Increasing TKN Sample Determinations Through Automation

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Introduction

MBAS Lab had traditionally analyzed TKN by SM-4500 NH3 BCE Kjeldahl Nitrogen. Our existing digestion block only held 20 places, limiting analysis to 16 unknown samples in an 8 hour day. This procedure required a 4-hour digestion time and 4 hours to determine concentration by titration. Automating this analysis and purchasing a new digestion block has allowed MBAS to increase sample output from 16 samples per day to 24 samples per day. Automating TKN has also cut analysis time by 3 hours, which now allows the analysts to focus on other tasks.

Solution

In order to increase sample output and decrease the amount of time spent on the analysis, MBAS switched to EPA Method 351.2, Revision 2.0: Determination of Total Kjeldahl Nitrogen by Semi-Automated Colorimetry. We purchased the Gerhardt Kjeldatherm for digestion, which holds 40 tubes. This allowed analysts to increase sample output from 16 to 24 samples per run. The digestion block is coupled with the OI Analytical FS3700, which automates the analysis after digestion, freeing analyst time. This has improved laboratory productivity by allowing more analyses to be accomplished with less labor.

Summary of Method 351.2

- 25mL samples are digested in the presence of sulfuric acid, potassium sulfate, cupric sulfate on the Gerhardt Kjeldatherm block. Step 1 of the digestion heats the samples at 160˚C for 90 minutes. Step 2 ramps the temperature up gradually for 60 minutes to a final temperature of 380˚C, where the samples then digest for 30 minutes. After the digestion cycle is complete, all samples are automatically raised off the digestion block. Once cool, the samples are diluted to 50mL.
- All digested samples along with calibration and QC standards are vortexed and transferred into 10mL test tubes. Once all samples are in place, the automated analyzer is started, which mixes each sample with a complexing reagent, raising the pH to >11. Ammonia is generated and passes through a gas diffusion membrane, which is then absorbed into a hypochlorite solution to form chloramine.
- The chloramine reacts with salicylate to form indophenol blue in an amount proportional to the ammonia concentration. Addition of sodium nitroferricyanide intensifies the blue color prior to the sample passing through the photometric detector for measurement at a wavelength of 660nm.

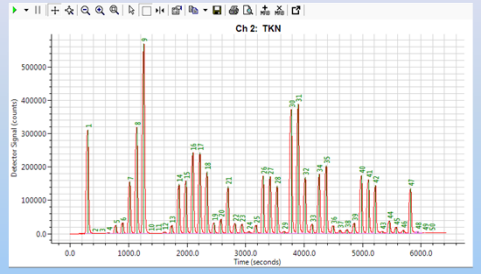
Equipment

Gerhardt Kjeldatherm
40 Place Block Digestor

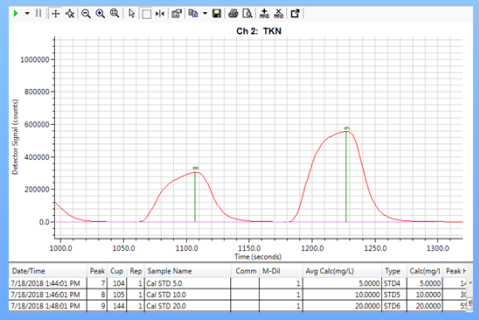
Gas Diffusion Module for TKN

OI Analytical FS3700 Segmented Flow Analyzer, Set Up for TKN by EPA 351.2

Decreasing Run Time



The graph above shows a Kjeldahl run by method EPA 351.2 consisting of calibration standards, quality control samples, matrix spikes, and 24 unknown samples with a total time of approximately 103 minutes.



Date/Time	Peak	Cup	Rep	Sample Name	Comm	M-Dil	Avg Calc(mg/L)	Type	Calc(mg/l	Peak h
7/18/2018 1:44:01 PM	7	104	1	Cal STD 5.0		1	5.0000	STD4	5.0000	1
7/18/2018 1:46:01 PM	8	105	1	Cal STD 10.0		1	10.0000	STD5	10.0000	3
7/18/2018 1:48:01 PM	9	144	1	Cal STD 20.0		1	20.0000	STD6	20.0000	5

TKN determination using peak height takes 2 minutes per sample vs. approximately 6-8 minutes for distillation and titration using method SM-4500 NH3, Kjeldahl Nitrogen.

Conclusion

Two TKN runs per week by SM-4500 NH3 Kjehldahl Nitrogen required 16 hours for 32 unknown samples to be analyzed. Switching methods to EPA 351.2 and using a 40-place Gerhardt Kjeldatherm and OI Analytical FS3700 analyzer reduced overall time for 2 TKN runs per week to 10 hours, while increasing sample output from 32 to 48 samples per week. The pairing of a 40-place digestor and automated analyzer has improved lab efficiency by increasing sample throughput for TKN while reducing labor requirements, freeing up the analyst for other tasks.

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