DISCRETE ANALYZER CHEMISTRY

Ammonia (NH_3) Method

Compatible with SEAL Methods

- SEAL Method EPA-148, Range: 0.01 – 1.0 mg N/L
- SEAL Method EPA-150, Range: 0.1 – 5.0 mg N/L
- SEAL Method EPA-153, Range: 0.2 – 10.0 mg N/L

Methods Referenced

- ISO 15923-1 – "Water quality — Determination of selected parameters by discrete analysis systems — Part 1: Ammonium, nitrate, nitrite, chloride, orthophosphate, sulfate and silicate with photometric detection"
- ISO 7150/1 – "Water quality – Determination of ammonium – Part 1: Manual spectrometric method"
- EPA Method 350.1
- Standard Methods 4500-NH_3 F

Reagent Composition

Two Reagents are used for the NH_3 method:

Reagent 1: **SEALAnalyticalAQ NH3 Salicylate**, contains the following in DI water:

130 g/L Sodium salicylate

130 g/L Sodium citrate dihydrate

1.0 g/L Sodium nitroferricyanide dihydrate
AKA Sodium nitroprusside dihydrate

Reagent 2: **SEALAnalyticalAQ NH3 DCI**, contains the following in DI Water:

37.5 g/L Sodium hydroxide

1.5 g/L Sodium dichloroisocyanurate anhydrous

Or

1.745 g/L Sodium dichloroisocyanurate dihydrate

NOTE: *We recommend using ICNNH41 to make your standards for this method. The solution is certified as the Nitrogen concentration in Ammonium, so no conversions are necessary — just dilute to your working concentrations and start testing*!

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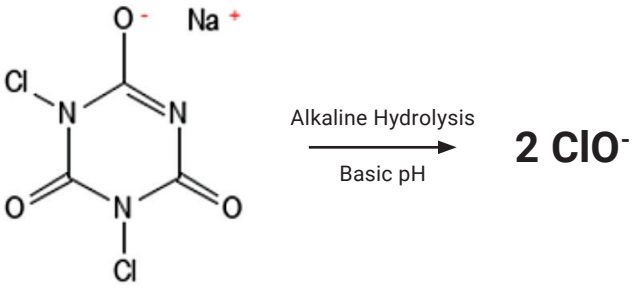
DISCRETE ANALYZER CHEMISTRY

Ammonia (NH_3) Method

Chemical Reactions

Ammonium reacts with hypochlorite, formed by alkaline hydrolysis of sodium dichloroisocyanurate, and with salicylate at a pH of at least 12.6 in the presence of sodium nitroprusside as a catalyst. This produces a compound with a blue color (Indophenol). Citrate is used as a chelating reagent to eliminate interferences by cation such as calcium and magnesium. This method is is a common, safer variation to the to the chemistry of Berthelot's reagent.

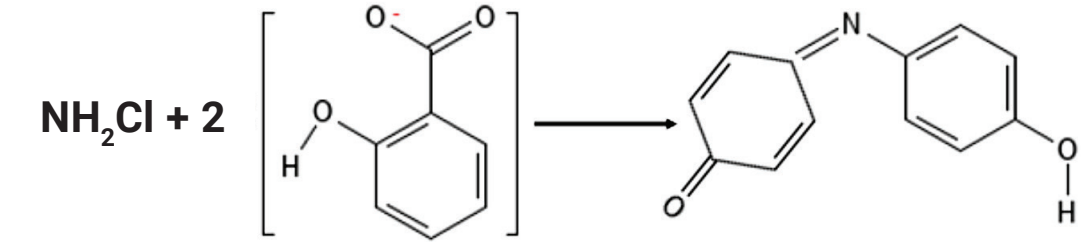
Step 1: Hypochlorite formed by alkaline hydrolysis of dichloroisocyanurate



Step 2: Ammonia reacts with hypochlorite to form chloramine

$$ClO^- + NH_3 \longrightarrow NH_2Cl$$

Step 3: Chloramine reacts with salicylate to form indophenol



Note: Step 3 requires the loss of 2(CO_2) by decarboxylation.

