

NC DEQ/DWR WASTEWATER/GROUNDWATER LABORATORY CERTIFICATION BRANCH

LABORATORY NAME:		CERT #:	
PRIMARY ANALYST:		DATE:	
NAME OF PERSON COMPLETING CHECKLIST (PRINT):			
SIGNATURE OF PERSON COMPLETING CHECKLIST:			

Parameter: **Vector Attraction Reduction**
 Method: **Option 1: Reduction in Volatile Solids Content [40 CFR 503.33(b)(1)]**
Control of Pathogens and Vector Attraction in Sewage Sludge, EPA/625/R-92/013

Equipment:

Muffle furnace for operation at 550°C	Mixing Paddle	Desiccator, provided with a desiccant containing a color indicator of moisture concentration or an instrumental indicator
Drying oven, for operation at 103 to 105°C	Evaporating dishes of 100-mL capacity	Analytical balance, capable of weighing to 10 mg

PLEASE COMPLETE CHECKLIST IN INDELIBLE INK
Please mark Y, N or NA in the column labeled LAB to indicate the common lab practice and in the column labeled SOP to indicate whether it is addressed in the SOP.

	GENERAL	L A B	S O P	EXPLANATION
1	Is the SOP reviewed at least every 2 years? What is the most recent review/revision date of the SOP? [15A NCAC 2H .0805 (a) (7)] Date:			Quality assurance, quality control, and Standard Operating Procedure documentation shall indicate the effective date of the document and be reviewed every two years and updated if changes in procedures are made. Verify proper method reference. During review notate deviations from the approved method and SOP.
2	Are all review/revision dates and procedural edits tracked and documented? [15A NCAC 2H .0805 (a) (7)]			Each laboratory shall have a formal process to track and document review dates and any revisions made in all quality assurance, quality control and SOP documents.
3	Has the laboratory developed and implemented a documented training program? [15A NCAC 2H .0805 (a) (7) (P)]			Each laboratory shall develop and implement a documented training program that includes documentation that: (i) that staff have the education, training, experience, or demonstrated skills needed to generate quality control results within method-specified limits and that meet the requirements of these Rules; (ii) that staff have read the laboratory quality assurance manual or applicable Standard Operating Procedures; (iii) that staff have obtained acceptable results on Proficiency Testing samples pursuant to Rule .0803(1) of this Section or other demonstrations of proficiency (e.g., side-by-side comparison with a trained analyst, acceptable results on a single-blind performance evaluation sample, an initial demonstration of capability study prescribed by the reference method).
4	Is there North Carolina data available for review?			
	PROCEDURE – Sample Preparation	L A B	S O P	EXPLANATION
5	Are clean evaporating dishes ignited at 550°C ±50°C for ≥15 min in a muffle furnace? [SM 2540 G-2015 (3) (a) (1)]			
6	Are ignited dishes then cooled in a desiccator to ambient temperature and weighed? [SM 2540 G-2015 (3) (a) (1)]			
7	Are evaporating dishes stored in a desiccator or 103-105°C oven until needed? [SM 2540 G-2015 (3) (a) (1)]			
8	Do samples qualify to be analyzed according to Option 1? [Control of Pathogens and Vector Attraction in Sewage Sludge, EPA/625/R-92/013, (July 2003) (8.2)]			This option is intended for use with biological treatment systems only. Under Option 1, reduction of vector attraction is achieved if the mass of volatile solids in the sewage sludge is reduced by at least 38%. This is the percentage of volatile solids reduction that can generally be attained by the “good practice” recommended conditions for anaerobic digestion of 15 days residence

