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# ***EasyChem Methodology***

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## **Nitrate by Discrete Analysis**

### **Systea Easy (1-Reagent) Nitrate Method**

### **(Colorimetric, Automated, 1 Reagent)**

Revision Date:  
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# EasyChem Applications

## **Systema Easy (1-Reagent) Nitrate**

### Nitrate in Water and Wastewater

#### 1.0 Scope and Application

1.1 This method is applicable to the analysis of nitrate (CASRN 14797-55-8) in wastewater and drinking water. If this method is being used for compliance with the Clean Water Act (CWA) the flexibility to modify this method as described in CFR 136.6 applies. Modifications to this method are not allowed when it is being used for compliance monitoring under the Safe Drinking Water Act (SDWA). Requirements for establishing method equivalency for CWA use are given in Section 9.1.2. Each laboratory must demonstrate that the performance of the modified method is acceptable using this procedure.

#### 2.0 Summary of Method

2.1 The automated procedure for the determination of nitrate utilizes the reaction whereby nitrate is reduced to nitrite by a proprietary Chemical R1. The reduced nitrate is then treated with sulfanilamide and N-1-naptylethylenediamine dihydrochloride under acidic conditions to form a soluble dye which is measured colorimetrically at 546nm. The final product measured represents the nitrite ion originally present plus that formed from the nitrate. Regardless of the sample matrix used, recovery of  $\text{NO}_3$  to  $\text{NO}_2$  is consistently between 95% and 105% recovery.

In order to determine nitrate levels, the nitrite alone must be subtracted from the total (nitrate+nitrite).

#### 3.0 Common Abbreviations

3.1 The abbreviations below are specific to this method, but have been conformed to common usage as much as possible.

CWA	Clean Water Act
DI Water	Deionized Water
EPA	Environmental Protection Agency
CASRN	Chemical Abstracts Service Registry Number
CERCLA	Comprehensive Environmental Resource Compensation Liability Act
IPR	Initial Precision and Recovery
MDL	Method Detection Limit
MS	Matrix Spike
MSD	Matrix Spike Duplicate
MSDS	Material Safety Data Sheets



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NIST	National Institute of Standards and Technology
OSHA	Occupational Safety and Health Administration
QC	Quality Control
RCRA	Resource Conservation and Recovery Act
SWDA	Safe Water Drinking Act

#### 4.0 Interferences

- 4.1 Particulates create an interference, samples may be pre-filtered.
- 4.2 Residual chlorine can produce a negative interference by limiting reduction efficiency. Before analysis, samples should be checked and if required, dechlorinated with sodium thiosulfate.

#### 5.0 Safety

- 5.1 The toxicity or carcinogenicity of each analyte or reagent has not been precisely determined; however, each chemical should be treated as a potential health hazard. Exposure to these chemicals should be reduced to the lowest possible level. It is suggested that the laboratory perform personal hygiene monitoring of each analyst using this method and the results of this be made available to the analyst.
- 5.2 This method does not address safety issues associated with its use. The laboratory is responsible for maintaining a safe work environment and a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of material safety data sheets (MSDS) should be available to all personnel involved in the analyses. Additional information on laboratory safety can be found in References 16.4-16.6.

#### 5.3 List of Raw Materials Safety Classification

Unless otherwise specified all chemicals should be of ACS grade or equivalent

5.3.1 R1 – Systea (1-Reagent) Nitrate Solution (Proprietary)	acidic
Contains:	
5.3.1.1 Hydrochloric acid, HCl (CASRN 7647-01-0)	corrosive
5.3.1.2 N-1-naptylethylenediamine dihydrochloride, (NEDD) C <sub>12</sub> H <sub>14</sub> N <sub>2</sub> •2HCl (CASRN 1465-25-4)	harmful
5.3.1.3 Sulfanilamide, C <sub>6</sub> H <sub>8</sub> N <sub>2</sub> O <sub>2</sub> S (CASRN 63-74-1)	--
5.3.2 Potassium nitrate, KNO <sub>3</sub> (CASRN 7757-79-1)	toxic

#### 6.0 Equipment and Supplies

- 6.1 Equipment
- 6.1.1 EasyChem analyzer
- 6.1.2 Computer for data collection
- 6.1.3 Printer

#### 7.0 Reagents and Standards



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## 7.1 Preparation of Reagents

### 7.1.1 R1 – Systea (1-Reagent) Nitrate Solution (Proprietary)

R1 - 1.0 Liter Size. Part Number E/R1-10

R1 - 0.5 Liter Size. Part Number E/R1-05

Available for purchase from:

Systea Scientific, LLC

900 Jorie Blvd., Suite 35

Oak Brook, IL 60523

Phone: 630 645-0600

Fax: 630 645-0601

Email: [info@easychem.com](mailto:info@easychem.com)

## 7.2 Preparation of Standards

### 7.2.1 Stock Standard, 1000mg/L N-NO<sub>3</sub>

Potassium nitrate

7.218g

DI water

to 1000mL

Dissolve 7.218g of potassium nitrate, in approximately 600mL of DI water. Mix thoroughly and dilute to one liter with DI water.

