Application Note

Environmental Analysis

Simple, Rapid and Accurate Determination of Ammoniacal Nitrogen in Ground Waters Using the Agilent Cary 60 UV-Vis Spectrophotometer

Abstract

Ammoniacal Nitrogen is a nutrient that is vital for plant life, however it is toxic to aquatic organisms. The level of Ammoniacal Nitrogen in ground water is monitored to ensure that high levels are not making their way into waterways. This report details how the Agilent Cary 60 UV-Vis spectrophotometer can be used for simple, sensitive and accurate measurement of Ammoniacal Nitrogen in ground waters following a method accredited by the United Kingdom Accreditation Service (UKAS).

Authors

Trevor Hardy, Terra-Tek Ltd, Birmingham

Rob Wills,
Agilent Technologies, Cheadle

Introduction

UV-Vis spectrophotometry is a widely used analytical technique in the quantitative analysis of many inorganic and organic compounds. This technique is used because of it's speed and relative ease of use. In this application note we will demonstrate how the Agilent Cary 60 UV-Vis spectrophotometer can be used to produce accurate, reproducible quantitative results for Ammoniacal Nitrogen, a toxic pollutant harmful to aquatic organisms that can make it's way into ground water via sewage effluent or runoff from land where manure has been applied or stored.

Experimental

Ground waters, when received, have particulate matter suspended in them which can interfere with the analysis, therefore a 100ml portion of the sample is first filtered through a Whatman 5 (or equivalent) filter paper into a glass beaker. Using a glass pipette, a 25ml aliquot is then pipetted into a 50ml volumetric flask (grade b or better). At the same time a series of standards (0-10ppm) are made from a 100ppm stock solution. 25ml aliquots of these standards are pipetted into 50ml volumetric flasks (grade b or better).

Using an automatic pipettor 2ml of Potassium Sodium Tartrate and 2ml of Nessler's Solution are added to each sample and standard. The solutions are made up to the mark with de-ionised water and stoppered before shaking to ensure they are properly mixed. The samples will turn orange – brown dependent on the concentration - the colour takes 5 minutes to develop and is stable for up to 1 hour. Standards and samples are then analysed using a Cary 60 with glass cuvette using the parameters shown in the table 1 below.

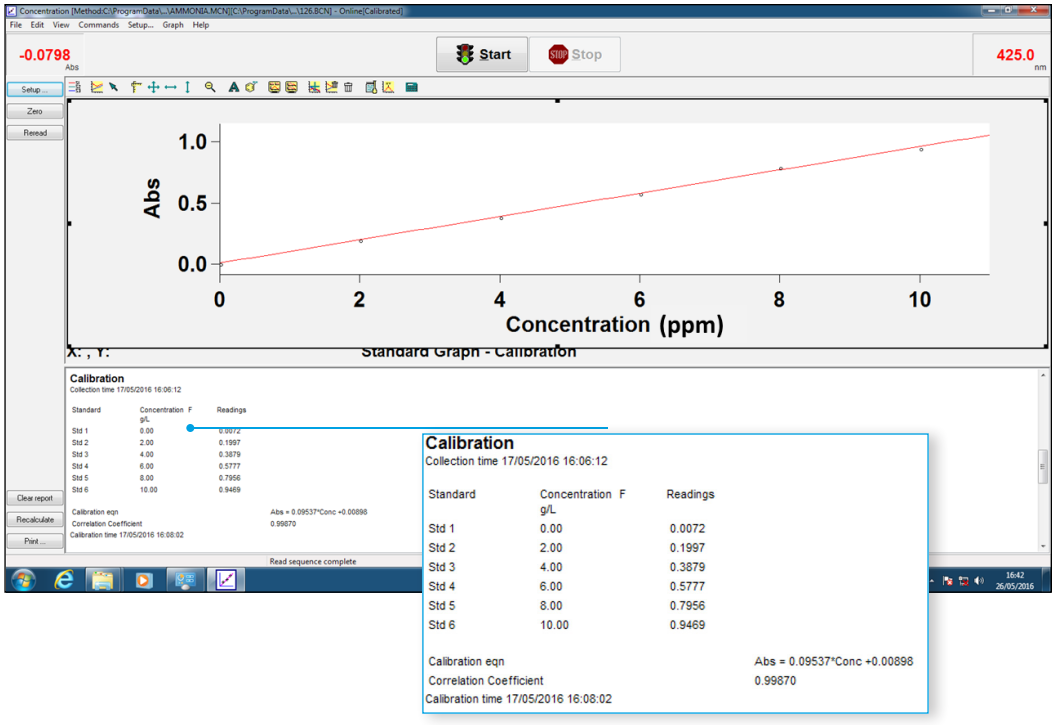
Instrument Wavelength	425nm
Signal Averaging Time (SAT)	0.1 secs
Replicates	1
Ymin	0 Abs
Ymax	1 Abs
Calibration Model	Linear Fit

