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EPA Method 353.2- Nitrite in Drinking Water

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1. Scope and Application

1.1. The purpose of this method is to determine nitrite in drinking, ground, surface, domestic and industrial wastewaters. Nitrite is determined by diazotizing with sulfanilamide followed by coupling with N- (1-naphthyl) ethylenediamine dihydrochloride. The resulting water soluble dye has a magenta color, which is read at 520 nm. The applicable range of 0.05 to 5.0 mg N/L as NO_2^- from the published methods has been modified to a range of 0.02 to 2.0 mg N/L as NO_2^- by the instrument manufacturer. This procedure is modified using Lachat QuikChem Method 10-107-04-1-C. Volumes of standards and reagents may be changed, provided the quality control and performance requirements stated in this SOP are met.

1.2. Restricted Procedure:

1.2.1. This procedure is restricted to use by an analyst experienced in the operation of a Lachat Quikchem (FIA) System. Additionally, the analyst must complete the requirements of the GaEPD Initial Demonstration of Analyst Proficiency prior to the analysis of actual samples. Analysts are further warned that performance of this analysis involves the use of potentially hazardous chemicals; refer to the GaEPD Chemical Hygiene Plan (see reference 13.9.) for additional information regarding chemicals required by this method.

2. **Definitions**

2.1. Refer to Chapter 3 of the Georgia EPD Laboratory Quality Assurance Plan (see reference 13.3.) for Quality Control definitions.

2.2. Primary Source (PS) – A standard that is used to make up the calibration points of a curve.

2.3. Second Source (SS) – A standard made from a manufacturer other than

that of the primary source.

2.4. Initial Calibration Verification (ICV) – An ICV is a second source standard that is used to verify the correctness of the primary source calibration curve. The ICV is run at a level equal to that of a Laboratory Control Sample (LCS) or the midpoint on the calibration curve.

2.5. Continuing Calibration Check (CCC) or Continuing Calibration Verification (CCV) – A standard used to verify that the response of the instrument has not changed since initial calibration.

2.6. Calibration Blank (CB), Initial Calibration Verification Blank (ICB), Method Blank(MBLK), Method Detection Limit Blank (MDLB) or Continuing Calibration Blank (CCB) – A volume of reagent water fortified with the same matrix as the calibration standards, but without the analytes.

2.7. MDLS (Method Detection Limit Spike) – MDLB spiked with analytes at the lowest calibration level to be used for the determination of MDL.

3. Interferences

3.1. Low results would be obtained for samples that contain high concentrations of iron, copper or other metals. In this method, EDTA is added to the buffer to reduce this interference.

3.2. Sample turbidity may interfere. Turbidity can be removed by filtration through a 0.45 µm pore diameter membrane filter prior to analysis.(See 5.6.)

4. Safety

4.1. Refer to the EPD Laboratory Safety / Chemical Hygiene Plan & Fire Safety Plan, online revision. Reference 13.9.

5. Apparatus and Equipment

5.1. Sample Container: 250 ml Nalgene bottle

5.2. Plastic culture tubes, O.D. x L: 13mm x 100mm, Fisher Scientific part # 14-956-8E or equivalent or glass culture tubes 12mm x 75 mm VWR part # 60825-502 or equivalent.

5.3. Glassware – Class A volumetric flasks, graduated cylinders and pipettes

5.4. Cadmium-Copper Reduction Column (Lachat Part No. 50237), pre-packed(switched in off position)

5.5. Lachat Quikchem flow injection analysis instrument

5.5.1. Lachat XYZ Autosampler

5.5.2. Auto-sampler racks (90 position)

5.5.3. Reagent pump

5.5.4. Reaction unit or manifold

5.5.5. Colorimetric detector with 520 nm interference filter

5.5.6. Computer with Microsoft Windows operating system with Lachat Omnion software or equivalent

5.6. Syringeless filter device containing 0.45 µm nylon membrane with glass microfiber prefilter (Whatman™ Autovial™ Syringeless Filters: 12mL Capacity, catalog number AV125UNAO or equivalent)

5.7. Air displacement pipettes of various volumes, auto- pipettor, pipette tips in various sizes. Air displacement pipettes and auto-pipettors may also be described as mechanical pipettes.