### 7.2.2 Working Standard Solution A, 100mg/L

1000 mg/L Stock Standard

10mL

DI water

to 100mL

Dilute 10mL of 1000 mg/L Stock Standard A in a 100mL volumetric flask with DI water

### 7.2.3 Working Standard Solution B, 10mg/L

100 mg/L Working Standard Solution A

10mL

DI water

to 100mL

Dilute 10mL of 100 mg/L Working Standard A in a 100mL volumetric flask with DI water

7.2.4 Working calibrants may be prepared to cover the desired range by adding the appropriate amount of working standard solutions (Section 7.2.3) to 100mL volumetric flasks that contain approximately 80mL DI water. Dilute the solutions to 100mL with DI water. Prepare working calibration standards daily. See the Table on page 7 for specific directions.

The following formula can be used to calculate the amount of stock (or working) calibrant to be used.

$$C_1V_1 = C_2V_2$$

**where:**



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**$C_1$  = desired concentration (in mg/L) of working calibrant to be prepared**

**$V_1$  = final volume (in mL) of working calibrant to be prepared (generally 100mL)**

**$C_2$  = concentration (in mg/L) of stock (or working) calibrant**

**$V_2$  = volume (in mL) of stock (or working) calibrant to be used**

Rearranging the equation to solve for  $V_2$  yields:

$$V_2 = \frac{C_1 V_1}{C_2}$$

For example, to prepare a 1.0mg/L working calibrant from a 1000mg/L stock calibrant, use 0.1mL (100 $\mu$ L) of the stock calibrant in 100mL final volume.

$$V_2 = \frac{(1.0\text{mg/L}) (100\text{mL})}{1000\text{mg/L}}$$

$$V_2 = 0.1\text{mL}$$

Add this amount of stock calibrant to the volumetric flask and then dilute to volume.

Standard curves in desired ranges can be derived from the formula above or the table on page 7.

The following table provides the quantity of secondary standard necessary to prepare working standards of the specified concentration.



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Working Calibration Standard Concentration (mg/L) (100mL)	Standard Solution Volume Added to Volumetric Flask		
	Working Standard B Concentration (Section 7.2.3) 10 mg/L	Working Standard A Concentration (Section 7.2.2) 100 mg/L	Stock Standard Concentration (Section 7.2.1) 1000 mg/L
0.0			
0.001	10 µL	1 µL	
0.005	50 µL	5 µL	
0.01	100 µL	10 µL	1 µL
0.05	500 µL	50 µL	5 µL
0.10	1,000 µL	100 µL	10 µL
0.50	5,000 µL	500 µL	50 µL
1.00	10,000 µL	1,000 µL	100 µL
5.00		5,000 µL	500 µL
10.0		10,000 µL	1,000 µL
50.0			5,000 µL

7.3 DI water refers to high quality water, Type I or Type II as defined in ASTM Standards, Part 31, D1193-91.

8.0 Sample Collection, Preservation, and Storage

8.1 Sample preservation and holding time requirements for drinking water samples are as follows:

8.1.1 For nitrate: Chill the sample to 4<sup>0</sup>C and analyze within 48 hours, unless the sample is chlorinated. If the sample is chlorinated, chill the sample to 4<sup>0</sup>C and analyze within 14 days.

8.1.2 For nitrite: Chill the sample to 4<sup>0</sup>C and analyze within 48 hours. Do NOT acid preserve!

8.1.3 For nitrate-nitrite (combined): Acidify to pH<2 with concentrated H<sub>2</sub>SO<sub>4</sub> at the time of collection, and analyze within 28 days.

8.2 For wastewater samples, requirements are nearly identical, except that samples to be analyzed for nitrate only must be analyzed within 48 hours whether or not chlorine is present, and sample to be analyzed for nitrate-nitrite must be acidified to pH<2 with concentrated H<sub>2</sub>SO<sub>4</sub> at the time of collection, chilled to <6<sup>0</sup>C and analyzed within 28 days.



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8.2.1 In all cases, samples should be analyzed as soon as possible after collection. For the analysis of nitrate or nitrite only, samples must be chilled to 6<sup>0</sup>C and analyzed within 48 hours.