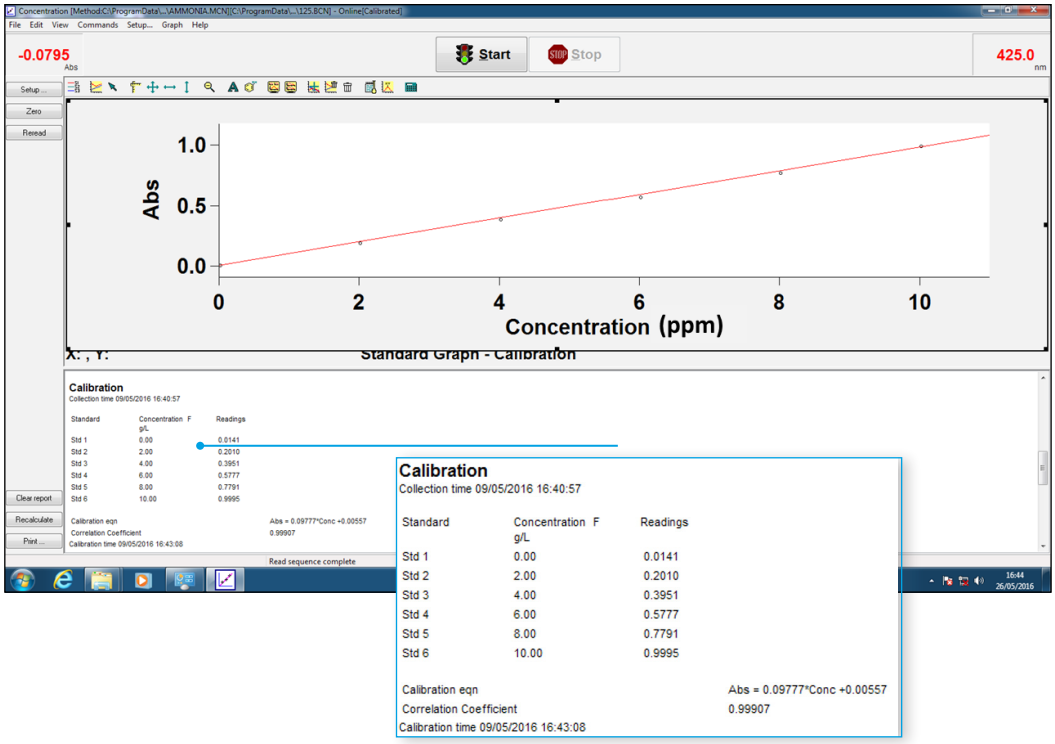
Table 1

NOTE: As the colour continues to develop with time, calibration standards should be made fresh with every analysis batch. This is done to ensure the accuracy of the results produced.

Results and Discussions

Below are screenshots showing three separate calibrations produced following this method. Clearly the calibration graphs exhibit a high degree of linearity, with correlation coefficients greater than 0.995 easily attainable.




