application note

Fully Automated Water Nitrate Analysis

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Introduction

Water quality laboratories or contract analytical laboratories may have to analyse thousands of water samples per year. These analyses are usually time consuming and may require the samples to be chemically pre- treated and then each sample manually presented to an instrument to be analysed.

Any subsequent corrections and/or calculations would then have to be manually done. This application note describes how the analysis of nitrate in water samples can be completely automated.

For this study, the American Public Health Association (APHA) Nitrate method[1] was adopted. This method measures the NO_3 content in water samples and is recommended for screening samples which have low organic matter content. The absorbance of the sample is measured at two wavelengths and a calculation is performed. The absorbance is measured at 220 nm and a correction for possible interferences due to dissolved organic matter is performed at 275 nm. The difference between the two absorbance measurements is then calculated by the formula,

$Abs_{220} - (2 * Abs_{275})$.

This analysis can be fully automated with up to 10 standards and 60 samples allowing the chemist to perform other tasks in the laboratory simultaneously.

Experimental

Equipment

- Cintra 20 double beam UV-Visible spectrometer
- Auto sipper with quartz flow through cell
- FS 3000 Autosampler
- Quantify Software Application

Reagents

- Potassium Nitrate (Analytical Reagent)
- 38% Hydrochloric Acid (Analytical Reagent)
- Distilled and deionised water

Preparation of Stock Nitrate solution (1,000 ppm NO_3)

- Oven dry KNO_3 at 105 °C for 24 hours and keep in a desiccator.
- 1.6307 g of the salt was dissolved in approximately 600 mL of water in a 1,000 mL standard flask.
- The flask was made to volume with water.
- This solution is to be prepared fresh monthly.

Preparation of Standard Nitrate Solution (100 ppm NO_3)

- 20.00 mL of the stock solution was diluted to 200 mL with water.
- This solution is to be prepared fresh weekly.

Calibration Standards

The calibration standards were prepared as per the table below. These are to be prepared fresh daily.

100 ppm NO_3 (mL)	No of drops of 38% HCl	Final Volume (mL)	Concentration NO_3 (ppm)
1	10	100	1.0
1	5	50	2.0
2	5	50	4.0
4	5	50	8.0
8	5	50	16.0

Sample preparation

2 drops of 38% HCl were added to each 20 mL test tube. The test tubes were filled with the water samples to be analysed.

If the samples contain fine particulate matter or are cloudy then they may require to be filtered. A Whatman No 542 filter paper is adequate for this task.

Instrument parameters

Figure 1 shows the Main Application Control Page of the Quantify Application.

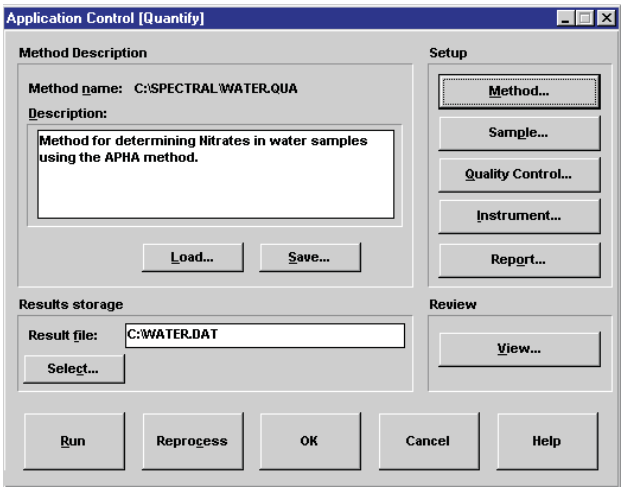

Figure 1 *The Main Application Control Page of Quantify Application*

Operating Parameters

Method

The method parameters used for this analysis are shown in figure 2.