				time at 35°C [95°F] in a completely mixed high-rate digester. The percent volatile solids reduction can include any additional volatile solids reduction that occurs before the biosolids leave the treatment works, such as might occur when the sewage sludge is processed on drying beds or in lagoons.
9	<p>What is the starting point where the samples are collected? [Control of Pathogens and Vector Attraction in Sewage Sludge, EPA/625/R-92/013, (July 2003) (8.2)] [Control of Pathogens and Vector Attraction in Sewage Sludge, EPA/625/R-92/013, (July 2003) Appendix C]</p> <p>Answer:</p>			<p>The starting point for measuring volatile solids in sewage sludge is at the point at which sewage sludge enters a sewage sludge treatment process. This can be problematic for facilities in which wastewater is treated in systems like oxidation ditches or by extended aeration. Sewage sludges generated in these processes are already substantially reduced in volatile solids content by their long exposure to oxidizing conditions in the process. If sewage sludge removed from these processes is further treated by anaerobic or aerobic digestion to meet VAR requirements, it is unlikely that the 38% reduction required to meet Option 1 can be met. In these cases, use of Options 2, 3, or 4 is more appropriate.</p> <p>For most processing sequences, the processing steps downstream from the digester, such as short-term storage or dewatering, have no influence on volatile solids content. Consequently, the appropriate comparison is between the sewage sludge entering the digester and the sewage sludge leaving the digester.</p>
10	<p>What is the end point where the samples are collected? [Control of Pathogens and Vector Attraction in Sewage Sludge, EPA/625/R-92/013, (July 2003) (8.2)] [Control of Pathogens and Vector Attraction in Sewage Sludge, EPA/625/R-92/013, (July 2003) Appendix C]</p> <p>Answer:</p>			<p>The end point where volatile solids are measured to calculate volatile solids losses can be at any point in the process. Because volatile solids continue to degrade throughout sewage sludge treatment, it is recommended that samples be taken at the end point of treatment.</p> <p>For most processing sequences, the processing steps downstream from the digester, such as short-term storage or dewatering, have no influence on volatile solids content. <i>Consequently, the appropriate comparison is between the sewage sludge entering the digester and the sewage sludge leaving the digester.</i></p> <p>The sampling point for the “after treatment” measurement can be immediately leaving the processing unit or at the point of use or disposal, provided there has been no significant dilution downstream with inert solids.</p>
	PROCEDURE – Sample Analysis	L A B	S O P	EXPLANATION
11	<p>What amount of biosolids were used or disposed in the previous year? [Control of Pathogens and Vector Attraction in Sewage Sludge, EPA/625/R-92/013, (July 2003) (9.5)]</p> <p>Answer:</p>			
12	<p>What is the lab’s sampling frequency? [15A NCAC 02T .1111 (c) and Control of Pathogens and Vector Attraction in Sewage Sludge, EPA/625/R-92/013, (July 2003) (9.5)]</p> <p>Answer:</p>			<p>It is obviously not feasible to sample and analyze every load of biosolids leaving a facility, nor is it necessary. However, a sampling plan does need to adequately account for the variability of the biosolids. This entails collecting samples at an adequate frequency and analyzing a sufficient number of samples. The minimum sampling frequency and number of samples to be analyzed are shown in 40 CFR Part 503. As shown in Table 3-4, the sample collection frequency is determined by the amount of biosolids used or disposed.</p>