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DISCRETE ANALYZER CHEMISTRY

Chloride (Cl^-) Method

Compatible with SEAL Methods

- SEAL Method EPA-105, Range: 2.0 – 100 mg Cl/L
- SEAL Method EPA-124, Range: 5.0 – 200 mg Cl/L

Methods Referenced

- Standard Methods 4500-Cl^- E — *Automated Ferricyanide Method*
- EPA Method 325.2 — *Chloride by Automated Colorimetry* AKA *Colorimetric, Automated Ferricyanide AAII*

Reagent Composition

Final reagent is made from the following sub-solutions:

1. Ferric nitrate, $Fe(NO_3)_3$
2. Mercuric thiocyanate, $Hg(SCN)_2$

Sub-solutions are combined to create the only reagent used for the Cl^- method.

Final concentrations are as follows:

SEALAnalyticalAQ Chloride Rgt

- 0.6255 g/L Mercury (II) thiocyanate
- 30.3 g/L Iron (III) nitrate nonahydrate

  Or
- 1.974 mmol Hg^{+2}
- 3.943 mmol $(SCN)^-$
- 75 mmol Fe+3

Note: *We recommend using ICCL1 to make your standards check solutions for this method.*

Chemical Reactions

$$Fe^{+3} + Hg(SCN)_2 + \text{Free } Cl^- \rightarrow HgCl_2 + Fe^{+3} + 2SCN^- \rightarrow HgCl_2 + Fe(SCN)_3$$

The thiocyanate ion (SCN^-) is liberated from mercuric thiocyanate through sequestration of mercury by chloride ion to form un-ionized mercuric chloride in the presence of ferric ion, the liberated SCN^- forms highly colored ferric thiocyanate in concentration proportional to the original chloride concentration.

Balanced Reaction

$$12Cl^- + 6Hg(SCN)_2 + 4Fe^{+3} \rightarrow 6HgCl_2 + 4Fe(SCN)_3$$

Note: Calculations using the limiting factor of Hg^{+2} (by moles) show that this reagent can react with a maximum of ~140 mg/L free chloride.

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DISCRETE ANALYZER CHEMISTRY

o-Phosphate (PO_4^{-3}) Method

Compatible with SEAL Methods

- SEAL Method EPA-118, Range: 0.005 – 1.0 mg P/L

Methods Referenced

- Standard Methods 4500-PO_4^{-3} E — *Automated Ascorbic Acid Reduction Method*
- EPA Method 365.1 Rev 2.0 — *Colorimetric, Automated, Ascorbic Acid*

Reagent Composition

Two reagents are used for the PO_4^{-3} method:

- Reagent 1, **SEALAnalyticalAQ PO4 Ascorbic** is 15 g/L Ascorbic acid in DI water.
- Reagent 2, **SEALAnalyticalAQ PO4 Color Rgt** is made from two separate sub-solutions combined in dilute sulfuric acid:
  - Sub-solution 1: 3 g/L Potasssium antimonyl tartrate in DI water
  - Sub-solution 2: 40 g/L Ammonium molybdate in DI water

Final Concentrations are as follows:

SEALAnalyticalAQ PO4 Ascorbic

15 g/L Ascorbic acid

SEALAnalyticalAQ PO4 Color Rgt

0.225 g/L Potassium antimonyl tartrate
8.8 g/L Ammonium molybdate
3.25 N Sulfuric acid

Chemical Reactions

Ammonium molybdate and antimony potassium tartrate react in an acid medium with dilute solutions of phosphorus to form an antimony-phospho-molybdate complex. This complex is reduced to an intensely blue-colored complex by ascorbic acid. The color is proportional to the phosphorus concentration.

