Nitrate analysis of water using the quartz fiber optics dip probe on the Cary 50/60 UV-Vis

Application Note

Environmental

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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.

Experimental

Equipment

- Cary 50 (Cary 60)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

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Method

The experimental procedure was taken from *Standard Methods for the Examination of Water and Wastewater*[1] and is also described in *UV Instruments At Work No. 59* [2]. In brief, standard solutions were prepared in the concentration range of 0 - 7mg 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) - 2xAbs(275) **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

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

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 **Eq. 2**

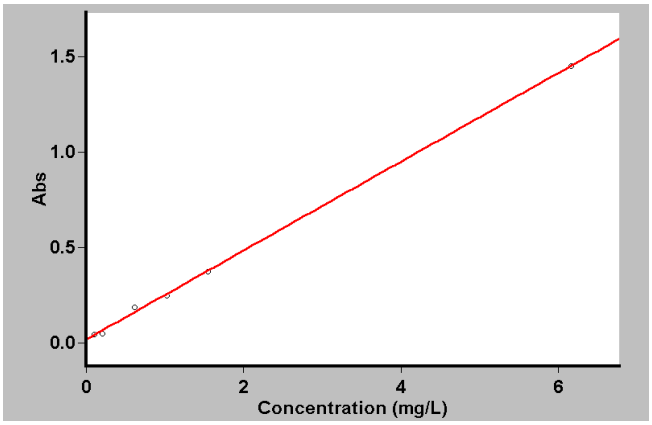

Figure 1. Calibration curve with quadratic fit

Table 1. Nitrate standards data for calibration curve

Std	Conc mg/L	Mean Abs	SD	%RSD	Raw Abs
Std 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

Two samples of tap water from different sources, A and B, were prepared as described in the 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
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

Discussion

The 3 replicates for each standard and sample, shown in Tables 1 and 2, are reproducible within instrumental uncertainty, which demonstrates the high precision possible using fiber optics on the Cary 50/60. There is negligible solution carry over between samples, washing with only distilled water for approximately 5 seconds.

The time taken to measure 24 solutions of 6 standards and 2 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 50/60 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, which makes 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, *UV-Instrument At Work; Automated nitrate analysis of water*, No. 59, Agilent Australia Pty. Ltd, Australia, 1993.

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