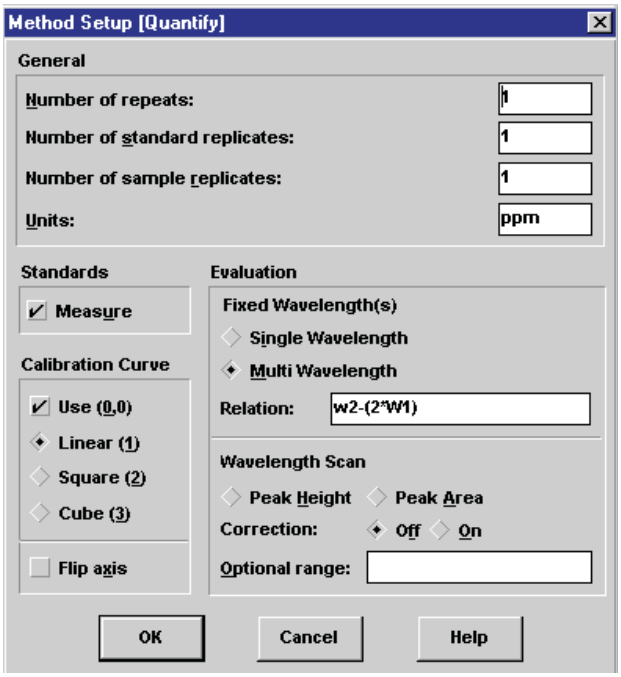

Figure 2 The Method Setup Page

Sample

The standards with their corresponding concentrations and samples were entered as per figure 3.

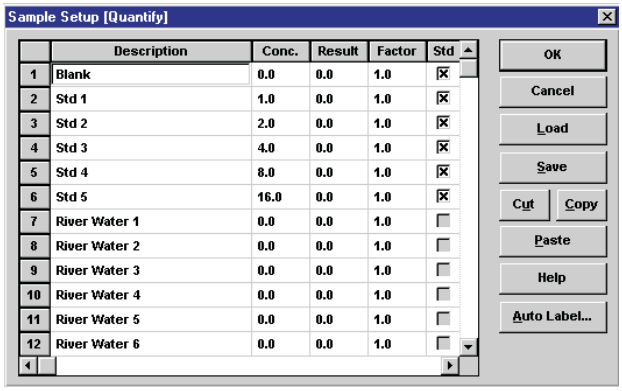

Figure 3 The Sample Setup in the Quantify Application

Quality Control

Quality control protocol can be set up if quality control check samples are to be analysed with the water samples.

Instrument

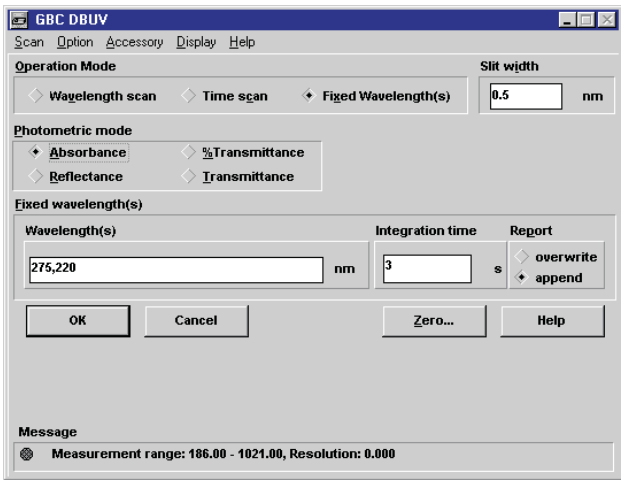

Figure 4 *The Instrument Page*

Accessories

In the Instrument page (as shown in figure 4) click on Options and then Configuration. Set the DBUV SIPPER to INSTRUMENT and the FS 3000 to COM 2. Click on Accessory and the following parameters can be assigned for the Auto Sipper and the for the Autosampler.

i) Auto Sipper

In the Auto Sipper page set up the following parameters;

Operation mode	Automatic
Speed	30 rpm
Fill time	15 sec
Empty Time	0 sec
Settle time	5 sec

Note: If you require the sample to be returned to the test tube and not to waste, then set the Empty time to 15 seconds.

ii) Autosampler

In the Autosampler page set up the following parameters;

Operation mode	Automatic
Rinse	5 sec
Air Slug	0 sec
Height	150 mm

Report

The various parameters on this page can be tailored to your requirements.

Performing the analysis

Load the autosampler with the standards, samples and rinse solution. On the Application Control page click on RUN.

The samples will be analysed and the nitrate concentration in ppm will be reported for each sample.

References

1. “Ultraviolet Spectrophotometric Screen Method”. Standard methods for examining water and wastewater, 16th Edition. American Public Health Association. Washington, 1985, pages 392–393.