Application Bulletin 121/2 e

Determination of nitrate with an ion-selective electrode

Industry sector

Chemical; environmental testing; food & beverage; fertilizers & explosives

Keywords

Nitrate; NO_3 ISE; polymer membrane; combined ion-selective electrode; standard addition; direct measurement; STDADD; soil; fertilizer; juice; water; purity; pharmaceutical products; nitrate salt; plating bath; 6.00510.120; S01; S010; S02; S020; S021; S023; S027; S07; S070; S078; S079; S11; S110; S111; S112

Summary

This bulletin describes several nitrate determinations in different matrices using the combined nitrate ion-selective electrode. Thanks to a fracture-proof shaft made of propylene and an impact protector for the polymer membrane, this sensor is mechanically very robust. The electrode is combined with a reference electrode.

In the first section of this bulletin, some general tips are given for the handling of the electrode and the determination of nitrate. In the second part, practical examples are used to demonstrate how determinations can be performed using either direct measurement or the standard addition technique. Further methods and matrices are described in the *Appendix*, including nitrate determination in animal feed, beer, soil, copper baths, and fertilizer.

Instruments and accessories

- Ion Meter or Titrator with the modes MEAS CONC and/or STDADD
- Stirrer
- Buret

Electrodes

Combined polymer membrane electrode, NO_3	6.00510.120
Pt1000 temperature sensor	6.1110.100

General tips

For optimal measurement results, the following points are essential to follow:

- Clean the electrode using the spray rinse and/or dip rinse after each measurement with deionized (DI) water.
- The sensing part of the combined nitrate ISE is made of a polymer membrane containing an ion specific ionophore. Avoid any fat or oil deposition or scratching of the membrane—this means it should neither be touched with bare hands nor cleaned with abrasive materials.
- The lifetime of a polymer membrane electrode is limited. The mean lifetime with normal lab use is about six months, however the lifetime strongly depends on the application type and how the electrode is maintained.
- The combined nitrate ISE must be stored dry with some residual moisture (e.g., some drops of deionized water) in the electrode vessel to keep the reference system ready.
- The electrode can be stored temporarily in $c(KNO_3) = 0.01$ mol/L. Storage in reference electrolyte ($c(KCl) = 3$ mol/L) is not recommended as the electrolyte does not contain any NO_3^- ions and therefore will leach out the ionophore, or other ions might block the connecting sites.
- If the electrode has not been used for a longer period, conditioning of the polymer membrane in $c(KNO_3) = 0.01$ mol/L for 30 minutes is recommended to shorten the response time.
- The electrode should only be used in aqueous solutions as otherwise the polymer membrane is affected (softeners are leached out, resulting in a hard membrane that does not respond properly anymore).
- When conducting a series of measurements and an increase of the measured content is visible under otherwise identical conditions, the electrode should be conditioned for 5 minutes in $c(KNO_3) = 0.01$ mol/L.

- The NO_3 ISE has a high cross sensitivity for chloride and bromide ions. Therefore, it is important to remove them from the sample matrix prior to analysis by using e.g., a Nitrate Interference Suppressor Solution (NISS). Information on interfering ions related to the NO_3 ISE can be found in the corresponding leaflet: Manual for ion-selective electrodes (8.109.8042).
- The pH of the sample must be adjusted from 2.5 to 11. If the solution is too acidic, then H^+ ions may interfere.
- To check the performance of the NO_3 ISE, the potentials of two solutions, first $c(KNO_3) = 10^{-4}$ mol/L and second $c(KNO_3) = 10^{-3}$ mol/L, are measured. The potential difference between the two measurements should be higher than 47.3 mV (this equates to a value higher than 80% of the Nernst voltage which is 59.16 mV at 298.15 K = 25 °C).

Choice of procedure

The procedure chosen depends on the sample matrix, the number of samples to be analyzed, and the concentration range of the samples.

- For samples with a complex or unknown matrix composition, standard addition is the procedure of choice.
- A direct measurement is recommended for samples with an unproblematic sample matrix, as well as for large sample series or online measurements.
- For low concentration measurements, it is recommended to either use direct measurement after calibration or spike the sample to a higher nitrate content. The reasoning is that the sensor may be at its limit of detection and outside of the linear range, giving results for the standard addition that are too high.

