and the procedures used to obtain those results. In the Biological & Agricultural Environmental Analysis Laboratory (EAL), that confidence is assured through standard good laboratory practices, including an effective quality assurance / quality control program. The heart of that program is the use of spiked and duplicate samples. The procedures for using and interpreting these tools are the subject of this document.

Spiked and duplicate samples, external quality control checks, and standard checks are used throughout the sample runs to ensure the accuracy and precision of the analytical results. All analytical runs begin with at least three working calibration standards to generate a standard curve. After initial calibration, a DI water blank is analyzed, followed by the external quality control check. Spiked and duplicate samples from the ETV project are run every 10 samples. The value of the spike is selected to produce results near 50% of the selected calibration range. A DI water blank and a mid-range continuing calibration standard are run every ten samples and at the end of the analytical run. Any sample result above the highest calibration standard will be diluted and reanalyzed.

Recovery of the spiked samples must fall within 85-115% of true value. Duplicate samples must fall within 25% of each other. If results fall outside of these ranges, the samples are reanalyzed. If the external quality control check and/or continuing calibration standard(s) are out of acceptance criteria, the analytical run is stopped until the cause is determined.