

ATTACHMENT A

Protocol for Nitrogen Sample Collection and Analysis for Waste Water Effluent

Approved Analytical Methods (from Table 1 B of Part 136 per the 2012 Method Update Rule): (laboratory must be certified for any method performed)

Total Kjeldahl Nitrogen (TKN):

Manual digestion and distillation or gas diffusion followed by any of the following	SM4500-Norg B-97 or C-97 and SM4500-NH3 B-97.	ASTM D3590-02 (06) (A)	I-4515-9145
Titration	SM4500-NH3 C-97	ASTM D3590-89, 02 (A)	973.48.3
Nesslerization		ASTM D1426-08 (A)	
Electrode	SM4500-NH3 D-97 or E-97	ASTM D1426-08 (B)	
Semi-automated phenate	EPA 350.1 Rev. 2.0 (1993)	SM4500-NH3 G-97 or H-97	
Manual phenate, salicylate, or other substituted phenols in Berthelot reaction based methods	SM4500-NH3 F-1997		
<i>Automated methods for TKN that do not require manual digestion</i>			
Automated phenate, salicylate, or other substituted phenols in Berthelot reaction based methods colorimetric (auto digestion and distillation)	EPA 351.1 (1978)		I-4551-788
Semi-automated block digester colorimetric (distillation not required)	EPA 351.2, Rev. 2.0 (1993)	SM4500-Norg D-97	ASTM D3590-02 (06) (B) I-4515-9145

Nitrate + Nitrite (NO₃ + NO₂):

Cadmium reduction, Manual		SM4500-NO3 E-00	ASTM D3867-04 (B)	
Cadmium reduction, Automated, or	EPA 353.2, Rev. 2.0 (1993)	SM4500-NO3 F-00	ASTM D3867-04(A)	I-4545-852
Automated hydrazine		SM4500-NO3 H-00		
Ion chromatography	EPA 300.0, Rev. 2.1 (1993) and EPA 300.1, rev. 1.0 (1997)	SM4110 B-00 or C-00	ASTM D4327-03	993.303
CIE/UV		SM4140 B-97	ASTM D6508-00 (05)	ASTM D6508, Rev. 2

Sample Collection: The Maine DEP is requesting that nitrogen analysis be conducted on composite effluent samples, unless a facility's Permit specifically designates grab sampling for this parameter. Facilities can use individual collection bottles or a single jug made out of glass or polyethylene. Bottles and/or jugs should be cleaned prior to each use with dilute H₂SO₄. This cleaning should be followed by several rinses with distilled water. Commercially purchased, pre-cleaned sample containers are an acceptable alternative. The sampler hoses should be cleaned; as needed.

Sample Preservation: During compositing the sample must be at 0-6 degrees C (without freezing). If the sample is being sent to a commercial laboratory or analysis cannot be performed the day of collection then the sample must be preserved using H₂SO₄ to obtain a sample pH of <2 su and refrigerated at 0-6 degrees C (without freezing). The holding time for a preserved sample is 28 days.

Laboratory QA/QC: Laboratories must follow the appropriate QA/QC procedures that are described in each of the approved methods.

Sampling QA/QC: If a composite sample is being collected using an automated sampler, then once per month run a blank on the composite sampler. Automatically, draw distilled water into the sample jug using the sample collection line. Let this water set in the jug for 24 hours and then analyze for total nitrogen. Preserve this sample as described above.