Sample preparation and parameters

The sample preparation and the parameters are mentioned in the *Practical examples* section and in the *Appendix*, respectively.

Direct measurement

The direct measurement option is recommended for unproblematic samples and in the case of low-level nitrate measurements (mg/L or µg/L range).

When using direct measurement, the following points must be considered:

- In general, standard solutions must have the same ionic background as the sample solutions. ISA (Ionic Strength Adjuster) should be used for such measurements.
- For calibration standards and samples with concentrations <1 mg/L, diluted ISA should be used. Otherwise, the response time of the sensor becomes noticeably longer.
- All measurements should be performed at a constant temperature (e.g., 25 °C) as the slope of the ISE is temperature-dependent (Nernst Equation).
- For more accurate results, the use of a temperature sensor or a thermostated titration vessel is recommended.
- Perform the calibration and the measurements with identical stirring conditions.

Standard addition (STDADD)

The standard addition is recommended for undefined or complex sample matrices.

There are two types of standard addition in OMNIS:

- STDADD ISE dos
- STDADD ISE auto

The quickest and recommended method is the automatic standard addition (mode: « STDADD ISE auto »). For exact results, the potential difference (ΔU) should be at least 12 mV per standard addition, and at least three standard additions should be performed (i.e., total ΔU at least 36 mV). If precisely defined volumes of the standard additions are required but the maximum ease of operation is desirable, the mode « STDADD ISE dos » is recommended. There the individual standard addition volumes can be defined (see manual of the device in use or the online help found in the software).

The calculation of the result is automatically carried out by OMNIS applying an iteration procedure.

When using standard addition, the following points must be considered:

- Stirring is necessary during additions. Additions without continuous stirring lead to false results.
- If the total volume added during standard addition is higher than 10% of the initial solution, then the sample should be diluted prior to the analysis with ISA or deionized water. Alternatively, try to reduce the sample size or use a stronger standard solution. If the added volume is too large, there is a risk of dilution error and the linearity can no longer be guaranteed, resulting in incorrect data.

- To ensure accurate evaluation of the standard addition, the following standard concentrations (c_{std}) for the different buret volumes (V_{buret}) as a function of the sample concentration (c_{smpl}) are recommended according to **Table 1**. Thereby any sample dilution should be considered (e.g., dilution with ISA).

Volume of buret (mL)	Ratio [c_{std} : c_{smpl}]
5	40 : 1
10	20 : 1
20	10 : 1
50	5 : 1

Table 1. Ratio of the standard concentration and sample concentration, dependent on the buret volume.

Example factor determination

Sample concentration, c_{smpl} :	100 mg/L
Buret volume, V_{buret} :	10 mL
Sample size :	10 mL
Addition of ISA :	20 mL
Addition of deionized water :	10 mL
Auxiliary solution volume :	30 mL
Total volume :	40 mL
Factor from Table 1 (c_{std} : c_{smpl}) :	20

Considering the dilution with ISA and deionized water, the initial sample concentration is 25 mg/L. Therefore, the optimal recommended concentration of the standard is: 25 mg/L × 20 = 500 mg/L.

Practical examples

Reagents

The following reagents and recommended concentrations are used to prepare the nitrate standard and ISA solution:

- Ammonium sulfate, $(NH_4)_2SO_4$, ≥ 99.0%
- Aluminum sulfate, $Al_2(SO_4)_3$, ≥ 99.0%
- Potassium nitrate, KNO_3, ≥ 99.0%

Or alternatively:

- $c(KNO_3)$ = 1.0 mol/L electrolyte solution from Metrohm, 62310010, corresponds to $\beta(KNO_3)$ = 101.1032 g/L = $\beta(NO_3^-)$ = 62.0049 g/L

For sample preparation purposes, other chemical reagents like acids, bases, or extraction solutions might be necessary. They are specified in the respective chapters.

Other standard concentrations can be prepared—with the appropriate dilution—from the standard stock solution with $\beta(NO_3^-)$ = 10.0 g/L or the Metrohm $c(KNO_3)$ = 1.0 mol/L electrolyte solution.