5.7.1. Each day of use, the volume dispensed by each mechanical pipette must be verified for the specific volume for which the pipette is being used.

5.7.1.1. Mechanical pipette volumes are verified by measuring the weight of a volume of water dispensed by the unit. At room temperature, 1 ml of water is equal to 1 g. Mechanical pipettes must be verified to be within ± 2.5% of the expected weight.

5.7.1.2. Auto-pipettors may be verified by measuring the volume dispensed with a graduated cylinder. The volume dispensed must be within ± 2.5 percent of the nominal volume.

5.7.1.3. Mechanical pipettes must be professionally calibrated every 6 months.

5.8. Balance: analytical, capable of accurately weighing to the nearest 0.0001g.

5.9. Flow cell: 10 mm path length, 80 µl, glass

5.10. Vacuum source for degassing mobile phases

5.11. Disposable transfer pipettes:

5.11.1. Plastic - VWR® Disposable Transfer Pipets PN # 16001-190 or Fisherbrand™ Standard Disposable Transfer Pipettes PN # 13-711-7 M

5.12. 50 ml Centrifuge Tubes: For standards. VWR Part Number 21008-240, or equivalent

6. Reagents

6.1. Reagent Water:

6.1.1. Purified water which does not contain any measurable quantities of target analytes or interfering compounds for each compound of interest (Deionized, HPLC, Milli-Q water, or equivalent. Milli-Q water has a resistivity of 18.2[MΩ·cm]@ 25°C and a TOC of 50 ug/L or less).

6.2. Sulfanilamide Color Reagent:

6.2.1. Add 600 ml of reagent water (see 6.1.) to a 1 L volumetric flask. Add 100 ml of 85% Phosphoric acid (H_3PO_4), 40.0 g Sulfanilamide, and 1.0 g N-(1-naphthyl) Ethylenediamine dihydrochloride (NED). Shake to wet. Stir for 30 minutes to dissolve. Dilute to the mark, and invert to mix. Store the solution in a dark bottle. Degas before use. This solution is

stable for one month. This solution is prepared using Lachat methodology.

6.3. Sodium Hydroxide (15 N):

6.3.1. Only needed if not using commercially prepared buffer (see Ammonium Chloride Buffer, pH 8.5 section 6.4.). Store at room temperature.

6.3.2. Add 150 g NaOH very slowly to 250 ml of reagent water (see 6.1.). CAUTION: The solution will get very hot! Swirl until dissolved. Cool and store in plastic bottle. This solution is stable for one year. This solution is prepared using Lachat methodology.

6.4. Ammonium Chloride Buffer, pH 8.5:

6.4.1. In a 1 L volumetric flask, dissolve 85.0 g ammonium chloride (NH_4Cl) and 1.0 g disodium ethylenediamine-tetraacetic acid dihydrate ($Na_2EDTA{\cdot}2H_2O$) in about 800 ml reagent water (see 6.1.). Dilute to the mark. Invert to mix. Adjust pH to 8.5 with 15N NaOH solution (see 6.3.). Degas before use. Store solution at room temperature for up to one year. This solution is prepared using Lachat methodology.

6.4.2. Commercially prepared buffer may also be purchased and used. This purchased buffer is stable until expiration date on bottle or within 1 year of opening date, whichever is sooner. Store solution at room temperature.

6.5. Carrier Solution/Diluent(Reagent Water):

6.5.1. Purified water which does not contain any measurable quantities of target analytes or interfering compounds for each compound of interest (Deionized, HPLC, Milli-Q water, or equivalent. Milli-Q water has a resistivity of 18.2[MΩ·cm]@ 25ºC and a TOC of 50 ug/L or less).

6.6. Sodium Nitrite($NaNO_2$):Crystalline, ACS grade or equivalent.

6.7. Nitrite Stock Standard A (100 mg N/L as NO_2^-):

6.7.1. Dissolve 0.493 g of $NaNO_2$ in 800 ml of reagent water (see 6.1.). Dilute to one liter with reagent water. Refrigerate. This solution is stable for 3 - 5 days. This solution is prepared using Lachat Methodology.