Note: The stoichiometry of these reaction is still in question, but the paper, *The molybdenum blue reaction for the determination of orthophosphate revisited: Opening the black box* by EA Nagul, goes into more detail about the formation of the blue color used for this analysis. This paper gives the following equations and mentions that the presence of antimony speeds up the formation of the blue color.

$$PO_4^{-3} + 12MoO_4^{-2} + 27H^+ \rightarrow H_3PO_4(MoO_3)_{12} + 12H_2O$$

$$H_3PMo(VI)_{12}O_{40} + \text{Reductant} \rightarrow [H_4PMo(VI)_8Mo(V)_4O_{40}]^{-3}$$

Note: We recommend using ICPPO41 to make your standards for this method. The solution is certified as the Phosphorous concentration, so no conversions are necessary — just dilute to your working concentrations and start testing!

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DISCRETE ANALYZER CHEMISTRY

Sulfate (SO_4^{-2}) Method

Compatible with SEAL Methods

- SEAL Method EPA-165, Range: 2.0 – 40 mg SO4/L

Methods Referenced

- EPA Method 375.4 — *Sulfate, Turbidimetric*
- Standard Methods 4500-SO_4^{-2} E — *Turbidimetric Method*
- ASTM D516-11 — *Standard Test Method for Sulfate Ion in Water*

Reagent Composition

Only one reagent is used for the SO_4^{-2} method. The **SEALAnalyticalAQ Sulfate Rgt** is made in trace HCl and final concentrations are as follows:

SEALAnalyticalAQ Sulfate Rgt

- 10 g/L Barium chloride dihydrate **or** 48 mmol Ba^{+2}
- 20 g/L Sodium chloride
- 0.5 g/L gelatin

Chemical Reactions

Ba^{+2} + Free SO_4^{-2} → $BaSO_4$

Sulfate ion is converted to a barium suspension under controlled conditions. The resulting turbidity is determined photometrically and compared to a curve prepared from standard sulfate solutions.

Note: Calculations using the limiting factor of Ba^{+2} (by moles) show that this reagent can react with a maximum of ~4600 mg/L free sulfate. However, method range is much lower due to instrument limitations.

Note: *We recommend utilizing ICSO41 to manufacture your standards and check solutions for this method. The SEAL method calls for a Sodium Sulfate starting material, though, we have not noticed any difficulties associated with using the Potassium Sulfate starting material.*

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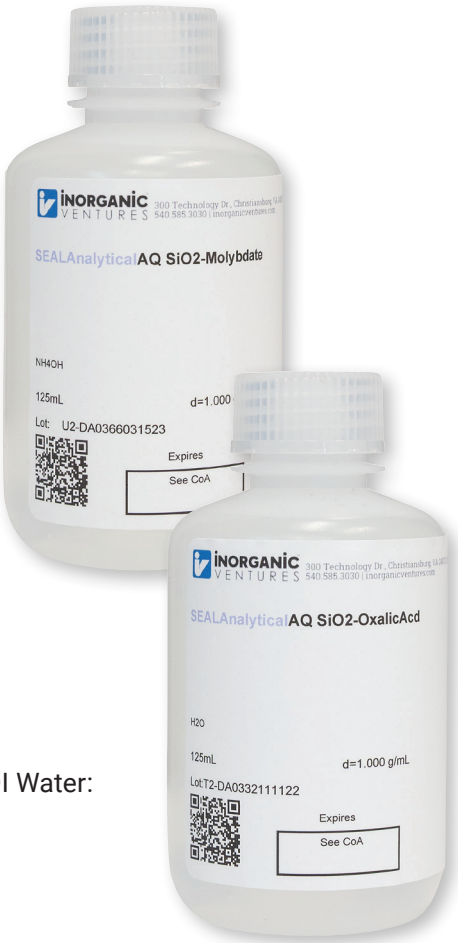
DISCRETE ANALYZER CHEMISTRY

Silica (SiO_2) Method

Compatible with SEAL Methods

- SEAL Method EPA-121, Range: 0.25 – 25 mg Silica (SiO2)/L

We recommend CGSIO1 or CGSIONA1 to make your standard for this method! Both solutions are made from a Silica starting material. The only difference is the matrix. CGSIO1 has a matrix of 1% v/v Nitric Acid /tr Hydrofluoric Acid, while CGSIONA1 has a matrix of 0.2% wt/v Sodium Hydroxide. Diluting either of these standards in DI H2O to achieve your working concentrations for this method will result in either of the matrix options being less than 0.03% of your final solution.