## 9.0 Quality Assurance/Quality Control

9.1 Each laboratory that uses this method is required to operate a formal quality assurance program (Reference 16.3). The minimum requirements of this program consist of an initial demonstration of laboratory capability, and the periodic analysis of QCSs and MS/MSDs as a continuing check on performance. If the determined concentrations are not within +/- 10% of the stated values, performance of the determinative step of the method is unacceptable. The source of the problem must be identified and corrected before continuing with on-going analysis.

9.1.1 The analyst shall make an initial demonstration of the ability to generate acceptable precision and accuracy with this method. This ability is established as described in Section 9.2.

9.1.2 In recognition of advances that are occurring in analytical technology, and to allow the analyst to overcome sample matrix interferences, the analyst is permitted certain options to improve performance or lower the costs of measurements. Alternate determinative techniques, such as the substitution of spectroscopic or immuno-assay techniques, and changes that degrade method performance, are not allowed. If an analytical technique other than the techniques specified in this method is used, that technique must have specificity equal to or better than the specificity of the techniques in this method for the analytes of interest.

9.1.2.1 Each time a modification is made to this method, the analyst is required to repeat the procedure in Section 9.2. If the detection limit of the method will be affected by the change, the laboratory is required to demonstrate that the MDL (40 CFR Part 136, Appendix B) is lower than one-third the regulatory compliance level or as low as or lower than that listed in Section 13. If calibration will be affected by the change, the analyst must recalibrate the instrument per Section 10. The laboratory is required to maintain records of modifications made to this method. These records include the information in this subsection, at a minimum.

9.1.2.2.1 The names, titles, addresses, and telephone numbers of the analyst(s) who performed the analyses and modification, and of the quality control officer who witnessed and will verify the analyses and modification.

9.1.2.2.2 A narrative stating the reason(s) for the modification.



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9.1.2.2.3 Results from all quality control (QC) tests comparing the modified method to this method including:

- (a) Calibration (Section 10.3)
- (b) Calibration verification (Section 9.5)
- (c) Initial precision and recovery – IPR (Section 9.2.2)
- (d) Laboratory reagent blank – LRB (Section 9.4)
- (e) Laboratory Control Sample – LCS (Section 9.6)
- (f) Matrix spike and matrix spike duplicate (Section 9.3)

9.1.2.2.4 Data that will allow an independent reviewer to validate each determination by tracing the instrument output (optical density and reading) to the final result. These data are to include:

- (a) Sample numbers and other identifiers
- (b) Analysis dates and times
- (c) Analysis sequence/run chronology
- (d) Sample weight or volume
- (e) Sample volume prior to each cleanup step, if applicable
- (f) Sample volume after each cleanup step, if applicable
- (g) Final sample volume prior to injection (Section 11.0)
- (h) Injection volume (Section 11.0)
- (i) Dilution data, differentiating between dilution of a sample or modified sample
- (j) Instrument and operating conditions
- (k) Other operating conditions (temperature, incubation times, etc.)
- (l) Detector (operating condition, etc.)
- (m) Printer tapes, disks, and other recording of raw data
- (n) Quantization reports, data system outputs, and other data necessary to link raw data to the results reported

9.1.3 Analyses of matrix spike and matrix spike duplicate samples are required to demonstrate method accuracy and precision and to monitor matrix interferences (interferences caused by the sample matrix). The procedure and QC criteria for spiking are described in Section 9.3.

9.1.4 Analyses of blanks are required to demonstrate freedom from contamination and that the compounds of interest and interfering compounds have not been carried over from a previous analysis. The procedures and criteria for analysis of a blank are described in Section 9.4.



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- 9.1.5 The laboratory shall, on an ongoing basis, demonstrate through the analysis of the LCS that the analysis system is in control. This procedure is described in Section 9.6.
- 9.1.6 The laboratory should maintain records to define the quality of data that is generated. Development of accuracy statements is described in Sections 9.3.8 and 9.6.3.
- 9.1.7 Accompanying QC for the determination of nitrate is required per analytical batch. An analytical batch is a set of samples analyzed at the same time, to a maximum of 10 samples. Each analytical batch of 10 or fewer samples must be accompanied by a laboratory blank (Section 9.4), an LCS (Section 9.6), and a matrix spike and matrix spike duplicate (MS/MSD, Section 9.3), resulting in a minimum of five analyses (1 sample, 1 blank, 1 LCS, 1 MS, and 1 MSD) and a maximum of 14 analyses (10 samples, 1 blank, 1 LCS, 1 MS, and 1 MSD) in the batch. If greater than 10 samples are analyzed at one time, the samples must be separated into analytical batches of 10 or fewer samples.

