Application Note
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Nitrate Analysis of Water Using a Fiber Optics Dip Probe

Enables quick measurements in the lab or out in the field

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Introduction

Environmental laboratories analyze thousands of water samples a year to determine the concentration levels of heavy metals and other ions, such as nitrates, phosphates and fluorides. To increase sample throughput and efficiency, optical fibers may be used to measure the absorbance of the sample. This allows for analysis on- or off-site, which is more appealing than a conventional cuvette. This paper presents and discusses results obtained from measuring the nitrate content in water using the quartz dip probe on the Cary 50 UV-Vis spectrophotometer. This experiment can also be done on the Cary 60 UV-Vis, which has superseded the Cary 50.

Experimental

Equipment

- Cary 50* UV-Vis spectrophotometer
- Dip probe fiber optics coupler
- Quartz fiber optic dip probe
- Cary WinUV software

Reagents

- Potassium nitrate (A.R.)
- 37% m/v Hydrochloric acid (A.R.)
- Chloroform (A.R.)
- Water - distilled and de-ionized

Method

The experimental procedure was taken from Standard Methods for the Examination of Water and Wastewater (*1*) and is also described in an Agilent publication (*2*). In brief, standard solutions were prepared in the concentration range of 0 - 7 mg NO_3 N/L and the absorbance measured at 220 and 275 nm. The measurement at two wavelengths allows correction for the interference due to dissolved organic matter, by calculating the difference between both absorbance readings (Equation 1).

Abs(220 nm) - 2xAbs(275 nm) **Equation 1**

The application used was the Cary WinUV Concentration software which evaluates the result of Abs(220)-2xAbs(275) dynamically as a function of concentration. The following instrument settings were used for data collection.

Instrument Settings

Parameter	Setting
User Result	= Read(220)-2*Read(275)
Ordinate Mode	Abs
Ave Time (sec)	1.0000
Replicates	3
Standard/Sample averaging	OFF
Weight and volume corrections	OFF
Fit type	Quadratic
Min R^2	0.95000
Concentration units	mg/L

Results and discussion

Figure 1 shows the calibration curve obtained using the quartz fiber optics dip probe. The Y axis, Abs, is the resultant from Equation 1 and the X-axis is the concentration of nitrate standards in mg/L.

A quadratic function, Equation 2, was fitted to 6 standards giving a correlation coefficient of 0.99931. The raw absorbance data and statistics for the calibration standards are shown in Table 1.

$Abs = -0.00017conc^2 + 0.23364conc + 0.01705$ **Equation 2**

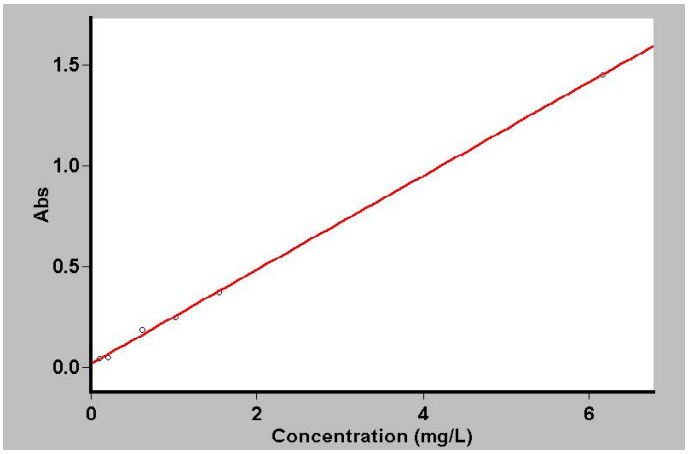

Figure 1. Calibration curve with quadratic fit.

Table 1. Nitrate standards data for calibration curve Std Conc mg/L Mean Abs SD %RSD Raw Abs.

Std	Conc (mg/L)	Mean Abs	SD	%RSD	Raw Abs
Std 1	0.103	0.0443	0.0036	8.03	0.0472 0.0404 0.0454
Std 2	0.205	0.0488	0.0002	0.37	0.0488 0.0490 0.0487
Std 3	0.616	0.1856	0.0011	0.57	0.1856 0.1846 0.1867
Std 4	1.027	0.2467	0.0030	1.22	0.2475 0.2492 0.2434
Std 5	1.541	0.3748	0.0006	0.17	0.3741 0.3750 0.3753
Std 6	6.162	1.4506	0.0011	0.07	1.4503 1.4496 1.4517

**The Cary 50 UV-Vis has been superseded by the Cary 60 UV-Vis.*

Two samples of tap water from different sources, A and B, were prepared as described in reference 1. The absorbance was measured and the concentration of nitrate determined from the calibration curve. The results are shown in Table 2.

Table 2. Raw data and statistics of Water samples Std Conc mg/L Mean Abs SD %RSD Raw Abs.

Std	Conc (mg/L)	Mean Abs	SD	%RSD	Raw Abs
A	0.145	0.0510	0.0009	1.78	0.0520 0.0504 0.0506
B	0.709	0.1825	0.0025	1.36	0.1797 0.1838 0.1841

The three replicates for each standard and sample, shown in Tables 1 and 2, are reproducible within instrumental uncertainty, demonstrating the high precision possible using fiber optics on the Cary 50 UV-Vis. There was negligible solution carry over between samples after washing with only distilled water for approximately 5 seconds.

The time taken to measure 24 solutions of 6 standards and two samples, each with 3 replicates, was approximately 5 minutes. This time included washing the probe with de-ionized water in between readings and drying with a tissue. Measurements with the dip probe are significantly faster and easier than using a conventional cuvette.

Conclusion

The quartz fiber optic dip probe on the Cary 50 or Cary 60 UV-Vis is highly precise and efficient for measuring the nitrate content in water. The time taken to measure 24 solutions is faster than using a cuvette, making the technique an attractive alternative for routine analytical measurements.

Reference

1. D. Eaton, L. S. Clesceri and A. E.Greenberg, Standard Methods for the Examination of Water and Wastewater, 19th Edition, American Public Health Association, Washington, **1995,** p4-85.
2. P. A. Liberatore, Automated nitrate analysis of water, Agilent publication UV59

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