6.8. Nitrite Stock Standard B (10 mg N/L as NO_2^-):

6.8.1. Pipette 10.0 ml of Nitrite Stock Standard A (see 6.7.) into a 100 ml flask and dilute to volume with reagent water (6.1.). Prepare fresh daily.

6.9. Calibration Standards

6.9.1. This method utilizes an 8-point calibration. The calibration includes a calibration blank.

Table 6.9.1. 1. – Nitrite Calibration Level Concentrations

Analyte	**(mg N/L as NO_2^-)**							
Nitrite	0	0.10	0.40	0.60	0.80	1.00	1.50	2.00

6.9.2. 8 levels of calibration standards are prepared by the addition of aliquots of

PS Nitrite Stock Standard B (10 mg N/L as NO_2^-) (see 6.8.)

Table 6.9.2. 1. –Nitrite Calibration Level Spike Volumes into 100 ml of Reagent Water

	Nitrite Standard Concentrations							
	0 (ICB)	**0.10 mg/L**	**0.40 mg/L**	**0.60 mg/L**	**0.80 mg/L**	**1.00 mg/L**	**1.50 mg/L**	**2.00 mg/L**
ml needed of PS Nitrite Stock Standard B (10 mg N/L as NO_2^-)	NA	1	4	6	8	10	15	20

6.9.3. The CCB, ICB, CCB, MDLB are composed of Reagent water (6.1.) and the CCC is the 1.00 mg N/L as NO_2^- standard.

6.9.4. Calibration standards must be made daily.

6.10. ICV Nitrite Stock Standard A(see 2.4.) or Second Source(SS):

6.10.1. ICV Nitrite Stock Standard A (100 mg N/L as NO_2^-):

6.10.2. The ICV stock standard is typically a standard intended as a "QC Sample" but used as a second source standard instead.

6.10.3. This stock standard must be from a different source than the stock standard used to make the calibration standards.

6.10.4. ICV Nitrite Stock Standard A(100 mg N/L as NO_2^-):

6.10.5. Dissolve 0.493 g of NaNO2 in 800 ml of reagent water (see 6.1.). Dilute to one liter with reagent water. Refrigerate. This solution is stable for 3 - 5 days. This solution is prepared using Lachat methodology.

6.10.6. ICV Nitrite Stock Standard B(10 mg N/L as NO_2^-):

6.10.6.1 Pipette 10 ml of Stock Standard A (100 mg N/L as NO_2^-) (6.10.4) into a 100 ml flask and dilute to volume with reagent water (6.1).

6.10.6.2 The ICV stock solution must be prepared fresh daily.

6.11. *ICV Nitrite Solution (1.00 mg N/L as NO_2^-):*

6.11.1. A 10 ml aliquot of the ICV Nitrite Stock Solution B (10 mg N/L as NO_2^-) (see 6.10.6.) is pipetted into a 100ml volumetric flask and diluted to volume with reagent water (see 6.1.).

6.11.1.1. The ICV solution must be prepared fresh daily.

7. Sample Collection

7.1. Samples are collected in 250 ml plastic bottles.

7.2. Sample preservation is not required.

7.3. Holding time is 48 hours.

7.4. Samples are stored at 0-6° C (Not Frozen).
7.5. The nitrite test code is NO2.

8. Calibration

8.1. Calibration Standards:
8.1.1. The calibration curve consists of the calibration standards and concentrations listed in Table 6.9.1. 1.