## 9.2 Initial demonstration of laboratory capability

- 9.2.1 Method Detection Limit (MDL)—To establish the ability to detect nitrate at low levels, the analyst shall determine the MDL per the procedure in 40 CFR 136, Appendix B using the apparatus, reagents, and standards that will be used in the practice of this method. An MDL less than or equal to the MDL listed in Section 13.0 must be achieved prior to practice of this method. Calculate the MDL as follows:

$$\text{MDL} = (t) \times (S)$$

Where,  $t$  = Student's  $t$  value for a 99% confidence level and a standard deviation estimate with  $n-1$  degrees of freedom ( $t = 3.143$  for seven replicates),  $S$  = standard deviation of the replicate analysis. MDLs should be determined every 6 months, when a new operator begins work, or whenever there is a significant change in the background or instrument response.

- 9.2.2 Initial Precision and Recovery (IPR)—To establish the ability to generate acceptable precision and accuracy, the analyst shall perform the following operations:

9.2.2.1 Analyze four samples of the LCS according to the procedure beginning in Section 10.0.

9.2.2.2 Using the results of the set of four analyses, compute the average percent recovery ( $X$ ) and the standard deviation of the percent recovery ( $s$ ) for



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nitrate. Use Equation 1 for calculation of the standard deviation of the percent recovery.

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**EQUATION 1**

$$s = \sqrt{\frac{\sum x^2 - (\sum x)^2}{n-1}}$$

where:

*x* = percent recovery in each sample

*n* = number of samples

*s* = standard deviation of the population

- 
- 9.2.3 Compare *s* and *X* with the acceptance criteria. If *s* exceeds the precision limit or *X* falls outside the range for recovery, system performance is unacceptable and the problem must be found and corrected before analyses can begin.
- 9.3 Matrix spike/matrix spike duplicate (MS/MSD)—The laboratory shall spike, in duplicate, a minimum of 10 percent of all samples (one sample in duplicate in each batch of ten samples) from a given sampling site.
- 9.3.1 The concentration of the spike in the sample shall be determined as follows:
- 9.3.1.1 If, as in compliance monitoring, the concentration of nitrate in the sample is being checked against a regulatory concentration limit, the spiking level shall be at that limit or at 1 to 5 times higher than the background concentration of the sample (determined in Section 9.3.2), whichever concentration is higher.
- 9.3.1.2 If the concentration of nitrate in a sample is not being checked against a limit, the spike shall be at the concentration of the LCS or at 1 to 5 times higher than the background concentration, whichever concentration is higher.
- 9.3.2 Analyze one sample aliquot out of each set of ten samples from each site or discharge according to the procedure beginning in Section 11.0 to determine the background concentration (B) of nitrate.
- 9.3.2.1 Spike this sample with the amount of nitrate stock solution (Section 7.2.1) to produce a concentration in the sample of 1 mg/L. If necessary, prepare another stock solution appropriate to produce a level in the



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sample at the regulatory compliance limit or at 1 to 5 times the background concentration (per Section 9.3.1).

9.3.2.2 Spike two additional sample aliquots with the spiking solution and analyze these aliquots to determine the concentration after spiking (A).

9.3.3 Calculate the percent recovery of nitrate in each aliquot using Equation 2.

---

#### EQUATION 2

$$P = \frac{100(A - B)}{T}$$

Where:

*P = Percent recovery*

*A = Measured concentration of nitrate after spiking*

*B = Measured background concentration of nitrate*

*T = True concentration of the spike*

- 
- 9.3.4 Compare the spike recovery percentage to the QC acceptance criteria of 90 to 110%. If spike percent recovery is outside of the acceptance criteria, and the recovery of the LCS in the ongoing precision and recovery test (Section 9.6) for the analytical batch is within the acceptance criteria, an interference is present. In this case, the result may not be reported for regulatory compliance purposes.
- 9.3.5 If the results of both the MS/MSD and the LCS test fail the acceptance criteria, the analytical system is judged to be out of control. In this case, the problem shall be identified and corrected, and the analytical batch reanalyzed.
- 9.3.6 Compute the relative percent difference (RPD) between the two spiked sample results using Equation 3.

---

#### EQUATION 3

$$RPD = \frac{|D_1 - D_2|}{[D_1 + D_2]/2} \times 100$$

where:

*RPD = Relative percent difference*

*D<sub>1</sub> = Concentration of nitrate in the spiked sample*

*D<sub>2</sub> = Concentration of nitrate in the spiked duplicate sample*



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- 9.3.7 The precision of the RPD for the MS/MSD should be less than 20%. If the RPD is greater than the acceptance criteria, the analytical system is judged to be out of control, and the problem must be immediately identified and corrected, and the analytical batch reanalyzed.
- 9.3.8 As part of the QC program for the laboratory, method precision and accuracy for samples should be assessed and records should be maintained. After the analysis of five spiked samples in which the recovery passes the test in Section 9.3.4, compute the average percent recovery ( $P_a$ ) and the standard deviation of the percent recovery ( $s_p$ ). Express the accuracy assessment as a percent recovery interval from  $P_a - 3s_p$  to  $P_a + 3s_p$ . For example, if  $P_a = 100\%$  and  $s_p = 5\%$  for five analyses, the accuracy interval is expressed as 85 – 115%. Update the accuracy assessment on a regular basis (e.g., after each five to ten new accuracy measurements).
- 9.4 Laboratory Reagent Blanks (LRB)—Laboratory reagent water blanks are analyzed to demonstrate freedom from contamination.
- 9.4.1 Analyze a reagent water blank initially and with each analytical batch. The blank must be subjected to the exact same procedural steps as the samples.
- 9.4.2 If nitrate is detected in the blank at a concentration greater than the MDL, analysis of samples is halted until the source of contamination is eliminated and a blank shows no evidence of contamination.
- 9.5 Calibration Verification—Verify calibration of the analytical equipment before and after each analytical batch of 14 or fewer measurements. (The 14 measurements will normally be 10 samples, 1 reagent blank, 1 LCS, 1 MS, and 1 MSD). This can be accomplished by analyzing the mid-range calibration standard and verifying that it is within +/- 10% of the true value. (The concentration of the calibration verification depends on the calibration range being used.) Failure to attain recoveries within the acceptance criteria requires recalibration of the analysis system.
- 9.6 Laboratory Control Sample (LCS)—To demonstrate that the analytical system is in control and acceptable precision and accuracy is being maintained with each analytical batch, the analyst shall perform the following operations.
- 9.6.1 Analyze a LCS with each analytical batch according to the procedure in Section 11.0.



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9.6.2 If the results for the LCS are within +/-10% of the true value, analysis of the batch may continue. If, however, the concentration is not within this range, the analytical process is not in control. In this event, correct the problem, repeat the LCS test, and reanalyze the batch.

9.6.3 The laboratory should add results that pass the specification in Section 9.6.2 to IPR and previous LCS data and update QC charts to form a graphic representation of continued laboratory performance. The laboratory should also develop a statement of laboratory data quality for nitrate by calculating the average percent recovery (R) and the standard deviation of the percent recovery ( $s_r$ ). Express the accuracy as a recovery interval from  $R - 3s_r$  to  $R + 3s_r$ . For example, if  $R = 100\%$  and  $s_r = 5\%$ , the accuracy is 85% to 115%.

9.7 Reference Sample—To demonstrate that the analytical system is in control, the laboratory may wish to periodically test an external reference sample, such as a Standard Reference Material (SRM) available from the National Institutes of Standards and Technology (NIST). Corrective action should be taken if the measured concentration significantly differs from the stated concentration.

## 10.0 Calibration and Standardization

- 10.1 Working Standards should be prepared as required to cover the range of analysis at a minimum of three standards and a blank. If analyzing samples not dissolved in pure DI water, dilute the working standards with sample matrix solution.
- 10.2 Calibration should include a minimum of 4 standards and a blank. The linear correlation coefficient ( $r^2$ ) should be greater than 0.995.
- 10.3 Prepare standard curve by plotting instrument response against concentration readings. A calibration curve may be fitted to the calibration standards response data using the computer. Acceptance or control limits should be established using the difference between the measured reading of the calibration standards and the “true” value concentration.
- 10.4 After the calibration has been accomplished, it must be verified by the analysis of a suitable Quality Control Sample (QCS). If measurement exceeds the +/-10% of the QCS true value the analysis should be terminated and the instrument recalibrated. The new calibration must be verified before continuing analysis. Periodic reanalysis of the CCV is recommended as a continuing calibration check.



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## 11.0 Procedure

### 11.1 Sample analysis

11.1.1 Detailed instructions are explained in the EasyChem operation manual

### 11.2 Following is a summary of the set up procedure

11.2.1 Prepare standards and reagents

11.2.2 Place standards and reagents in EasyChem instrument

11.2.3 Choose appropriate analysis

11.2.4 Set up tray protocol

11.2.5 Analyze samples

### 11.3 Flow Cell

<b>Standard</b>	10 mm pathlength
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### 11.4 User parameter settings

#### 11.4.1 Method Parameters

Range 0.001-0.150mg/L

**Methods list**

Nr.	Name	Code
1	Easy Nitrate (1-Reagent)	NO3
2	Easy Nitrite SM20 4500B-0	NO3 R
3	Easy Nitrate Cadmium Red	CDNO3
4	PO4-SEA-01-2008 L	PO4 L
5	PO4-SEA-01-2008 M	PO4 M
6	NO3 Hydrazine	NO3 H
7	Alkalinity	ALK