The choice of ISA depends on the sample and the matrix; it is important to pay attention to the necessary ionic background and that no side reactions happen. Aluminum sulfate, for example, is recommended for juices, especially beetroot juice. Aluminum sulfate has additional advantages – it destroys HCO_3^- and blocks interfering R-COOH groups.

Solutions

Nitrate standard stock solution	$\beta(NO_3^-)$ = 10.0 g/L 16.31 g KNO_3 is weighed into a 1 L volumetric flask. 500 mL deionized water is added, and the potassium nitrate is dissolved while swirling. Then, the flask is filled up to the mark with deionized water. Alternatively: 161.32 mL $c(KNO_3)$ = 1.0 mol/L electrolyte solution is added into a 1 L volumetric flask and filled up to the mark with deionized water.
ISA Ammonium sulfate	$c((NH_4)_2SO_4)$ = 1.0 mol/L 13.21 g $(NH_4)_2SO_4$ is weighed into a 100 mL volumetric flask. 50 mL deionized water is added, and the ammonium sulfate is dissolved while

	swirling. Then, the flask is filled up to the mark with deionized water.
ISA Aluminum sulfate	$c(Al_2(SO_4)_3) = 0.1$ mol/L 3.42 g $Al_2(SO_4)_3$ is weighed into a 100 mL volumetric flask. 50 mL deionized water is added, and the aluminum sulfate is dissolved while swirling. Then, the flask is filled up to the mark with deionized water.
Conditioning solution	$c(KNO_3) = 0.01$ mol/L 1.01 g KNO_3 is weighed into a 1 L volumetric flask. 500 mL deionized water is added, and the potassium nitrate is dissolved while swirling. Then, the flask is filled up to the mark with deionized water.
Nitrate Interference Suppressor Solution (NISS)	It is highly recommended to buy this solution directly from a supplier.

Comments

- Historically, interfering ions were removed by using different chemicals. Carbonates and bicarbonates were removed by adjusting the pH of the sample to 4.5 with sulfuric acid. Silver sulfate was added to remove halogens, cyanides, and phosphates (0.1 g Ag_2SO_4 removes about 22 mg Cl^-).
- Today's NISS solutions remove most of the same interfering ions and adjust the pH value to the acidic range. It is therefore recommended to use this.
- A solution with properties similar to NISS, an ISS (Interference Suppressor Solution), can be prepared as follows*:
  0.125 g Ag_2SO_4, 0.125 g sulfamic acid (H_2NSO_3H), 0.125 g $Al_2(SO_4)_3$, 0.106 g $(NH_4)_2SO_4$, and 0.012 g H_3BO_3 are weighed into a 25 mL volumetric flask. 20 mL deionized water is added, and the salts are dissolved while swirling. Then, the flask is filled up to the mark with deionized water.

*Lit.: Fresenius, Z. A double-membrane nitrate ion-selective electrode based on aliquat-nitrate in paraffin. *Anal. Chem.,* **1989**, *333*, 619-623.

Nitrate in surface water by means of direct measurement

Sample

- Surface water, nitrate content approximately 54 mg/L

Solutions

- Nitrate standard solution, $\beta(NO_3^-) = 1000$ mg/L; made of nitrate standard stock solution/deionized water (1:10)
- ISA, aluminum sulfate, $c(Al_2(SO_4)_3) = 0.1$ mol/L
- Conditioning solution, $c(KNO_3) = 0.01$ mol/L

Sample preparation

No sample preparation is required.

Standard preparation

It is highly recommended to prepare all standard solutions directly in-situ by Metrohm devices with the nitrate standard solution $\beta(NO_3^-) = 1000$ mg/L. The following table shows the preparation for each 40 mL ready to measure standard solution.

Standard	1	2	3	4
Addition of $\beta(NO_3^-) = 1000$ mg/L	1 mL	2 mL	3 mL	4 mL
Addition of deionized water	37 mL	36 mL	35 mL	34 mL
Addition of ISA $c(Al_2(SO_4)_3) = 0.1$ mol/L	2 mL			
Final volume	40 mL			
Concentration in mg/mL	**25**	**50**	**75**	**100**

Analysis

Calibration

The prepared ready to measure standard solutions (numbers 1 to 4) are stirred, and the potential of each standard is measured. In between each measurement, the electrode is rinsed well with deionized water and then conditioned in $c(KNO_3) = 0.01$ mol/L for 30 s prior to measuring the next standard.