8.2. Calibration Curve:
8.2.1. The Lachat Quikchem is calibrated daily. Eight standards are used to construct the Nitrite calibration curve. Minimum acceptable correlation coefficient is 0.995 using a linear regression. Dilute all samples with a response greater than the high standard 2.00 mg N/L.
8.3. Calibration Verification:
8.3.1. An Initial Calibration Verification standard (ICV) (see 2.4.), a Continuing Calibration Check (CCC) (see 2.5. and 6.9.3.) and an Initial Calibration Blank (ICB) (see 2.6. and 6.9.3.) must be analyzed immediately after the calibration standards.
8.3.1.1. The %Drift (see calculation 11.1.) of the ICV from the true value must be within ±10%. Repeat once if it fails. If it fails the second attempt, determine the source of the problem, correct and recalibrate.
8.3.1.2. The ICB, CCB and MBLK values must be less than the method RL or the run will have to be repeated.
8.3.1.3. A CCC and a Continuing Calibration Blank (CCB) must be analyzed every 10 samples and at the end of the sample run and must meet the same criteria as the ICV and ICB respectively.
8.3.1.3.1. The CCC may be from the same source as the calibration standards.
8.3.1.4. If the CCC or CCB do not meet acceptance criteria, then all samples affected by the out of control CCC or CCB are to be rerun.
8.4. A low level calibration check standard at a concentration of 0.10 mg N/L as NO_2^- must be analyzed once per analytical run. Recovery of the standard must be ±50%.
8.5. A MDLS (low level mdl spike) at the concentration of 0.10 mg/L NO_2^- must be analyzed with each batch to perform an ongoing MDL study. All batch QC must be valid to report this result.
8.6. A MDLB(MDL blank) must be analyzed once per analytical batch to perform an ongoing MDL study. All batch QC must be valid to report this result.

9. Quality Control

9.1. Refer to Table 14. 1. for Reporting Limits (RL's), Appendix A, Table A.1

for Quality Control Acceptance Criteria, and Table 14. 2. for Quality Control Procedures associated with this method.

9.2. See reference 13.6. for control charting procedures.

9.3. See reference 13.5. for training and certification procedures.

9.3.1. For Initial Demonstrations of Capability (IDC), Method 353.2 requires a recovery range of 90% - 110% (see calculation 11.8.).

9.3.1.1. The EPD Laboratory sets a 20% RSD requirement for IDC replicates (see calculation 11.4.).

9.3.2. The EPD Laboratory uses the most current accuracy and precision control ranges in use for samples for Continuing Demonstrations of Capability (CDC). If 4 replicates are performed (as opposed to two LCS/LCSD pairs) a 20% RSD is required (see calculation 11.5.).

9.4. Control Limits:

9.4.1. Method 353.2 requires control limits to be adjusted through the use of control charts.

9.4.2. Default control limits for recovery for LCS/LCSD pairs are based on Section 9.3.3 of EPA Method 353.2 (reference 13.1.) as noted in Table 9.4.7. 1. The default limits are 90% - 110% recovery.

9.4.3. The EPD Laboratory sets default LCS/LCSD precision control limits to 0-15% RPD.

9.4.4. Default control limits for recovery for MS/MSD pairs are based on Section 9.4.2 of EPA Method 353.2. The default limits are 90% - 110% recovery. These limits are static by EPA Method/EPD Lab default.

9.4.4.1. Method 353.2 section 9.4.1 requires that 10% of all routine samples must be spiked. See Section 9.5. for batching criteria.

9.4.5. MS/MSD default precision limits are set by the EPD lab as 0 – 15% RPD. These limits are static by EPA Method/EPD Lab default.

9.4.6. In-house limits based on control charts may never exceed the default limits.

9.4.7. See Administrative SOP for Control Charting and Control Limits, reference 13.6. for further details.

Note: The default control limits are presented to assist in defining control limits established with control charts and are not used as batch acceptance criteria.

Table 9.4.7. 1. – Default Quality Assurance Criteria for Method EPA 353.2[1]-Nitrite

QC Type	Analyte	Accuracy(%R) LCL	Accuracy(%R) UCL	Precision (%RPD)
LCS/LCSD	Nitrite	90 – 110		0 – 15

Table 9.4.7. 1. – Default Quality Assurance Criteria for Method EPA 353.2[1]-Nitrite

QC Type	Analyte	Accuracy(%R) LCL	UCL	Precision (%RPD)
MS/MSD	Nitrite	90 – 110		0 – 15

9.5. MDL Study:

9.5.1. MDL (method detection limit) is the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero.

9.5.2. The actual MDL varies depending on instrument and matrix.

9.5.3. The MDL must be determined annually for each instrument prior to results being reported for that instrument. The MDL determined for each compound must be less than the reporting limit for that compound.

9.5.4. The Method Detection Limit Study for all analytes must be performed initially on a new instrument and performed after major instrument repairs or changes to procedures. There are two ways to perform the MDL. The first is with 7 samples and 7 blanks over 3 separate days. The second preferred way the MDL is run as a continuous format.

