Ministry of Environment and Water
Executive Environment Agency
Sofia, Bulgaria

Analytical problems in case of ion-chromatographic determinations of anions and cations in water with low contamination

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Basic agreements of "Water Quality" Labs at Executive Environment Agency

- Programs for control, operative and study monitoring of ground, surface, waste and drinking water
- To implement requirements of Water Framework Directive 2000/60/EU (application V)
- To use appropriate instruments for all analytical activities on these monitoring programs

Ion-chromatographic system

- Metrohm

830 IC Interface

761 Compact IC

766 IC Sample Processor

Ion-chromatographic system

- Module for anions – F, Cl, NO_2, NO_3, PO_4, SO_4
- Column: Metrosep A Supp 5

Ion-chromatographic system

- Module for cations – Li, Na, NH_4, K, Ca, Mg
- Column: Metrosep C 4 250

Problems???

- Determination of ions at low concentration level, in particular NO_2, NH_4 and PO_4

- Use of sertified reference materials

Comparison of concentration range

According to the International standard EN ISO 10304 – 1,2

- Nitrite NO_2

from 0,05 to 20 mg/l

- Orthophosphate PO_4

from 0,1 to 20 mg/l

In our lab

- Nitrite NO_2

from 0,1 to 10 mg/l

- Orthophosphate PO_4

from 0,1 to 10 mg/l

Comparison of concentration range

According to the International standard EN ISO 14911

- Ammonium NH_4

from 0,1 to 10 mg/l by using loop 10 µl

In our lab

- Ammonium NH_4

from 0,1 to 20 mg/l by using loop 20 µl

Comparison of limits of detection

	Limits with IC system	Limits with UV spectra-photometric instrument
NO_2	0,05 mg/l	0,0011 mg/l
PO_4	0,05 mg/l	0,01 mg/l
NH_4	0,05 mg/l	0,005 mg/l

What about pure water matrixes?

- NO_2 and NH_4 at concentration level 0,00…mg/l
- PO_4 at concentration level 0,0… mg/l

Sertified reference materials

- Regulation of Accreditation Body

- Higher class SRM

Certificate, published from accredited in ISO 17025 Lab

INORGANIC VENTURES

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CERTIFICATE OF ANALYSIS

1.0 **INORGANIC VENTURES** is an ISO Guide 34:2000 registered Certified Reference Material (CRM) Manufacturer (Certificate #883-02). The certificate is designed and the data is determined in accordance with ISO Guide 31:2000 (Reference Materials-Contents of Certificates and Labels), ISO Guide 34:2000 "Quality System Guidelines for the Production of Reference Materials," and ISO Guide 35:1989 "Certification of Reference Materials - General and Statistical Principals."

2.0 **DESCRIPTION OF CRM** **1000 µg/mL Nitrate in Water**

Catalog Number:	ICNO31-1 and ICNO31-5
Lot Number:	**B2-NOX02057**
Starting Material:	Sodium Nitrate
Starting Material Purity (%):	99.000000
Starting Material Lot No:	12616AC
Matrix:	Water

3.0 **CERTIFIED VALUES AND UNCERTAINTIES**

Certified Concentration: 1000 ± 3 µg/mL

Certified Density: 0.998 g/mL (measured at 22° C)

The following equations are used in the calculation of the certified value and the uncertainty

Certified Value ($\bar{x}$) = $\frac{\Sigma x_i}{n}$

Uncertainty (±) = $\frac{2[(\Sigma s_i)^2]^{1/2}}{(n)^{1/2}}$

($\bar{x}$) = mean
x_i = individual results
n = number of measurements
Σs_i = The summation of all significant estimated errors (Most common are the errors from instrumental measurement, weighing, dilution to volume, and the fixed error reported on the NIST SRM certificate of analysis.)

4.0 **TRACEABILITY TO NIST AND VALUES OBTAINED BY INDEPENDENT METHODS**

"Property of the result of a measurement or the value of a standard whereby it can be related to stated references, usually national or international standards, through an unbroken chain of comparisons all having stated uncertainties." (ISO VIM, 2nd ed., 1993, definition 6.10)

This product is Traceable to NIST via an unbroken chain of comparisons. The uncertainties for each certified value are reported, taking into account the SRM uncertainty error and the measurement, weighing and volume dilution errors. In rare cases where no NIST SRMs are available, the term 'in-house std.' is specified.

4.1 **Assay Method #1** **1000 ± 3 µg/mL (Avg. of 2 Runs)**
IC Assay NIST SRM 3185 Lot Number: 050517

Assay Method #2 **1001 ± 5 µg/mL**
Calculated NIST SRM Lot Number: See Sec. 4.2

MERCK

Certificate of Analysis

http://certificates.merck.de

Date of print: 30.09.2008

1.19811.0500 Nitrate standard solution traceable to SRM from NIST $NaNO_3$ in H_2O 1000 mg/l NO_3^- CertiPUR®

Batch HC754533

	Batch Values	
Concentration β (NO_3^-)	1002	mg/l

Determination method: Alkalimetric titration.
(traceable to NIST - SRM 723d)
Accuracy of the method: +/- 5 mg/l

Test date (DD.MM.YYYY): 05.07.2007
Minimum shelf life (DD.MM.YYYY): 31.07.2010

Wolfgang Gernand
responsible laboratory manager quality control

This document has been produced electronically and is valid without a signature

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Thank you for your attention