Sample

2 mL of ISA is added to 40 mL of sample solution and the potential is measured. In between each measurement, the electrode is rinsed well with deionized water and then conditioned in $c(KNO_3) = 0.01$ mol/L for 30 s before measuring the next sample. The dilution factor of the sample must be considered for the result calculation.

Parameters

Calibration

Mode	CAL Conc
Stirring rate	8
Calibration standards	4 (25, 50, 75, and 100 mg/L)
Signal drift	0.5 mV/min
Min. waiting time	30 s
Max. waiting time	215 s

Sample

Mode	MEAS Conc
Stirring rate	8
Measuring parameters	Drift-controlled measurement
Signal drift	0.5 mV/min
Min. waiting time	10 s
Max. waiting time	215 s

Calculations

Nitrate calibration

The calibration is automatically calculated by OMNIS after the « CAL CONC » command is done. The calibration data can be found in the sample list under calibration curves.

The « CAL WRITE » command offers an additional option of writing the calibration data directly to the electrode.

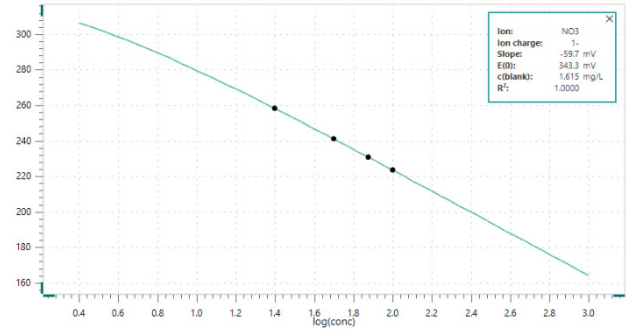
Nitrate content

$$NO_3^- = \frac{\text{MEAS Conc}_{Final}}{m_v} \times (m_v + ISA_v)$$

NO_3^-:	Nitrate content in mg/L
MEAS Conc_{Final}:	Final meas value of the « MEAS CONC » command in mg/L
m_v:	Sample size in mL
ISA_v:	Volume of added ISA in mL

Example

Calibration



Subsample name	Concentration / mg/L	Potential – End value / mV	Temperature / °C	Time / s
1000 mg/L Std1	25.000	258.2	25.0	31.4
1000 mg/L Std1	50.000	241.1	25.2	31.4
1000 mg/L Std1	75.000	230.8	25.3	40.1
1000 mg/L Std1	100.000	223.5	25.3	31.4

Figure 1. Calibration curve of four standard solutions with 25 mg/L, 50 mg/L, 75 mg/L, and 100 mg/L nitrate.

Slope in mV	E(0) in mV	c(blank) in mg/L	R^2	Variance
-59.7	343.3	1.615	1.000	0.008

Sample

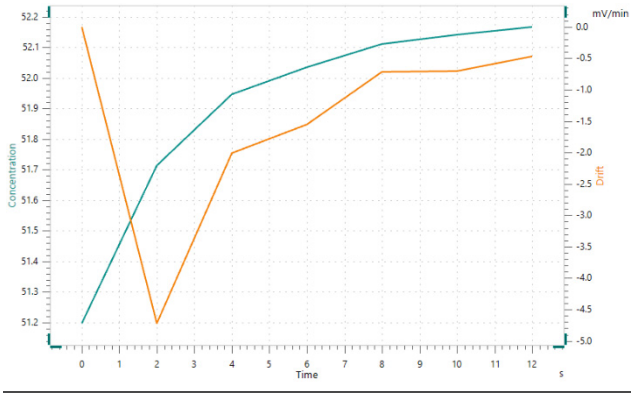


Figure 2. Measurement of surface water with approximately 54 mg/L nitrate.

Comments

The addition of ISA to the sample must be considered in the calculation because the ISA dilutes the sample a little.

