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An Easier, Faster, and Cost-Effective Option for Measuring Total Nitrogen

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The Total Nitrogen Analysis test kit was developed to provide users with a single test method for determining total nitrogen levels in process water and wastewater. Traditionally, users have had to perform total Kjeldahl nitrogen (TKN), nitrate and nitrite analysis, then sum them to obtain a value for Total Nitrogen. The new total nitrogen test kit is faster, safer, more accurate and costs less than the traditional method.

Total nitrogen (TN) concentration has become a concern because of its impact on eutrophication of water sources. And as more US states begin to set limits for TN, accurate testing becomes paramount. Unfortunately, multiple labs and variable test procedures can lead to disparities in final results. Many of today's test methods are also time consuming, expensive, and even unsafe for lab technicians to use.

"Current methods introduce variability and human error into the mix, so there is no question that existing methods are suboptimal," says Taylor Reynolds, program marketing manager for Environmental and Ag Bio Testing.

Traditional Total Nitrogen Test Methods are Susceptible to Variability

Probably the most common method to test for TN is summation determination, which measures Total Kjeldahl Nitrogen (TKN), nitrite, and nitrate, requiring three separate tests to be run. Total nitrogen is not defined as a single analyte in 40 CFR Part 136 from U.S. EPA. As a result, individual U.S. states are left to their own devices as to how they define the methods and test for TN. Most states have fallen back on defining it as TKN + nitrite + nitrate (summation determination). While this is a robust methodology, and labs already have the equipment to conduct the test, there are a few obvious drawbacks:

- **Lengthy.** The process takes about 2.5 hrs at a minimum to complete.
- **Hazardous.** The test has to be performed under a fume hood, as there is risk of splatter and acid exposure.
- **Generates waste.** This waste comes from the acids and liquids.
- **Accuracy errors.** Each individual test has its own +/- accuracy. When the test results are added together, the accuracy of the summation is subject to the sum of all the error margins of those tests.
- **May require additional steps.** If there are elevated levels of nitrate in the effluent, it will suppress the Total Kjeldahl Nitrogen values. Additional steps need to then be taken to reduce that nitrate into ammonia.

Additionally, there is some wiggle room related to summation determination because there are many different methods to determine nitrite and nitrate. In the state of Iowa, for example, it was discovered that some operators were farming out samples to multiple labs that used different methods. Plant operators then cherry-picked the data, chose the lowest numbers, and added them together to get the TN number that would ensure compliance. "This is not ideal for monitoring the health of a waterbody and is quite a slippery slope to go down as it opens plants up to variability and error," says Reynolds. "Even in a less ambiguous setting, I have yet to talk to a lab tech that is comfortable with the TKN test."

A newer test is the chemiluminescence method. The test requires a small portion of sample to be injected into a plain ionization unit. The ionizer runs at 720°C. The color of the flame, which changes as sample is injected, correlates to TN levels. Some drawbacks to this method include:

- **Not regulatorily sound.** The method is less than ideal for regulatory compliance because at 720°C, all of the nitrogen in the sample is converted—including the mineral species of nitrogen not considered bioavailable (sand, grit, sediment)—which has no impact on the health of the waterway. And, because the mineral species are recorded, artificially high values of TN are presented. As a result, the method can really only be used for internal reference.
- **Requires specialized equipment.** Chemiluminescence requires sophisticated equipment that not every lab has, such as a platinum catalyst, which is expensive to replace and complex to operate. The bottom line is that a lab is committing to buying an instrument that will only run one test.

"With these tests, there's a lot of potential for things to go wrong, particularly in the areas of reliability and result repeatability, but there hasn't been an alternative," says Reynolds.

A Standardized Total Nitrogen Test Offers Confidence in Results

Labs across the state of Iowa are, in fact, testing an alternative total nitrogen methodology that offers promise in the areas of reliability, repeatability, and ease of use. The test is based on a chemical reaction known as Koroleff digestion. The operator simply puts a sample into a digestion tube and adds sodium hydroxide and potassium persulfate, which oxidizes and converts all of the nitrogen derivatives into nitrate. After the digestion process is complete, a smaller subsection of the sample is transferred into another vial and color-forming reagents are added, creating a light or dark pink color, depending on concentrations. A spectrophotometer is used to run an absorption analysis to determine the TN level in the sample.

Spectrophotometric Total Nitrogen Test Kit

This new test offers the possibility of having one standardized test for TN. In addition, the test offers several undeniable benefits:

- **Simple to use.** A single test to get a single TN result, and the steps to are simplified compared to other options.
- **Safety**. The test does not require a fume hood as everything is done in enclosed vessels.
- **No special equipment required**. The test can be run on a typical spectrophotometer found in every lab.
- **Speed**. A TN test is completed in about an hour. A "Commercial lab's goal is to run as many tests as possible in a day to make the most money," says Reynolds. "This new test will increase their throughput."
- **Eliminates uncertainty**. Rather than running three different tests for TKN, nitrite, and nitrate, and adding those values, just one test offers the highest degree of confidence in the results.
- **Low costs.** Because existing lab equipment is utilized, a TN test will cost around $3.75 to run, compared to $15 to run a complete summation determination test.

The test has been used in Europe and around the world for decades and is just now being introduced to the North American market. The last method update by the EPA was in 2017, but industry insiders are hopeful that EPA will promulgate this new method for TN as a means of unifying the country's testing.

"Aggregate methods common in the industry introduce variability and opportunities for error," says Reynolds. "If an operator has variability in test results, be sure that is not because of a poor test. This new test can eliminate that variability."

SIGMA-ALDRICH®

Product #	Description
1.15348	Kjeldahl tablets (without addition of Se and Hg) 5 g/tablet
1.16469	Kjeldahl tablets (without addition of Se and Hg / Missouri catalyst) 5 g/tablet
1.18348	Kjeldahl tablets (without addition of Se and Hg) 3.5 g/tablet
1.18469	Kjeldahl tablets (Missouri catalyst) 3.5g/tablet
1.14763	Nitrogen (total) Cell Test Method: photometric, DMP 10 - 150 mg/l N Spectroquant®
1.14537	Nitrogen (total) Cell Test Method: photometric 0.5 - 15.0 mg/l N Spectroquant®
1.00613	Nitrogen (total) Cell Test Method: photometric, DMP 0.5 - 15.0 mg/l N Spectroquant®
173016	Prove 100 VIS Spectrophotometer 4 nm spectral bandwidth Spectroquant®
173017	Prove 300 UV/VIS spectrophotometer 4 nm spectral bandwidth Spectroquant®
173018	Prove 600 UV/VIS spectrophotometer 1,8 nm spectral bandwidth Spectroquant®