9.5.5. The 7 MDL samples study is performed by preparing 7 spiked vials, MDLSpike, spiked at the lowest calibration point of the curve, and preparing 7 clean blank vials filled with DI water, MDLBlank. These 7 sets of spiked and blank vial "pairs" are analyzed over 3 separate days, there may or may not be a non-analysis day between each of the 3 days. A total of 14 vials are prepared, 7 spiked and 7 blanks.

9.5.6. A continuous format MDL study is performed where one vial is spiked as an MDLSpike, at the lowest point of the calibration curve and analyzed with every batch of samples along with the method blank vial as an MDLBlank.

9.5.7. The results of the MDLBlank will be entered into Labworks using the Method Blank test code, B_NO2. The MDLSpike result will be entered using the MLNO2. The MDL Spiked Amount will be entered into the test code MANO2. The instrument used for the MDL and Blank analysis will be selected using the test code INSTR-NO2.

9.5.8. MDL study must be performed every six months and before the MDL for the instrument expires.

9.5.9 MDL data is pulled from a two year period.

9.6. Batching:

9.6.1. Batch samples in groups of 20. For each batch, analyze a Matrix Spike

(MS) and a Matrix Spike Duplicate (MSD) for a minimum of 10% of routine samples.

9.6.2. For batches of 1 to 10 routine samples, one MS/MSD pair must be spiked. For batches of 11 to 20 routine samples, two MS/MSD pairs must be spiked using different samples for each pair.

9.6.3. Each batch must have an LCS, LCSD and a Method Blank.

9.7. MDL Studies:

9.7.1. MDL studies must be performed every 6 months (twice annually). See reference 13.8. for further details.

10. Procedure

10.1. Remove sample bottles, standards, and reagents from cold storage and allow equilibration to room temperature prior to sample preparation or analysis.

10.1.1. If using an instrument with an auto-dilutor, refer to Appendix B for the procedure.

10.2. Air displacement and other mechanical pipettors must have the delivery volume verified each day of use for each specific volume for which the device is used on that day.

10.3. Prepare the color reagent, ammonia buffer, and Carrier Solution and make sure the buffer and color reagents have been degassed. (see 6.2, 6.4. and 6.2. 5. respectively).

10.4. From Labworks, print a backlog of pending samples. Samples are batched in groups of 20. For each batch, select one or two QC samples to use as a matrix spike (MS) and matrix spike duplicate (MSD). See batching requirements Section 9.5.

10.5. Turn on the instrument computer, printer, sampler, pump, and colorimetric detector.

10.6. Log into the network and click on the Omnion 3.0 icon. Then log in to the Omnion software.

10.7. Click on the open button to open the folder containing all methods. Open the methods folder and click on the nitrite folder.

10.8. Input the sample list from the batch sheet into the appropriate template. If there are any dilutions make sure to type the dilution in the MDF column. Make sure the MDF box has a checkmark by it. MDF stands for manual dilution factor.

10.9. Select the run pull down menu and click on the Save As option.

10.10. Check pump tubes for wear. Replace if necessary. Secure lines to pump manifold. Make sure the cadmium column is offline. Insert waste lines into properly labeled waste container. If starting an empty waste

container, be sure to update the label with the accumulation start date.

10.11. Press the manual start/stop button on the pump and make sure the pump speed is at 35. Pump DI water through all reagent lines and check for leaks and smooth flow. Let the water run for approximately 5 minutes.

10.12. Switch lines from DI water to reagents and allow system to equilibrate. Record maintenance checks and baseline readings in maintenance log.

10.13. QC Samples and Standard Preparation:

10.13.1. *Initial Calibration Blank (ICB) / Continuing Calibration Blank (CCB/MDLB/MBLK)*:

10.13.1.1. Pour an aliquot of the Reagent Water that was stored in a 250 ml sample collection bottle into a culture tube (see 5.2.) for each ICB, MBLK, MDLB and CCB needed for the instrument sequence. Record the lot# of bottle used.

10.13.2. Pour an aliquot of the 1.00 mg N/L as NO_2^- standard (also the CCC, FCCC) into a culture tube for each CCC needed for the instrument sequence.