**Method**

Method name: **Easy Nitrate (1-Reagent)**  
Method code: **NO3**

**Method type**: Endpoint

**Sample**: Sample volume (µl): **725**

**General setup**

Measuring unit: **ppm**  
Number of decimals: **5**  
Stabilization time (sec): **31**  
Measurement time (sec): **3**

Use net Ods  Show Blank: OD

**Washes**

Number of final washes: **1**  
Wash every:  R1  R2  R3

**O.D. Correction**

Carry over: **0.0000**  
 Blank  Gain

**Reagent 1**

Start position: **1**  
Volume (µl): **225**  
Incubation time (sec): **1105**

**Reagent 2**

Volume (µl): **0**  
Incubation time (sec): **0**

**Reagent 3**

Volume (µl): **0**  
Incubation time (sec): **0**

Reagent prime  CD Reduction

**Filter**: **5-546**

Buttons: Add, Delete, Rename, General, Calibration, Limits and Q.C., Ok, Cancel



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### 11.4.2 Method Parameters Range 0.020-2.0mg/L

**Methods setup**

Nr.	Name	Code
1	Easy Nitrate (1-Reagent)	NO3
2	Easy Nitrite SM20 4500B-0	NO3 R
3	Easy Nitrate Cadmium Redi	CDNO3
4	PO4-SEA-01-2008 L	PO4 L
5	PO4-SEA-01-2008 M	PO4 M
6	NO3 Hydrazine	NO3 H
7	Alkalinity	ALK

**Method**

Method name: **Easy Nitrate (1-Reagent)**  
Method code: **NO3**

Method type: **Endpoint**

Sample: Sample volume (µl) **150**

**General setup**

Measuring unit: **ppm**  
Number of decimals: **3**  
Stabilization time (sec): **31**  
Measurement time (sec): **3**

Use net Ods  Show Blank O.D.

**Reagent 1**

Start position: **1**  
Volume (µl): **450**  
Incubation time (sec): **1105**

**Reagent 2**

Volume (µl): **0**  
Incubation time (sec): **0**

**Reagent 3**

Volume (µl): **0**  
Incubation time (sec): **0**

Reagent prime  CD Reduction

**Washes**

Number of final washes: **1**  
Wash every:  R1  R2  R3

**O.D. Correction**

Carry over: **0.0000**  
 Blank  Gain

**Filter**: **5-546**

Buttons: **Add** **Delete** **Rename** **General** **Calibration** **Limits and Q.C.** **Ok** **Cancel**



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### 11.4.3 Method Parameters Range 0.050-10mg/L

**Methods setup**

Nr.	Name	Code
1	Easy Nitrate (1-Reagent)	NO3
2	Easy Nitrite SM20 4500B-0	NO3 R
3	Easy Nitrate Cadmium Red	CDNO3
4	PO4-SEA-01-2008 L	PO4 L
5	PO4-SEA-01-2008 M	PO4 M
6	NO3 Hydrazine	NO3 H
7	Alkalinity	ALK

**Method**

Method name: **Easy Nitrate (1-Reagent)**  
Method code: **NO3**

**Method type**: Endpoint

**General setup**

Measuring unit: ppm  
Number of decimals: 3  
Stabilization time (sec): 31  
Measurement time (sec): 3

Use net Ods  Show Blank OD

**Washes**

Number of final washes: 1  
Wash every:  R1  R2  R3

**O.D. Correction**

Carry over: 0.0000  
 Blank  Gain

**Sample**

Sample volume (µl): 50

**Reagent 1**

Start position: 1  
Volume (µl): 700  
Incubation time (sec): 1122

**Reagent 2**

Volume (µl): 0  
Incubation time (sec): 0

**Reagent 3**

Volume (µl): 0  
Incubation time (sec): 0

Reagent prime  CD Reduction

**Filter**: 5-546

Buttons: Add, Delete, Rename, General, Calibration, Limits and Q.C., Ok, Cancel



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### 11.4.4 Method Parameters Range 0.25-50mg/L

**Methods setup**

Methods list			Method	
Nr.	Name	Code	Method name	Method code
1	Easy Nitrate (1-Reagent)	NO3	<b>Easy Nitrate (1-Reagent)</b>	NO3
2	Easy Nitrite SM20 4500B-0	NO3 R		
3	Easy Nitrate Cadmium Red	CDNO3		
4	PO4-SEA-01-2008 L	PO4 L		
5	PO4-SEA-01-2008 M	PO4 M		
6	NO3 Hydrazine	NO3 H		
7	Alkalinity	ALK		

  

**Method type**  
Endpoint

**General setup**  
 Measuring unit: ppm  
 Number of decimals: 3  
 Stabilization time (sec): 31  
 Measurement time (sec): 3  
 Use net Ods     Show Blank OD