Nitrate in juices with automatic standard addition

Samples

- Beetroot juice
- Carrot juice

Solutions

- Nitrate standard solution, $\beta(NO_3^-) = 1000$ mg/L; made of nitrate standard stock solution/deionized water (1:10)
- ISA, aluminum sulfate, $c(Al_2(SO_4)_3) = 0.1$ mol/L
- Conditioning solution, $c(KNO_3) = 0.01$ mol/L

Sample preparation

The juice must be shaken well before the analysis to ensure that it is as homogeneous as possible.

Analysis

Sample

To 0.5 mL (beetroot juice) or 2 mL (carrot juice) of sample, 2 mL of ISA is added and the total volume is adjusted to 50 mL with deionized water. Afterwards, the standard addition is carried out with the nitrate standard solution. In between each measurement, the electrode is rinsed well with deionized water and then conditioned in $c(KNO_3) = 0.01$ mol/L for 30 s before measuring the next sample.

Parameters

Sample

Mode	STDADD ISE auto
Stirring rate	8
Auxiliary solution volume	Total volume of ISA and deionized water
Number of additions	3
Delta U	20 mV
Dosing rate	Fast
Stop volume	Buret volume
Signal drift	0.5 mV/min
Min. waiting time	10 s
Max. waiting time	215 s

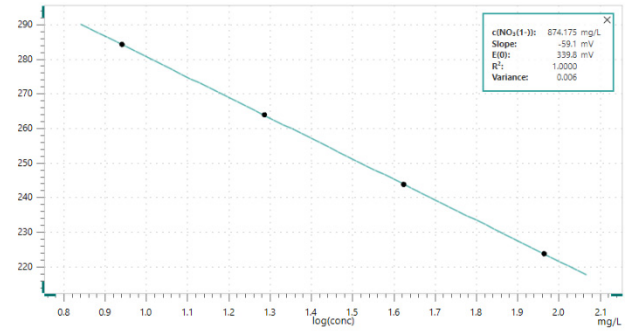
Calculations

Nitrate linear regression

The linear regression is automatically executed by OMNIS after the « STDADD ISE auto » command is finished. The data can be found in the sample list under calibration curves.

Example

Sample



	Volume increment / mL	Potential / mV	Potential difference / mV	Temperature / °C	Duration / s
Sample		284.2		24.8	12.4
Increment 1	0.541	263.9	-20.3	24.8	44.4
Increment 2	1.201	243.8	-20.1	24.9	56.2
Increment 3	2.860	223.7	-20.0	25.0	89.7

Figure 3. Standard addition of beetroot juice with three increments made with $\beta(NO_3^-) = 1000$ mg/L.

Slope in mV	E(0) in mV	R^2	Variance
-59.1	339.8	1.000	0.006

Comments

Aluminum sulfate is better suited as ISA for juices because it destroys HCO_3^- and blocks interfering R-COOH groups.

If the sample has a particularly sodium chloride high content or (more) other interfering ions, it is advisable to replace the ISA completely with NISS.

Information about interfering ions regarding the NO_3 ISE can be found in the corresponding leaflet: Manual for ion-selective electrodes (8.109.8042).

References

Monograph: Electrodes in Potentiometry, 8.015.5013

Leaflet: Manual for ion-selective electrodes, 8.109.8042

APHA 4500-NO3- Nitrogen (Nitrate) Method D: Nitrate-selective electrode method

COMISSION REGULATION (EU) No 1258/2011

EPA:
SW-846 Test Method 9216: Potentiometric Determination of Nitrite in Aqueous Samples with Ion-Selective Electrode

Fresenius, Z. A double-membrane nitrate ion-selective electrode based on aliquat-nitrate in paraffin. *Anal. Chem.,* **1989**, *333*, 619-623.

AOAC method 986.31, Nitrate in Forages. Potentiometric method. *AOAC,* **1989**, *2*

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Appendix

Sample preparations for further application examples

1. Purity of lucigenin by determination of nitrate with automatic standard addition

Sample preparation

Approximately 70 mg of dried lucigenin is weighed into a 50 mL volumetric flask, dissolved in approximately 30 mL deionized water, and the flask is then filled to the mark with deionized water.