10.13.3. Prepare an LCS and LCSD by adding approximately 10 ml of reagent water (6.1.) to a 25 ml volumetric flask for each. Add a 250 µl aliquot of the Nitrite Stock Standard A (100 mg N/L as NO_2^-) (see 6.7.) to each flask and fill to volume with the reagent water that was stored in a 250 ml sample collection bottle (6.1.), cover, and mix. Label each flask appropriately. LCS and LCSD concentrations are 1.00 mg N/L as NO_2^-. Record the lot# of bottle used.

10.13.4. Prepare an MS and MSD by adding approximately 10 ml of the selected QC sample to a 25 ml volumetric flask for each. Add a 250 µl aliquot of the Nitrite Stock Standard A Solution (100 mg N/L as NO_2^-) (see 6.7.) to each flask, fill to volume with the QC sample, cover and mix. Label each flask appropriately. MS and MSD spiked concentrations are 1.00 mg N/L Nitrite over the Nitrite value of the QC Sample.

10.13.5. The MDLS(0.10 mg N/L as NO_2^- standard) must be poured into a 250 ml sample collection bottle before it is poured into a culture tube for analysis. Record the lot# of bottle used.

10.14. Analysis:

10.14.1. Load samples/standards into the auto-sampler. If samples need to be filtered, the method blank, LCS, LCSD in that batch must be filtered to ensure filtration does not negatively affect results. Initial calibration standards are loaded in order from high to low in the 50 ml centrifuge tubes in the standard rack.

10.14.2. Place the reagent line in the color reagent, the buffer line in the ammonia

buffer and the carrier line in Carrier Solution (Reagent Water) See 6.1.

10.14.3. Let the reagents run through the lines for approximately 5 minutes.

10.14.4. Click the start button located above the run worksheet to start the run.

10.14.5. After the run is finished, select the tools pull down menu and click on custom report. Click on the custom report format and select layout. Once the report is created, the auto fields should contain the name of the individual that is logged in, the current date and the page numbers of the report.

10.14.6. Click Apply once, when all layout modifications have been made. If there are no layout modifications, click ok. Exit the custom report menu. Click on the printer button to print the report. The report should contain the calibration curve.

10.14.7. From the main menu, select the run button and click on export worksheet to print the run log.

10.14.8. *Dilutions*:

10.14.8.1. If the response of any sample or QC sample is greater than the high standard 2.0 mg N/L, those samples must be diluted and rerun in a valid sequence or at the end of the run, followed by an ending CCC and CCB. Dilution ratios should be determined, as nearly as possible, so that the response is near the mid-point of the calibration range.

10.14.8.2. To prepare dilutions, use Reagent water (see 6.1.) to dilute the samples.

10.14.8.3. If using the auto-dilutor to prepare dilutions, see Appendix B for instructions.

10.15. Volumes and amounts of reagents, chemicals, and standards may be altered as long as the final concentrations remain the same. Sample volumes and injection amounts may be altered as long as required detection limits can be met and sample/reagent ratios remain the same.

10.16. Shutdown Procedure:

10.16.1. Place the color reagent and buffer lines into reagent water for 5 minutes and then place all lines in a dry beaker for 5 minutes.

10.16.2. Exit the Omnion program and shut down the computer, sampler, pump and colorimetric detector.

10.16.3. Be sure to loosen the platens on the pump tubes so that there isn't excessive wear.

10.16.4. Cap reagent waste container when not in use or neutralize waste as noted in the Laboratory Waste Management SOP, Reference 13.7.)

11. Calculations

11.1. The weighted external standard calibration is calculated by the instrument software and is documented in the instrument software.

