

## Sludge Methods Interlaboratory Validation Study

EPA's Office of Water (OW) is reinitiating efforts to validate methods for measurement of parameters regulated at 40 CFR part 503. To this end, OW is planning to conduct an interlaboratory validation study of several draft method procedures for analysis of biosolids. This study will involve at least two representative biosolids matrices.

EPA intends to involve at least eight volunteer participant laboratories for validation of each method. Participant laboratories may choose to participate in the validation of any or all of the methods described below. A referee laboratory will collect, analyze, and prepare biosolids samples of known composition and distribute samples to participant laboratories for analysis using the study methods. In addition to sample analyses, participant laboratories will be required to prepare and analyze all quality control samples and follow all procedures included in the methods. Analytical results generated by the participant labs will be used to develop method performance specifications for each method, and draft methods will be finalized based on the results of the study. Depending on interest in study participation, the study may be streamlined by 1) developing one set of method performance criteria for either Method 1685 or Method 1686 for nitrite/nitrate nitrogen; 2) developing one set of method performance criteria for either Method 1687 or Method 1688 for TKN; and 3) testing only one method for ammonia nitrogen.

### Analytical Methods Targeted for Interlaboratory Validation

DRAFT BIOSOLIDS METHOD	SOURCE METHOD	SUMMARY OF PROCEDURE
<b>Method 1683:</b> Specific Oxygen Uptake Rate (SOUR)	Standard Method 2710 B (18 <sup>th</sup> Edition)	Oxygen consumption rate is measured in an aerated sample using an oxygen-sensing probe, manometer, or respirometer. Dissolved oxygen (DO) is recorded over a 15-minute period or until the DO becomes rate-limiting. The SOUR is obtained by dividing the oxygen consumption rate by total solids.
<b>Method 1684:</b> Total, Fixed, and Volatile Solids  (Gravimetric)	Standard Method 2540 G (18 <sup>th</sup> Edition)	Total, fixed, and volatile solids are determined by oven drying at 103-105°C, then at 550°C, and weighing the cooled samples after each drying process.
<b>Method 1685:</b> Nitrate/Nitrite Nitrogen  (Automated Colorimetry)	EPA Method 353.2	Oxidized nitrogen is extracted in reagent water, filtered, and passed through an ammonium chloride-EDTA activated cadmium-copper reduction column to convert nitrate to nitrite. Any fat, oil, and grease is removed by adjusting the sample to pH 2 with HCl, followed by hexane extraction. The sample is then adjusted to a pH of 5 with HCl or NH <sub>4</sub> OH. Nitrite is measured using an automated colorimeter (photometer).
<b>Method 1686:</b> Nitrate/Nitrite Nitrogen  (Manual)	EPA Method 353.1	Oxidized nitrogen is extracted in reagent water, filtered, and passed through an ammonium chloride-EDTA activated cadmium-copper reduction column to convert nitrate to nitrite. Any fat, oil, and grease is removed by adjusting the sample to pH 2 with HCl, followed by hexane extraction. The sample is then adjusted to a pH of 5 with HCl or NH <sub>4</sub> OH. A "color reagent" is added to the sample, and absorbance is measured at 540nm.

DRAFT BIOSOLIDS METHOD	SOURCE METHOD	SUMMARY OF PROCEDURE
<p><b>Method 1687:</b> Total Kjeldahl Nitrogen</p> <p>(Flask Digester, Distillation, Automated colorimetry)</p>	<p>EPA Method 351.3</p>	<p>A 5-g (or 300-mL) sample is added to a Kjeldahl flask, digested with K<sub>2</sub>SO<sub>4</sub>, CuSO<sub>4</sub>, H<sub>2</sub>SO<sub>4</sub>, and distilled into H<sub>2</sub>SO<sub>4</sub> after the addition of NaOH and sodium thiosulfate. TKN is measured by automated colorimetry with acidified water, EDTA, sodium phenolate NaOCl and nitroprusside. (Cupric sulfate has been substituted for the historically-used mercuric sulfate due to toxicity and waste disposal problems associated with mercury.)</p>
<p><b>Method 1688:</b> Total Kjeldahl Nitrogen</p> <p>(Block Digester, Automated colorimetry)</p>	<p>EPA Method 351.2</p>	<p>A 5-g (or 20-mL) sample is added to a digester tube and digested in a block digester with K<sub>2</sub>SO<sub>4</sub>, CuSO<sub>4</sub>, H<sub>2</sub>SO<sub>4</sub>. TKN is measured by automated colorimetry with acidified water, EDTA, sodium phenolate NaOCl and nitroprusside solutions. (Cupric sulfate has been substituted for the historically-used mercuric sulfate due to toxicity and waste disposal problems associated with mercury.)</p>
<p><b>Method 1689:</b> Ammonia Nitrogen</p> <p>(Ion selective electrode)</p>	<p>EPA Method 350.2 followed by 350.3</p>	<p>Distillation of the ammonia from the sample is followed by analysis using an ion-selective probe. Results are compared to a calibration curve.</p>
<p><b>Method 1690:</b> Ammonia Nitrogen</p> <p>(Automated colorimetry)</p>	<p>EPA Method 350.2 followed by 350.1</p>	<p>Distillation of the ammonia from the sample is followed by the colorimetric measurement of indophenol blue using an automated colorimetric analyzer.</p>
<p><b>Method 245.x:</b> Total Mercury</p> <p>(Cold Vapor Atomic Absorption Spectroscopy (CVAAS))</p>	<p>EPA Methods 245.1 Revision 3.0 (1994) and 245.5 Revision 2.3 (1991)</p>	<p>Samples with ≤1% total solids are digested with potassium permanganate-potassium persulfate and oxidized for 2 h at 95°C. Samples with &gt;1% total solids are digested using aqua regia at 95°C, followed by dilution with reagent water and oxidation with potassium permanganate for 30 minutes at 95°C. Mercury is reduced to elemental mercury with stannous chloride and measured using cold vapor atomic absorption .</p>

## Outreach Sheet - Interest in EPA's Biosolids Methods Validation Study

(NOTE: Laboratories may chose to participate in validation of one or more of the methods listed below)

METHOD	SOURCE METHOD	Are you currently using this procedure for analysis of biosolids ?		Are you interested in voluntary participation in a validation study for this method?	
		Yes	No	Yes	No
1683: Specific Oxygen Uptake Rate (SOUR)	Standard Method 2710 B				
1684: Total, Fixed, and Volatile Solids	Standard Method 2540 G				
1685: Nitrate/Nitrite Nitrogen (Automated)	EPA Method 353.2				
1686: Nitrate/Nitrite Nitrogen (Manual)	EPA Method 353.1				
1687: Total Kjeldahl Nitrogen (Flask Digester, Distillation, Colorimeter)	EPA Method 351.3				
1688: Total Kjeldahl Nitrogen (Block Digester, Colorimeter)	EPA Method 351.2				
1689: Ammonia Nitrogen (Ion selective electrode)	EPA Method 350.2 followed by 350.3				
1690: Ammonia Nitrogen (Automated analysis)	EPA Method 350.2 followed by 350.1				
245.x: Total Mercury (Cold Vapor Atomic Absorption)	EPA Methods 245.1 Rev. 3.0 (1994) and 245.5 Rev. 2.3 (1991)				

**Laboratory Name:** \_\_\_\_\_

**Laboratory Address:** \_\_\_\_\_

**Laboratory Contact (include email address):** \_\_\_\_\_

**Phone Number:** \_\_\_\_\_ **Fax Number:** \_\_\_\_\_

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