ISA

Aluminum sulfate, $c(Al_2(SO_4)_3) = 0.1$ mol/L

Standard solution

Nitrate standard solution, $c(KNO_3) = 0.1$ mol/L corresponds to $\beta(NO_3^-) = 6200$ mg/L

Analysis

To 5 mL of sample, 2 mL of ISA is added and the total volume is adjusted to 50 mL with deionized water. Afterwards, the standard addition is carried out with the nitrate standard solution $\beta(NO_3^-) = 6200$ mg/L. In between each measurement, the electrode is rinsed well with deionized water and then conditioned in $c(KNO_3) = 0.01$ mol/L for 30 s before the next sample is measured.

Calculation

OMNIS directly calculates the concentration in mg/L iteratively. The purity of the sample must be calculated taking dilution into account:

$$\text{Purity} = \frac{\text{Sample Conc}_{\text{Result}} \times 50 \times d_f \times M_{\text{Lucigenin}}}{m_s \times M_{NO3}}$$

Purity:	Purity of lucigenin in %
$\text{Sample Conc}_{\text{Result}}$:	Command variable « SampleConcentration.Result."STDADD ISE auto" » in mg/L
50:	Conversion factor including 100 for % divided by the stoichiometric factor 2
d_f:	Dilution factor of the sample, here 10
$M_{\text{Lucigenin}}$:	Molecular weight of lucigenin, $M(C_{28}H_{22}N_4O_6) = 510.506$ g/mol
m_s:	Sample size of sample preparation in mg
M_{NO3}:	Molecular weight of nitrate, $M(NO_3^-) = 62.0049$ g/mol

2. Nitrate in soil with automatic standard addition

Sample preparation

10 g of dried soil is weighed into a 100 mL glass beaker and slurried in 50 mL deionized water* with a magnetic stirrer for 10 min. Then, the soil-water-solution is filtered into a 100 mL volumetric flask through a folded filter (the filter is rinsed several times with deionized water) and filled to the mark with deionized water.

*Please note, it is advisable adjust the pH value below 4 in soil samples. In this way, disruptive influences from HCO_3^- ions can be minimized. While preparing the sample, 10 mL $c(H_2SO_4) = 0.1$ mol/L can be added additionally to lower the pH value.

ISA

Aluminum sulfate, $c(Al_2(SO_4)_3) = 0.1$ mol/L

Standard solution

Nitrate standard solution, $\beta(NO_3^-) = 1000$ mg/L

Analysis

To 10 mL of prepared sample solution, 2 mL of ISA* is added and the total volume is adjusted to 50 mL with deionized water. Afterwards, the standard addition is carried out with the nitrate standard solution $\beta(NO_3^-) = 1000$ mg/L. In between each measurement, the electrode is rinsed well with deionized water and then conditioned in $c(KNO_3) = 0.01$ mol/L for 30 s before the next sample is measured.

*Alternatively, the ISA can be replaced by NISS.

Calculation

OMNIS directly calculates the concentration in mg/L iteratively. The nitrate content of the sample must be calculated taking dilution into account:

$$NO_3^- = \frac{\text{Sample Conc}_{\text{Result}} \times V_F}{m_s}$$

NO_3^-: Nitrate content in mg/kg

$\text{Sample Conc}_{\text{Result}}$: Command variable « SampleConcentration.Result."STDADD ISE auto" » in mg/L

V_F: Final volume of sample preparation in mL

m_s: Sample size of sample preparation in g

Comment

The nitrate levels in soil are usually between 1 and 60 mg/kg NO_3^-.

3. Nitrate in lettuce or spinach with automatic standard addition

Sample preparation

10 g of lettuce or spinach leaves are weighed into a suitable food blender and approximately 50 mL deionized water is added. After mixing, the solution is filtered into a 100 mL volumetric flask through folded filter paper and filled to the mark with deionized water.

ISA

Aluminum sulfate, $c(Al_2(SO_4)_3) = 0.1$ mol/L

Standard solution

Nitrate standard solution, $\beta(NO_3^-) = 1000$ mg/L

Analysis

To 1 mL of prepared sample solution, 2 mL of ISA is added and the total volume is adjusted to 50 mL with deionized water. Afterwards, the standard addition is carried out with the nitrate standard solution $\beta(NO_3^-) = 1000$ mg/L. In between each measurement, the electrode is rinsed well with deionized water and then conditioned in $c(KNO_3) = 0.01$ mol/L for 30 s before the next sample is measured.