11.2. Mean ($\overline{X}$):

$$\overline{X} = \frac{X_1 + X_2 + \cdots X_n}{n}$$

11.2.1. Where:

$X_1 + X_2 + \cdots X_n$ = The sum of a set of values X_i, i = 1 to n

n = The number of values in the set

11.3. Standard Deviation (n – 1) (σ_{n-1}):

$$\sigma_{n-1} = \sqrt{\sum_{i=1}^{n} \frac{(X_i - \overline{X})^2}{n-1}}$$

11.3.1. Where:

$\overline{X}$ = Mean of the values

X_i = Individual values 1 through i

n = Number of values

11.4. Percent Relative Standard Deviation (%RSD):

$$\%RSD = \frac{\sigma_{n-1}}{\overline{X}} * 100$$

11.4.1. Where:

σ_{n-1} = Sample Standard Deviation

$\overline{X}$ = Mean of the values

11.5. Relative Percent Difference (%RPD or RPD):

$$\%RPD = \frac{|X_1 - X_2|}{\frac{(X_1 + X_2)}{2}} * 100$$

11.5.1. Where:

$|X_1 - X_2|$ = Absolute difference between two values

$\frac{(X_1 + X_2)}{2}$ = Average of two values

11.6. Percent Drift, %Drift:

$$\%Drift = \frac{(Concentration_{Calculated} - Concentration_{Expected})}{Concentration_{Expected}} * 100$$

11.6.1. Where:

$Concentration_{Calculated}$ = Concentration calculated from result

$Concentration_{Expected}$ = Theoretical concentration of the standard

11.7. Extract Concentration:

11.7.1. The extract concentration is calculated relative to the calibration curve by the instrument software.

11.8. Percent Recovery:

11.8.1. *LCS/LCSD*:

$$\%\text{Recovery} = \frac{\text{Conc}_{\text{spiked}}}{\text{Conc}_{\text{expected}}} * 100$$

11.8.1.1. Where:

$\text{Conc}_{\text{spiked}}$ = Concentration found in the spiked sample

$\text{Conc}_{\text{expected}}$ = Expected concentration

11.8.2. *MS/MSD*:

$$\%\text{Recovery} = \frac{\text{Conc}_{\text{spiked}} - \text{Conc}_{\text{unspiked}}}{\text{Conc}_{\text{expected}}} * 100$$

11.8.2.1. Where:

$\text{Conc}_{\text{spiked}}$ = Concentration found in the spiked sample

$\text{Conc}_{\text{unspiked}}$ = Concentration found in unspiked sample

$\text{Conc}_{\text{expected}}$ = Expected concentration

11.9. Calculation of Dilution Factors

$$C \times D = F$$

11.9.1. Where:

C = concentration from instrument in mg N/L

D = dilution factor, if any

F = final concentration in mg N/L

12. Waste Management

12.1. See GA EPD Laboratory SOP-EPD Laboratory Waste Management Standard Operating procedures, reference 13.7.

13. References

13.1. EPA Method 353.2, Determination of Nitrate-Nitrite Nitrogen by Automated Colorimetry, Rev. 2.0, August 1993

13.2. Manual for the Certification of Laboratories Analyzing Drinking Water, EPA/815-R-05-004, January 2005.
13.3. EPD Laboratory Quality Assurance Plan, online revision.
13.4. Determination of Nitrate/Nitrite in Surface and Wastewaters by Flow Injection Analysis, Lachat QuikChem Method 10-107-04-1-C, August 28, 2000.
13.5. GA EPD Laboratory SOP's – Initial Demonstration of Capability SOP 6-001, online revision or Continuing Demonstration of Capability SOP 6-002, online revision.
13.6. GA EPD Laboratory SOP-EPD Laboratory Procedures for Control Charts and Control Limits SOP, SOP 6-025, online revision.
13.7. GA EPD Laboratory SOP-EPD Laboratory Waste Management SOP, SOP 6-015, online revision.
13.8. GA EPD Laboratory SOP – Determination of Method Detection Limit, Method Detection Limit SOP 6-007, online revision.
13.9. GA EPD Laboratory Safety Plan – EPD Laboratory Safety / Chemical Hygiene Plan & Fire Safety Plan, online revision.