-0.0795
Abs
Start
Stop
425.0
nm
Setup...
Zero
Reread
1.0
0.5
0.0
Abs
0
2
4
6
8
10
Concentration (ppm)
X: , Y:
Standard Graph - Calibration
Calibration
Collection time 09/05/2016 16:40:57
Standard
Concentration F g/L
Readings
Std 1 0.00 0.0141
Std 2 2.00 0.2010
Std 3 4.00 0.3951
Std 4 6.00 0.5777
Std 5 8.00 0.7791
Std 6 10.00 0.9995
Clear report
Recalculate
Print ...
Calibration eqn
Abs = 0.09777*Conc +0.00557
Correlation Coefficient
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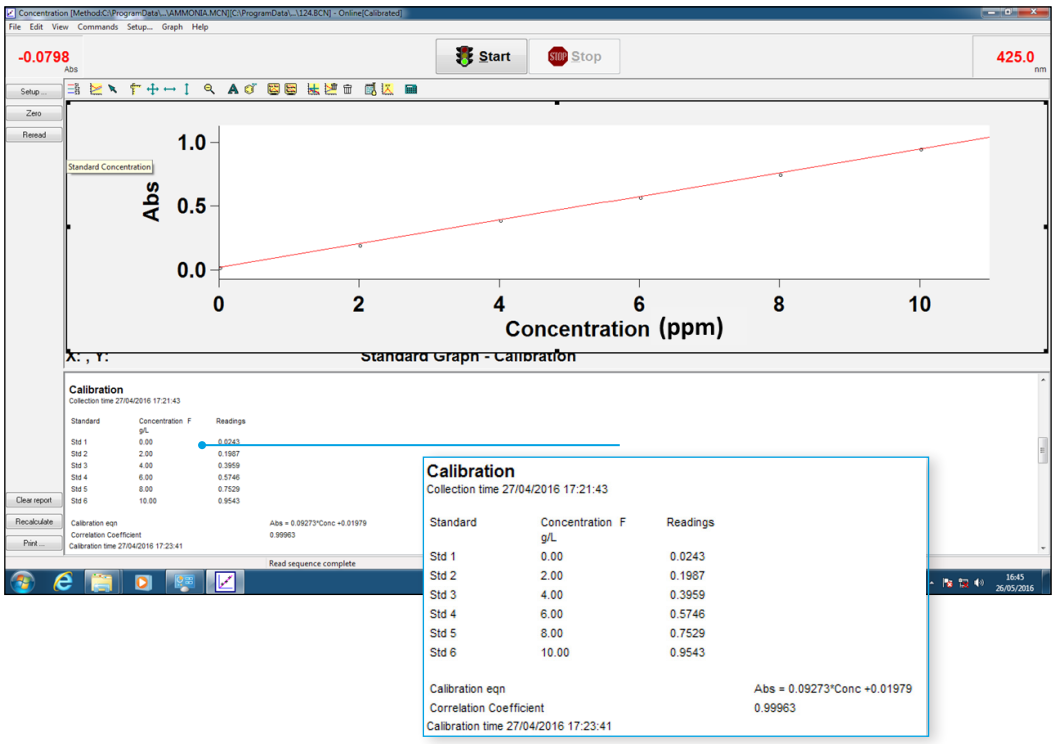
Calibration time 09/05/2016 16:43:08
Read sequence complete
16:44
26/05/2016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Abs
Start
Stop
425.0
nm
Setup...
Zero
Reread
Standard Concentration
1.0
0.5
0.0
Abs
0
2
4
6
8
10
Concentration (ppm)
X: , Y:
Standard Graph - Calibration
Calibration
Collection time 27/04/2016 17:21:43
Standard
Concentration F g/L
Readings
Std 1 0.00 0.0243
Std 2 2.00 0.1987
Std 3 4.00 0.3959
Std 4 6.00 0.5746
Std 5 8.00 0.7529
Std 6 10.00 0.9543
Clear report
Recalculate
Print ...
Calibration eqn
Abs = 0.09273*Conc +0.01979
Correlation Coefficient
0.99963
Calibration time 27/04/2016 17:23:41
Read sequence complete
16:45
26/05/2016

Standard Concentration (ppm)	Measured Concentration (ppm)	Absorbance Reading (Abs)
8.0	8.04	0.7548
8.0	8.03	0.7537
8.0	8.03	0.7540
8.0	8.03	0.7540
8.0	8.05	0.7558

Table 2. instrumental reproducibility based on replicate readings of the same standard.

Using this method it is possible to achieve an LOD of <0.1mg/L, which is far below the 0.5mg/L limit for ammonia in EU drinking water.

Conclusion

The results clearly demonstrate that the 60 UV-Vis spectrophotometer is suitable for the analysis of Ammoniacal Nitrogen in ground waters.

The calibration graphs show that a high degree of correlation is easily repeatable, and the data shown in Table 2 shows that measurements can be made with high accuracy and reproducibility.

The limit of detection using this approach is significantly lower than the LOD required in the legislation.

Acknowledgements

Agilent Technologies gratefully acknowledge the help of Terra Tek Ltd in the production of this application note. Terra Tek are one of the UK's largest providers of in-situ and laboratory geotechnical and chemical testing services. Their laboratories hold UKAS/Mcerts accreditation for a wide range of tests on soils, stabilised soils, aggregates, rock, construction materials and waters to British, European and International Standards.

Printed in the Europe, February 21, 2018
5991-9099EN

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