**Washes**  
 Number of final washes: 1  
 Wash every:  R1    R2    R3

**O.D. Correction**  
 Carry over    0.0000  
 Blank     Gain

**Sample**  
 Sample volume (µl): 10

**Reagent 1**  
 Start position: 1  
 Volume (µl): 940  
 Incubation time (sec): 918

**Reagent 2**  
 Volume (µl): 0  
 Incubation time (sec): 0

**Reagent 3**  
 Volume (µl): 0  
 Incubation time (sec): 0  
 Reagent prime     CD Reduction

**Filter**  
 5-546

Buttons: Add, Delete, Rename, General, Calibration, Limits and Q.C., Ok, Cancel



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## 12.0 Data Analysis and Calculations

12.1 Report results to three significant figures for nitrate and/or nitrite concentrations above the MDL. Do not report results below the MDL unless required by the permitting authority or in the permit.

## 13.0 Method Performance

13.1 Performance data using aqueous standards

Test Range	0.050-10 mg/L N-NO <sub>3</sub>
Sensitivity at 10 mg/l as N	1.8 O.D.
Reagent Absorbance	0.07
Linear Correlation Coefficient	0.999
Recovery ratio 10 ppm N-NO <sub>2</sub> versus 10 ppm N-NO <sub>3</sub>	90-110%
Method Detection Limit (determined according to 40 CFR 136, Appendix B)	0.011mg/L

Note: the above performance specifications were developed with the exclusive use of genuine Systea parts and consumables.

## 14.0 Pollution Prevention

14.1 Standards should be prepared in volumes consistent with laboratory use to minimize the volume of expired standards to be disposed.

## 15.0 Waste Management

15.1 It is the laboratory's responsibility to comply with all federal, state, and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions, and to protect the air, water and land by minimizing and controlling all releases from fume hoods and bench operations. Compliance with all sewage discharge permits and regulations is also required.

15.2 Samples preserved with HCl or H<sub>2</sub>SO<sub>4</sub> to a pH<2 are hazardous and must be neutralized before being disposed, or must be handled as a hazardous waste.

15.3 For further information on waste management, consult "The Waste Management Manual for laboratory Personnel," and "Less is Better: Laboratory Chemical Management for Waste Reduction," both available from the American Chemical Society's Department of Government Relations and Science Policy, 1155 16th Street N.W., Washington DC, 20036.



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## 16.0 References

- 16.1 Methods for the Determination of Inorganic Substances in Environmental Samples – Nitrate 353.2 Rev. 2.0 Determination of Nitrate-Nitrite by Automated Colorimetry, U.S. EPA National Exposure Research Laboratory EPA-600/R93/100 (NTIS PB 94-120821), 1993.
- 16.2 Grasshoff, K., Technicon International Congress.
- 16.3 “Handbook of Analytical Quality Control in Water and Wastewater Laboratories,” USEPA EMSL, Cincinnati, OH EPA-600/4-79-019, March 1979.
- 16.4 “Carcinogens - Working With Carcinogens,” Department of Health, Education, and Welfare, Public Health Service, Center for Disease Control, National Institute for Occupational Safety and Health, Publication No. 77-206, August 1977.
- 16.5 “OSHA Safety and Health Standards, General Industry,” (29 CFR 1910), Occupational Safety and Health Administration, OSHA 2206 (Revised, January 1976).
- 16.6 “Safety in Academic Chemistry Laboratories,” American Chemical Society, Committee on Chemical Safety, 3rd Edition, 1979.
- 17.0 Tables, diagrams, and validation data  
See Sections 7.0 and 13.0
- 18.0 The definition and purposes below are specific to this method, but have been conformed to common usage as much as possible.
- 18.1 **Calibration Blank (CB):** A volume of reagent water in the same matrix as the calibration standards, but without analyte.
- 18.2 **Calibration Standard (CAL):** A solution prepared from the primary dilution standard solution or stock standard solutions. The CAL solutions are used to calibrate the instrument response with respect to analyte concentration.
- 18.3 **Clean Water Act (CWA):** An act to provide for water pollution control in the Public Health Services of the Federal Security Agency and in the Federal Works Agency and for other purposes as specified in the Federal Water Pollution Control Act.
- 18.4 **Chemical Abstracts Service Registry Number (CASRN):** The largest substance identification system in existence. When a chemical substance, newly encountered in the literature, is processed by CAS, its molecular structure diagram, systematic chemical name, molecular formula, and other identifying information are added to the Registry and it is assigned a unique CAS Registry Number.



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**18.5 Comprehensive Environmental Resource Compensation Liability Act (CERCLA):** The 1980 Superfund statute established liability to the U.S. Government for damage to natural resources over which the U.S. has sovereign rights and requires the President to designate Federal officials to act as trustees for natural resources.