14. Reporting Limits (RL's), Precision and Accuracy Criteria, and Quality Control Approach

Table 14.1. – Reporting Limits for EPA 353.2 – Nitrite

Parameter/Method	Analyte	Matrix (aqueous) Drinking Water (DW)	
		RL	Unit
EPA 353.2	Nitrite	0.20	mg N/L

Table 14. 2.- **Summary of Calibration and QC Procedures for Method EPA 352**

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria
EPA 353.2	Nitrite	Initial calibration for all analytes	Initial calibration prior to sample analysis	Correlation coefficient $\geq$ 0.995 linear regression	Correct problem then repeat initial calibration	

Table 14. 2.- **Summary of Calibration and QC Procedures for Method EPA 352**

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria
EPA 353.2	Nitrite	Second source calibration verification (ICV)	Once per initial calibration or quarterly, whichever is sooner	Nitrite concentration within 10% of expected value	Correct problem then repeat initial calibration	
		Initial Calibration Blank (ICB)	Once per initial calibration	Nitrite value must be below reporting limit	Correct problem then repeat initial calibration	
		Initial Demonstration: Demonstrate ability to generate acceptable accuracy and precision using four analysis of a QC check sample, a method blank, a blind sample, and an MDL. In addition, the analyst must prepare one standard.	Once per analyst	Default QC Acceptance Criteria SOP 3-033 Appendix A and Initial demonstration SOP	Recalculate results: locate and fix problem with system and then rerun demonstration for those analytes that did not meet criteria	
		Continuing Demonstration: Demonstrate ability to generate acceptable accuracy and precision using a variety of analysis options of a QC sample(s)	Every 6 months	QC Acceptance Criteria SOP 3-033Appendix A and Continuing demonstration SOP	Recalculate results: locate and fix problem with system and then rerun demonstration for those analytes that did not meet criteria	
		Method Blank	One per batch	Nitrite value must be below reporting limit.	Correct problem then analyze method blank and all samples processed with the contaminated blank	If unable to reanalyze, flag with a “B”
		Laboratory Control Sample (LCS/ LCSD)	One LCS/LCSD per analytical batch	QC Acceptance Criteria Table SOP 3-033 Appendix A	Correct problem then reanalyze the LCS/LCSD and all samples in the affected batch	If unable to reanalyze, flag with a “J”
		Matrix Spike (MS/MSD)	10% of Samples	QC Acceptance Criteria Table SOP 3-033 Appendix A	Evaluate out of control event, reanalyze or flag data	
		Continuing Calibration Check (CCC)	Prior to sample analysis, after every 10 samples and at the end of a sample run	Nitrite concentration within 10% of expected value	Correct problem then reanalyze all samples associated with out of control CCC.	

Table 14. 2.- **Summary of Calibration and QC Procedures for Method EPA 352**

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria
EPA 353.2	Nitrite	Continuing Calibration Blank (CCB)	After every 10 samples and at the end of a sample run	Nitrite concentration must be below reporting limit	Correct problem then reanalyze all samples associated with out of control CCB.	
		Low level standard (0.10 mg N/L as NO_2^-)	Once per analytical run	Value must be ± 50% of expected value	Evaluate recovery exceedances, reanalyze or recalibrate	
		MDL Low level Spike (MDLS) (0.10 mg N/L as NO2-)	Once per analytical batch	All batch QC must be valid	Correct problem then reanalyze the affected batch	
		MDL Blank (MDLB) *Can be combined with MBLK	Once per analytical batch	All batch QC must be valid	Correct problem then reanalyze the affected batch	
		MDL study	Every six months or after major maintenance of the instrument	All Spiked MDLs must have a value greater than 0. Minimum Detection Limits established shall be < the RLs in Table 14.1	Re-do MDL Study	
		MDL analysis	Once per batch or as needed to acquire data points per SOP 6-007, online revision	All Spiked MDLs must have a value greater than 0. All other QC in the MDL blank and MDL sample (i.e. Surrogate Spike or Internal Standard, etc. if included) must meet established criteria	Correct problem and re-run the MDL sample or MDL blank once and initiate a corrective action. If the re-run fails a second time, do not use MDL data. Update corrective action, and use associated sample data	

Appendix A – for EPA 353.2 – Nitrite in Drinking Water

Table A.1 Quality Assurance Criteria for Method EPA 353.2			
QC Type	Analyte	Accuracy(%R) LCL UCL	Precision (%RPD)
LCS/LCSD	Nitrite	90 - 110	15
MS/MSD	Nitrite	90 - 110*	15*
*MS/MSD Control limits are static by EPA Method/EPD Lab default. Control chart data generated from 01/01/2018 – 01/01/2021			

Updates to Previous Version:
Section 9
Table A.1
Table 14.2