			<p>Table 3-4. Frequency of Monitoring for Land Application and Surface Disposal</p> <table border="1"> <thead> <tr> <th>Amount of Biosolids¹ (metric tons dry solids per 365-day period)</th> <th>Minimum Frequency</th> </tr> </thead> <tbody> <tr> <td>Greater than zero but less than 290²</td> <td>Once per year</td> </tr> <tr> <td>Equal to or greater than 290 but less than 1,500²</td> <td>Once per quarter (four times per year)</td> </tr> <tr> <td>Equal to or greater than 1,500 but less than 15,000²</td> <td>Once per 60 days (six times per year)</td> </tr> <tr> <td>Equal to or greater than 15,000²</td> <td>Once per month (12 times per year)</td> </tr> </tbody> </table> <p>¹Either the amount of bulk biosolids applied to the land, or the amount of sewage sludge received by a person who prepares biosolids that are sold or given away in a bag or other container for application to the land (dry weight basis), or the amount of biosolids (excluding domestic septage) placed on a surface disposal site.</p> <p>²290 metric tons = 320 tons (approximately 0.9 tons/day for a year). 1,500 metric tons = 1,653 tons (approximately 4.5 tons/day for a year). 15,000 metric tons = 16,534 tons (approximately 45 tons/day for a year).</p>	Amount of Biosolids ¹ (metric tons dry solids per 365-day period)	Minimum Frequency	Greater than zero but less than 290 ²	Once per year	Equal to or greater than 290 but less than 1,500 ²	Once per quarter (four times per year)	Equal to or greater than 1,500 but less than 15,000 ²	Once per 60 days (six times per year)	Equal to or greater than 15,000 ²	Once per month (12 times per year)
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13	<p>How are samples collected? [Control of Pathogens and Vector Attraction in Sewage Sludge, EPA/625/R-92/013, (July 2003) Appendix C]</p> <p>Answer:</p>		<p>The principal cause of error is poor sampling. Samples should be representative, covering the entire charging and withdrawal periods. Averages should cover extended periods of time during which changes in process conditions are minimal.</p>										
14	<p>How is the sample homogenized? [SM 2540 G-2015 (3) (a) (2) (a) and (b)]</p> <p>Answer:</p>		<p>Fluid samples: If sample contains enough moisture to flow readily, then stir or shake to homogenize</p> <p>Solid samples: If sample consists of discrete pieces of solid material (e.g., dewatered sludge) then take care to obtain a representative sample whose particle size will not impede drying...</p> <p>Manually process samples as quickly as possible to prevent moisture loss. Processing via mechanical grinding is not recommended because moisture levels could drop during processing.</p>										
15	<p>Is 25 to 50 grams of homogenized sample placed in a prepared evaporating dish and weighed? [SM 2540 G-2015 (3) (a) (2) (a) and (b)]</p>												
16	<p>Are fluid samples evaporated to dryness and then dried in an oven at 103 to 105°C for ≥ 1 hour? [SM 2540 G-2015 (3) (a) (2) (a)]</p>		<p>Evaporate to dryness on a water bath, on a hot plate or block, or in a drying oven, then dry the evaporated sample at 103 -105°C for ≥ 1 h.</p>										
17	<p>Are solid samples placed in a 103 to 105°C oven for ≥ 1 hour? [SM 2540 G-2015 (3) (a) (2) (b)]</p>												
18	<p>Are samples cooled to ambient temperature in a desiccator, and weighed? [SM 2540 G-2015 (3) (a) (2) (a) and (b)]</p>		<p>cool to ambient temperature in a desiccator, and weigh.</p>										
19	<p>Is the heating, cooling, desiccating, and weighing procedure repeated until the weight change is <50 mg? [SM 2540 G-2015 (3) (a) (2) (a) and (b)]</p>		<p>Repeat cycle (drying, cooling, desiccating, and weighing) until the weight change is <50 mg.</p>										
20	<p>Is the dried residue transferred to a cool muffle furnace, and then ignited at 550°C ±50°C for ≥ 1 hour? [SM 2540 G-2015 (3) (b)]</p>		<p>Fixed and volatile solids: Transfer the dried residue to a cool muffle furnace, heat furnace to 550°C ±50°C, and the allow ignition to occur for ≥ 1 h.</p>										
21	<p>Are samples cooled to ambient temperature in a desiccator, and weighed? [SM 2540 G-2015 (3) (b)]</p>		<p>Cool in desiccator to ambient temperature and weigh.</p>										
22	<p>Are the igniting, cooling, desiccating and weighing steps repeated until the weight change is <50 mg? [SM 2540 G-2015 (3) (b)]</p>		<p>Repeat cycle (igniting, cooling, desiccating, and weighing) until the weight change is <50 mg.</p>										
23	<p>How are percent volatile solids calculated? [SM 2540 G-2015 (4)]</p> <p>Answer:</p>		<p>This equation is simplified from SM to determine percent volatile solids in the decimal form.</p> $\text{Percent volatile solids (in decimal form)} = \frac{A-C}{A-B}$ <p>where <i>A</i> = weight of dish + dried residue in milligrams <i>B</i> = weight of dish in milligrams <i>C</i> = weight of residue + dish after ignition in milligrams</p>										
24	<p>How is the fractional volatile solids reduction calculated?</p>												