Calculation

OMNIS directly calculates the concentration in mg/L iteratively. The nitrate content of the sample must be calculated taking dilution into account:

$$NO_3^- = \frac{\text{Sample Conc}_{\text{Result}} \times V_F}{m_s}$$

NO_3^-: Nitrate content in mg/kg

Sample $Conc_{Result}$: Command variable « SampleConcentration.Result."STDADD ISE auto" » in mg/L

V_F: Final volume of sample preparation in mL

m_S: Sample size of sample preparation in g

4. Nitrate in beer with automatic standard addition

Sample preparation

The beer must be completely degassed. Purging with nitrogen has proven to be a suitable method.

ISA

Aluminum sulfate, $c(Al_2(SO_4)_3)$ = 0.1 mol/L

Standard solution

Nitrate standard solution, $\beta(NO_3^-)$ = 1000 mg/L

Analysis

To 20 mL of prepared sample, 20 mL* of ISA is added and the total volume is adjusted to 50 mL with deionized water. Afterwards, the standard addition is carried out with the nitrate standard solution $\beta(NO_3^-)$ = 1000 mg/L. In between each measurement, the electrode is rinsed well with deionized water and then conditioned in $c(KNO_3)$ = 0.01 mol/L for 30 s before the next sample is measured.

*In application tests, the 1:1 mixture of beer with ISA has proven as the most suitable procedure for reproducible results. Smaller additions of ISA (for example only 5 mL) could also work, depending on the beer sample.

Calculation

OMNIS directly calculates the concentration in mg/L iteratively.

Comment

The nitrate levels in beer are usually between 10 and 50 mg/L NO_3^-.

5. Nitrate in orange juice with automatic standard addition

Sample preparation

No sample preparation is required.

ISA

Aluminum sulfate, $c(Al_2(SO_4)_3)$ = 0.1 mol/L

Standard solution

Nitrate standard solution, $\beta(NO_3^-)$ = 1000 mg/L

Analysis

To 20 mL of sample, 5 mL of ISA is added and the total volume is adjusted to 50 mL with deionized water. Afterwards, the standard addition is carried out with the nitrate standard solution $\beta(NO_3^-)$ = 1000 mg/L. In between each measurement, the electrode is rinsed well with deionized water and then conditioned in $c(KNO_3)$ = 0.01 mol/L for 30 s before the next sample is measured.

Calculation

OMNIS directly calculates the concentration in mg/L iteratively.

Comment

The nitrate levels in orange juice are usually between 20 and 50 mg/L NO_3^-.

6. Nitrate in fertilizer with automatic standard addition

Sample preparation

Approximately 1 g of fertilizer is weighed into a 1 L volumetric flask. 10 mL $c(H_2SO_4) = 0.1$ mol/L and approximately 100 mL deionized water are added and the fertilizer is dissolved while swirling. Afterwards, the flask is filled up to the mark with deionized water.

ISA

Aluminum sulfate, $c(Al_2(SO_4)_3) = 0.1$ mol/L

Standard solution

Nitrate standard solution, $\beta(NO_3^-) = 10.0$ g/L

Analysis

To 25 mL of prepared sample solution, 5 mL of ISA is added and the total volume is adjusted to 50 mL with deionized water. Afterwards, the standard addition is carried out with the nitrate standard solution $\beta(NO_3^-) = 10.0$ g/L. In between each measurement, the electrode is rinsed well with deionized water and then conditioned in $c(KNO_3) = 0.01$ mol/L for 30 s before the next sample is measured.

Calculation

OMNIS directly calculates the concentration in mg/L iteratively. The nitrate content of the sample must be calculated taking dilution into account:

$$NO_3^- = \frac{\text{Sample Conc}_{\text{Result}} \times V_F}{m_s \times 10}$$

NO_3^-: Nitrate content in %

$\text{Sample Conc}_{\text{Result}}$: Command variable « SampleConcentration.Result."STDADD ISE auto" » in mg/L

V_F: Final volume of sample preparation in L

m_s: Sample size of sample preparation in g

10: Conversions factor for %

7. Nitrate in a copper bath with automatic standard addition

Sample preparation

Approximately 50 mL of the copper bath sample is added into a 1 L volumetric flask. 500 mL deionized water is added and the sample is dissolved while swirling. Then, the flask is filled up to the mark with deionized water.