Methods Referenced

- SEAL Analytical AQ400 Method EPA-121-C Rev. 2A
- EPA Method 370.1 — (Colorimetric)
- Standard Methods 4500-SiO2 C — "Molybdosilicate Method"

This method covers the determination of Silica in drinking, surface, and saline waters, and domestic and industrial wastes.

Reagent Composition

Two Reagents are used for the SiO_2 method:

Reagent 1: **SEALAnalyticalAQ SiO2-Molybdate** — Ammonium Molybdate Reagent in DI Water:

100 g/L Ammonium molybdate (VI) tetrahydrate

Trace Ammonium hydroxide for pH adjustment

Reagent 2: **SEALAnalyticalAQ SiO2-OxalicAcd** – Oxalic Acid Reagent in DI Water:

100 g/L Oxalic acid dihydrate

Reagent 3: 10% v/v HCl

Note: Inorganic Ventures and Seal Analytical don't offer a 10% HCl reagent for the 25 ppm Silica method because it's much more cost-effective for you to prepare it in-house. Making this reagent is simple (combine acid and water in the appropriate amounts), and there will be little room for error when following the standard procedure. Plus, it doesn't have any stability concerns, so you can be confident in its performance. By mixing it yourself, you save on costs, make volumes that make sense for your lab, and still get accurate and reliable results, when used in combination with the pre-made reagents above.

Maintenance Tip: After Silica analyses are complete for the day it is recommended to run an alkaline EDTA solution through the instrumentation to remove any reagent deposits and prevent contamination of subsequent samples. Prepare this solution by dissolving 5g of disodium EDTA dihydrate and 10g of Sodium Hydroxide pellets in 500 mL DI H2O.

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DISCRETE ANALYZER CHEMISTRY

Nitrate/Nitrite (Cadmium Coil) Method

Compatible with SEAL Methods

- SEAL Method EPA-114, Range: 0.25 – 15 mg N/L
- SEAL Method EPA-126, Range: 0.04 – 5 mg N/L

  We recommend ICNNO31 and ICNNO21 to make your standards and reduction efficiency check solutions for this method. These solutions are already certified as Nitrate or Nitrite as Nitrogen, so no conversions are needed, just simple dilution factors to get these diluted to your working concentrations.
- SEAL Method EPA-127, Range: 0.012 – 2 mg N/L

Methods Referenced

- SEAL Analytical AQ400 Method EPA-126-C Rev. 2
- EPA Method 353.2 Rev 2.0
- Standard Methods 4500-NO_3^- F
  This method covers the determination of Nitrate-N and Nitrite-N in sewage and effluents, raw and finished drinking waters, and industrial wastes.

Reagent Composition

Two Reagents are used for the Nitrate+Nitrite Cadmium Coil method:

Reagent 1: **SEALAnalyticalAQ NOx Cad-Buffer** – Working Buffer Reagent in DI Water:

179.1 g/L Ammonium Chloride

1.99 g/L EDTA Disodium Salt Dihydrate

0.3731 g/L Triton X-100

Ammonium Hydroxide for pH adjustment

Reagent 2: **SEALAnalyticalAQ NOx Cad-Color** – Sulfanilamide-NEDD Reagent in DI Water:

2 g/L NaOH

15 g/L Sulfanilamide

0.75 g/L N-(1-naphthyl)-ethylenediamine dihydrochloride

4% v/v Phosphoric Acid

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DISCRETE ANALYZER CHEMISTRY

Nitrate/Nitrite (Cadmium Coil) Method

Method Description

Before conducting analyses using this method, confirm that the field preservation technique does not require matrix matching. To do so, combine 5 mL of the field-preserved sample with an equal volume of working buffer (**SEALAnalyticalAQ NOx Cad-Buffer**), and repeat this procedure using the calibration standard. The pH of the two resulting samples should be within 0.25 pH units of each other and should fall within the range of pH 8.2 to 8.6.