	[Control of Pathogens and Vector Attraction in Sewage Sludge, EPA/625/R-92/013, (July 2003) Appendix C] Answer:			The following equation appears on the NC Operator's Exam formulas page as a version of the Van Kleeck formula. % Volatile Solids Destroyed in a digester: % reduction = $\frac{(\text{in}) - (\text{out})}{(\text{in}) - (\text{in} * \text{out})} * 100$
25	Is the calculated fractional volatile solids reduction result at least 38%? [15A NCAC 02T .1107 (a) (1)]			The mass of the volatile solids in the biological residuals shall be reduced by a minimum of 38 percent between the time that the biological residuals enter the digestion process and the time it is land applied.
	QUALITY ASSURANCE	L A B	S O P	EXPLANATION
26	Are duplicates analyzed at a frequency of ≥5% of a batch of ≤20 samples each day? [SM 2540 A-2015 (5)]			Analyze ≥5% of all samples in duplicate or at least one duplicate sample with each batch of ≤20 samples.
27	What is the acceptance criterion for duplicates? [15A NCAC 2H .0805 (a) (7) (A)] Answer:			SM 2540 A-2015 (5) states: Typically, the relative percent difference (RPD) of duplicates should not exceed 10%, but RPDs may vary considerably due to sample matrix and concentration. This is not required, and the acceptance criterion is to be set by the laboratory.
28	What corrective action does the laboratory take if the duplicate samples results are outside of established control limits? [15A NCAC 2H .0805 (a) (7) (B)] Answer:			If quality control results fall outside established limits or show an analytical problem, the laboratory shall identify the Root Cause of the failure. The problem shall be resolved through corrective action, the corrective action process documented, and any samples involved shall be reanalyzed, if possible.
29	Is the analytical balance being serviced every 12 months by a qualified vendor/technician? [15A NCAC 2H .0805 (a) (7) (J)]			Laboratory analytical balances shall be serviced by a metrology vendor or technician every 12 months to verify that the balance is functioning within manufacturer's specifications.
30	Does the laboratory have documentation to verify that the balance has been serviced? [15A NCAC 2H .0805 (a) (7) and (a) (7) (E)]			Supporting Records shall be maintained as evidence that these practices are implemented. All analytical data and records pertinent to each certified analysis shall be available for inspection upon request.
31	Is the laboratory using ASTM Type 1, Class 1 or 2, or equivalent weights? [15A NCAC 2H .0805 (a) (7) (J)]			The analytical balance shall be checked with one ASTM Type 1, Class 1 or 2, or equivalent standard weight each day used.
32	Are the weights being verified every 5 years? [15A NCAC 2H .0805 (a) (7) (J)] [NC WW/GW LCB Policy]			These weights shall be verified every five years. Verification may be accomplished by: 1. Sending laboratory weights back to the manufacturer for recertification - reference weights shall be calibrated by a body that can provide traceability to ASTM specifications, or 2. Checking laboratory weights against certified reference weights (i.e., weights that have been recertified as above) and found to be within ASTM Type I tolerances (see Weight Verification policy) - often the balance service technician may provide this service.
33	Does the laboratory have documentation indicating that the weights were verified? [15A NCAC 2H .0805 (a) (7)]			Supporting Records shall be maintained as evidence that these practices are implemented.

	Date Verified:		Documentation of weight verifications or recertification must be maintained for 5 years. If the condition of a weight(s) is in question at any time due to damage (e.g., corrosion, nicks, scratching, etc.), the laboratory must have that weight(s) re-verified as described above.
34	Is the balance checked with a weight each day of use? [15A NCAC 2H .0805 (a) (7) (J)] List weight:		The analytical balance shall be checked with one ASTM Type 1, Class 1 or 2, or equivalent standard weight each day used.
35	Is this documented? [15A NCAC 2H .0805 (a) (7) (J)]		The values obtained shall be recorded, dated, and initialed.
36	Is the balance checked with at least three weights monthly? [15A NCAC 2H .0805 (a) (7) (J)] List weights:		The analytical balance shall be verified monthly with three ASTM Type 1, Class 1 or 2, or equivalent standard weights across the range of use.
37	Is this documented? [15A NCAC 2H .0805 (a) (7) (J)]		The values obtained shall be recorded, dated, and initialed.
38	Is the data qualified on the EPA Biosolids Annual Report (NPDES Form 6100-035) or client report if Quality Control (QC) requirements are not met? [15A NCAC 2H .0805 (a) (7) (B)]		If quality control results fall outside established limits or show an analytical problem, the laboratory shall identify the Root Cause of the failure. The problem shall be resolved through corrective action, the corrective action process documented, and any samples involved shall be reanalyzed, if possible. If the sample cannot be reanalyzed, or if the quality control results continue to fall outside established limits or show an analytical problem, the results shall be qualified as such.

Additional Comments:

Inspector: _____ Date: _____