Application Note AN-T-214

Kjeldahl nitrogen in waste water

Easy determination by titration according to ASTM D3590

Nitrogen-based compounds are widely distributed in the environment. They are essential growth nutrients for photosynthetic organisms (e.g., plants and algae). Therefore, it is important to monitor and control the amount of nitrogen compounds which are released into the environment.

In this Application Note, a method to determine the nitrogen content in water by Kjeldahl digestion and distillation followed by a photometric or potentiometric titration according to ASTM D3590 is presented. Nitrogen determination by Kjeldahl digestion and distillation has been performed since 1883. The universality, precision, and reproducibility of the Kjeldahl method have made it the internationally recognized method for e.g. estimating the protein content in many matrices and it is the standard method to which all other methods are judged against.

After the Kjeldahl distillation, the nitrogen content is determined by acid-base titration. This can either be a photometric or potentiometric titration depending on the sample and the preferences of the user. Both titration methods offer a reliable and inexpensive determination.

SAMPLE AND SAMPLE PREPARATION

This application is demonstrated on wastewater for the photometric titration, and on spiked water for the potentiometric titration.
Each sample is transferred into the Kjeldahl tube for digestion. The digestion is performed automatically using a commercially available digester. After the digestion, a sodium hydroxide solution is added to the mixture and the resulting ammonia is automatically distilled into a collecting vessel containing boric acid using a steam distillation apparatus.

EXPERIMENTAL

This analysis is carried out on an OMNIS Advanced Titrator equipped with an Optrode for the photometric titration, and with a dEcotrode plus for the potentiometric titration.
The prepared samples are titrated with sulfuric acid until after the equivalence point is reached. To ensure good recovery and reproducibility, it is important that the distillation apparatus used for this sample preparation is leak-proof and that the water, which is used for the blank, is nitrogen-free.

Figure 1. OMNIS Advanced Titrator equipped with a dEcotrode plus for the potentiometric determination of Kjeldahl nitrogen in water.

RESULTS

The recovery and standard deviation of the two different titrations could not be compared, as different samples were used. However, for both methods relative standard deviations are below 2%, which is acceptable for this application.

Table 1. Results of the nitrogen determinations in water by titration after the Kjeldahl digestion and distillation.

	Photometric titration (n = 4)	Potentiometric titration (n = 3)
Mean	33.63 mg/L	19.78 mg/L
SD(abs)	0.45 mg/L	0.26 mg/L
SD(rel)	1.33%	1.34%

CONCLUSION

Titration is an easy method to determine Kjeldahl nitrogen in wastewater according to ASTM D3590. The titration can be either performed photometrically or potentiometrically. The potentiometric method provides the advantage that no indicator is needed. On the other hand, the Optrode for photometric measurements is maintenance-free. Which titration is used depends on the sample and the preferences of the user.

For both methods, an OMNIS Titrator can be used. This allows you to customize the system according to your needs and expand it for other titration applications required for the quality control of water.

Internal reference: AW TI CH1-1301-032020

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CONFIGURATION

OMNIS Advanced Titrator with magnetic stirrer

Innovative, modular potentiometric OMNIS Titrator for stand-alone operation or as the core of an OMNIS titration system for endpoint titration and equivalence point titration (monotonic/dynamic). Thanks to 3S Liquid Adapter technology, handling chemicals is more secure than ever before. The titrator can be freely configured with measuring modules and cylinder units and can have a rod stirrer added as needed. If required, the OMNIS Advanced Titrator can be equipped for parallel titration via a corresponding software function license.

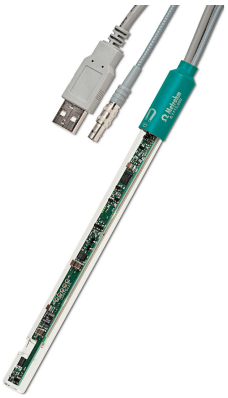
- Control via PC or local network
- Connection option for up to four additional titration or dosing modules for additional applications or auxiliary solutions
- Connection option for one rod stirrer
- Various cylinder sizes available: 5, 10, 20 or 50 mL
- Liquid Adapter with 3S technology: Secure handling of chemicals, automatic transfer of the original reagent data from the manufacturer

Measuring modes and software options:

- Endpoint titration: "Basic" function license
- Endpoint and equivalence point titration (monotonic/dynamic): "Advanced" function license
- Endpoint and equivalence point titration (monotonic/dynamic) with parallel titration: "Professional" function license

Ecotrode Plus

Combined pH electrode for aqueous acid/base titration. The fixed ground-joint diaphragm is insensitive to contamination. Reference electrolyte: c(KCl) = 3 mol/L, storage in storage solution.



Optrode

Optical sensor for photometric titrations offering 8 different wavelengths. The wavelength can be switched using the software (tiamo 2.5 or higher) or with a magnet. The glass shaft is completely solvent-resistant and easy to clean. For example, this space-saving sensor is suitable for:

- Non-aqueous titrations in accordance with USP or EP
- Determinations of carboxyl end groups
- TAN/TBN in accordance with ASTM D974
- Sulfate determination
- Fe, Al, Ca in cement
- Water hardness
- Chondroitin sulfate in accordance with USP

The sensor is not suitable for determinations of concentrations via measurement of color intensity (colorimetry).