The sample is first mixed with pH buffer (**SEALAnalyticalAQ NOx Cad-Buffer**) and then transferred to a copperized cadmium coil, where nitrate is chemically reduced to nitrite. The resulting solution is subsequently mixed with color reagent (**SEALAnalyticalAQ NOx Cad-Color**). Both original nitrite and chemically reduced nitrite react with sulfanilamide, in dilute phosphoric acid, to form a diazonium compound. The reaction between the diazonium ion and NEDD results in the formation of an AZO bond (-C=N-) between the benzene ring and the naphthyl group, which is a reddish-purple AZO dye. The absorbance of this dye is measured photometrically at 520 nm.

For nitrite-only measurements, omit the use of the cadmium coil to prevent the reduction of nitrate to nitrite.

Note: Ensure that the cadmium column or coil used for this method is functioning correctly. It is recommended to perform a reduction efficiency check following any maintenance of the coil or column to verify its optimal performance prior to conducting nitrate/nitrite analysis.

Reduction of Nitrate to Nitrite (via cadmium coil):

$$NO_3^- + 2e^- + 2H^+ \rightarrow NO_2^- + H_2O$$

(Nitrate is reduced to nitrite in the presence of cadmium and acid.)

Reaction of Nitrite with Sulfanilamide:

$$NO_2^- + C_6H_4NH_2SO_3H \rightarrow \text{Diazonium compound}$$

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DISCRETE ANALYZER CHEMISTRY

Nitrate/Nitrite (Hydrazine Reduction) Method

Compatible with SEAL Methods

- SEAL Method EPA-141, Range: 0.02 – 1.5 mg N/L

  We recommend ICNNO31 and ICNNO21 to make your standards and reduction efficiency check solutions for this method. These solutions are already certified as Nitrate or Nitrite as Nitrogen, so no conversions are needed, just simple dilution factors to get these diluted to your working concentrations.

Methods Referenced

- EPA Method 353.1 Rev 2.0
- Standard Methods 4500-NO_3^- H
  This method covers the determination of Nitrate-N and Nitrite-N in sewage and effluents, raw and finished drinking waters, and industrial wastes.

Reagent Composition

Three Reagents are used for the Nitrate/Nitrite (Hydrazine Reduction) method:

Reagent 1: **SEALAnalyticalAQ NOx Hyd-Buffer** – Alkaline Buffer Reagent in DI Water:

46 g/L Sodium Hydroxide

25 g/L Sodium phosphate dibasic

Reagent 2: **SEALAnalyticalAQ NOx Hydrazine** – Working Hydrazine Reagent in DI Water:

1.875 g/L Hydrazine Sulfate

0.0125 g/L Copper (II) Sulfate pentahydrate

0.2 g/L Zinc (II) Sulfate heptahydrate

Reagent 3: **SEALAnalyticalAQ NOx Hyd-Color** – Sulfanilamide-NEDD Reagent in DI Water

4% v/v Phosphoric Acid

6 g/L Sulfanilamide

0.3 g/L N-(1-naphthyl)-ethylenediamine dihydrochloride

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DISCRETE ANALYZER CHEMISTRY

Nitrate/Nitrite (Hydrazine Reduction) Method

Method Description

The sample is held in an alkaline solution of hydrazine sulfate (**SEALAnalyticalAQ NOx Hydrazine** & **SEALAnalyticalAQ NOx Hyd-Buffer**) with Cu (II) present for catalysis, which reduces nitrate in solution to nitrite. Both original nitrite and chemically reduced nitrite react with sulfanilamide, in dilute phosphoric acid, to form a diazonium compound. The reaction between the diazonium ion and NEDD results in the formation of an AZO bond (-C=N-) between the benzene ring and the naphthyl group, which is a reddish-purple AZO dye. The absorbance of this dye is measured photometrically at 520 nm.