ISA

Aluminum sulfate, $c(Al_2(SO_4)_3) = 0.1$ mol/L

Nitrate Interference Suppressor Solution (NISS)

Standard solution

Nitrate standard solution, $\beta(NO_3^-) = 10.0$ g/L

Analysis

To 20 mL of prepared sample solution, 5 mL of ISA and 5 mL of NISS are added and the total volume is adjusted to 50 mL with deionized water. Afterwards, the standard addition is carried out with the nitrate standard solution $\beta(NO_3^-) = 10.0$ g/L. In between each measurement, the electrode is rinsed well with deionized water and then conditioned in $c(KNO_3) = 0.01$ mol/L for 30 s before the next sample is measured.

Calculation

OMNIS directly calculates the concentration in mg/L iteratively. The nitrate content of the sample must be calculated taking dilution into account:

$$NO_3^- = \frac{\text{Sample Conc}_{\text{Result}} \times V_F}{m_v}$$

NO_3^-: Nitrate content in mg/L

$\text{Sample Conc}_{\text{Result}}$: Command variable « SampleConcentration.Result."STDADD ISE auto" » in mg/L

V_F: Final volume of sample preparation in mL

m_v: Sample size of sample preparation in mL

Comment

If the nitrate content of the copper bath is low (<50 mg/L), it is recommended to use $\beta(NO_3^-)$ = 1000 mg/L as nitrate standard solution.

8. Nitrate in animal feed (forage) with automatic standard addition

Sample preparation

Since there are many different forms of forage (e.g., pressed, milled, dried, pellets, water-soluble, non-water-soluble, partially water-soluble, etc.), only general sample preparation (extraction) is discussed here.

The dried sample is ground in a mortar until the particle size is about 2 mm. If the mortar is not a suitable method for grinding the sample, an alternative can be chosen (e.g., a food blender).

Approximately 10 g of the prepared sample is weighed into a 500 mL glass beaker, 10 mL $c(H_2SO_4)$ = 0.1 mol/L and approximately 200 mL deionized water are added, and the sample is extracted with a magnetic stirrer for 15 min. After extraction, the solution is filtered into a 1 L volumetric flask through a folded filter paper (the filter is rinsed several times with deionized water) and filled to the mark with deionized water.

ISA

Aluminum sulfate, $c(Al_2(SO_4)_3)$ = 0.1 mol/L

Nitrate Interference Suppressor Solution (NISS)

Standard solution

Nitrate standard solution, $\beta(NO_3^-)$ = 10.0 g/L

Analysis

To 20 mL of prepared sample solution, 5 mL of ISA and 5 mL of NISS are added and the total volume is adjusted to 50 mL with deionized water. Afterwards, the standard addition is carried out with the nitrate standard solution $\beta(NO_3^-)$ = 10.0 g/L. In between each measurement, the electrode is rinsed well with deionized water and then conditioned in $c(KNO_3)$ = 0.01 mol/L for 30 s before the next sample is measured.

Calculation

OMNIS directly calculates the concentration in mg/L iteratively. The nitrate content of the sample must be calculated taking dilution into account:

$$NO_3^- = \frac{\text{Sample Conc}_{\text{Result}} \times V_F}{m_s \times 10}$$

NO_3^-: Nitrate content in %

Sample $Conc_{Result}$: Command variable « SampleConcentration.Result."STDADD ISE auto" » in mg/L

V_F: Final volume of sample preparation in L

m_S: Sample size of sample preparation in g

10: Conversions factor for %

Comments

If the nitrate content of the prepared sample is low (<50 mg/L), it is recommended to use $\beta(NO_3^-)$ = 1000 mg/L as nitrate standard solution.

Essentially, nitrate contents in animal feeds below 0.5% (calculated on dry matter) are harmless for animals. On the other hand, a nitrate content above 3% (calculated on dry matter) is considered unsuitable for feeding. Values in between must be assessed individually.