**18.6 Deionized Water (DI Water):** The vast majority of dissolved impurities in modern water supplies are ions such as calcium, sodium, chlorides, etc. The deionization process removes ions from water via ion exchange.

**18.7 Environmental Protection Agency (EPA):** The mission of the Environmental Protection Agency is to protect human health and the environment.

**18.8 Instrument Precision Check (IPC):** Also known as a Continuing Calibration Verification (CCV) used to evaluate the performance of the instrument throughout the analysis.

**18.9 Linear Calibration Range (LCR):** The concentration range over, which the instrument response is linear.

**18.10 Laboratory Reagent Blank (LRB):** An aliquot of reagent water or other blank matrices that is digested exactly as a sample including exposure to all glassware, equipment, and reagents that are used with other samples. The LRB is used to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or other apparatus.

**18.11 Method Detection Limit (MDL):** The EPA defines MDL as “the minimum concentration that can be determined with 99% confidence that the true concentration is greater than zero.” This procedure is outlined in 40 CFR 136. Prepare a solution of the analyte that is one to five times the estimated detection. Test this solution seven or more times, then determine the standard deviation of the data set. The method detection limit is calculated according to the formula:  $MDL = \text{Student's } t \text{ value} \times \text{the standard deviation}$ .

**18.12 Matrix Spike (MS):** Also known as a Laboratory Spiked Blank (LSB). An aliquot of reagent water or other blank matrices to which known quantities of the method analytes are added in the laboratory. The MS is analyzed exactly like a sample, and its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements.

**18.13 Matrix Spike Duplicate (MSD):** Also known as a Laboratory Spiked Sample Matrix (LSM). An aliquot of an environmental sample to which known quantities of the method analytes are added in the laboratory. The MSD is analyzed exactly like a sample, and its purpose is to determine whether the sample matrix contributes bias to the analytical results.



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**18.14 Material Safety Data Sheets (MSDS):** Written information provided by vendors concerning a chemical's toxicity, health hazards, physical properties, fire, and reactivity data including storage, spill, and handling precautions.

**18.15 National Institute of Standards and Technology (NIST):** Founded in 1901, NIST is a non-regulatory federal agency within the U.S. Commerce Department's Technology Administration. NIST's mission is to develop and promote measurement, standards, and technology to enhance productivity, facilitate trade, and improve the quality of life.

**18.16 Occupational Safety and Health Administration (OSHA):** OSHA's mission is to assure the safety and health of America's workers by setting and enforcing standards; providing training, outreach, and education; establishing partnerships; and encouraging continual improvement in workplace safety and health.

**18.17 Quality Control Sample (QCS):** Also known as a Laboratory Control Sample (LCS). A solution of method analytes of known concentrations that are used to verify stock standard solutions or spike an aliquot of MS or MSD. The QCS is obtained from a source external to the laboratory and different from the source of the calibration standards. It is used to check laboratory performance with externally prepared test materials.

**18.18 Resource Conservation and Recovery Act (RCRA):** RCRA gave EPA the authority to control hazardous waste from the "cradle-to-grave." This includes the generation, transportation, treatment, storage, and disposal of hazardous waste. RCRA also set forth a framework for the management of non-hazardous wastes.

**18.19 Safe Water Drinking Act (SWDA):** The Office of Ground Water and Drinking Water (OGWDW), together with states, tribes, and its many partners, protects public health by ensuring safe drinking water and protecting ground water. OGWDW, along with EPA's ten regional drinking water programs, oversees implementation of the Safe Drinking Water Act, which is the national law safeguarding tap water in America.

**18.20 Stock Standard Solution (SSS):** A concentrated solution containing one or more method analytes prepared in the laboratory using reference materials or purchased from a reputable commercial source.



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19.0 Consumables used for this method.

P/N	Description	Sales unit	Estimated consumption
CUP-EASY-02	SAMPLE CUPS 2.0 ml	Pk/500	500 tests
E/F25	FILTERS - SAMPLE PROBE CLEANER	Pk/25	1 pk per month
E/R18	REAGENT BOTTLES	Pk/18	1 pk per 12 months
E/RZ24	REACTION CUVETTES	Pk/100	2400 tests
E/T25	STOPPER - REAGENT BOTTLES	Pk/25	1 pk per month
E/K01	TUBE KIT	EA	1 yearly
E/K02	PROBES KIT	EA	1 yearly
E/LA01	SOURCE LAMP ASSEMBLY	EA	1 yearly
E/WT2X4-01	WASTE LINE TUBE	meter	5m per year
E/FC-10	EASYCHEM 10MM FLOWCELL	EA	1 yearly