Note: It is essential to precisely adjust the volume of **SEALAnalyticalAQ NOx Hydrazine** used to ensure that equimolar Nitrate and Nitrite standards yield consistent results. An excess of hydrazine will lead to the reduction of Nitrite to nitrogen gas (N_2), while insufficient hydrazine will fail to completely reduce all Nitrate (NO_3^-) to Nitrite (NO_2^-). The required hydrazine concentration is influenced by several factors, including temperature, reaction time, pH, and metal ion concentration. Variations of ±25% from the original method are not uncommon.

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COMING SOON!

DISCRETE ANALYZER CHEMISTRY

Total Hardness Method

Compatible with SEAL Methods

- SEAL Method EPA-106, Range: 25 – 400 mg CaCO3/L

  We recommend ICCA1 to make your standard for this method. This solution is made from CaCO3 starting material and is compatible with this method. A simple conversion from Ca to CaCO3 is all that is needed — divide the standard concentration by the weight fraction of Ca/CaCO3 (0.4004) — then you can dilute this standard to your required working concentrations.

Note: When using the SEAL Method, manual construction of the calibration curve is required, as the small sample volume is insufficient for the instrument to generate this curve automatically.

Methods Referenced

- EPA Method 130.1 — (Colorimetric, Automated EDTA)
- Standard Methods 2340 C — "EDTA Titrimetric Method"
  This method covers the determination of Total Hardness in drinking, surface, and saline waters.

Reagent Composition

Two Reagents are used for the Total Hardness method:

Reagent 1: **SEALAnalyticalAQ Hard-Buffer** — Mg-EDTA Buffer in DI water:

5 g/L EDTA disodium magnesium salt hydrate

67.6 g/L Ammonium chloride

572 mL/L Ammonium hydroxide

Reagent 2: **SEALAnalyticalAQ Hard-Calmagite** — Calmagite Reagent in DI Water:

0.5 g/L Calmagite

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DISCRETE ANALYZER CHEMISTRY

Total Hardness Method

Method Description

Total Hardness quantifies the concentration of magnesium and calcium ions, and other dissolved minerals in a sample. This method utilizes Mg-EDTA from **SEALAnalyticalAQ Hard-Buffer** to exchange free calcium ions for magnesium, thereby forming a more stable EDTA chelate. The remaining free magnesium reacts with calmagite in **SEALAnalyticalAQ Hard-Calmagite** at a pH of 10, producing a wine-red color that is measured photometrically at 520 nm. This method is largely unaffected by most interferences, with the exception of color and turbidity, which can be removed via filtration or corrected for in the analysis.

Historically, the Calmagite reagent also contained the buffer, with the EDTA solution being separate. The Calmagite & Buffer reagent also included SDS. To improve the long-term stability of the reagents without compromising method performance, the EDTA reagent was combined with the buffer, while the Calmagite was kept separate in deionized water. Additionally, SDS was removed, as it was suspected to contribute to reagent degradation.

Tip: Due to the absence of SDS, it is recommended that technicians run a thorough cleaning solution through the system following Total Hardness analysis to prevent reagent buildup and ensure the continued accuracy and longevity of the instrumentation.

Ca^{2+}+Mg-EDTA→Ca-EDTA+Mg^{2+}

Mg^{2+}+Calmagite→Mg-Calmagite Complex (wine-red)

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Compatible with SEAL Methods

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Methods Referenced

- EPA Method 130.1 — (Colorimetric, Automated EDTA)
- Standard Methods 2340 C — "EDTA Titrimetric Method"
  This method covers the determination of Total Hardness in drinking, surface, and saline waters.

Reagent Composition

Two Reagents are used for the Total Hardness method:

Reagent 1: **SEALAnalyticalAQ Hard-Buffer** — Mg-EDTA Buffer in DI water:

5 g/L EDTA disodium magnesium salt hydrate

67.6 g/L Ammonium chloride

572 mL/L Ammonium hydroxide

Reagent 2: **SEALAnalyticalAQ Hard-Calmagite** — Calmagite Reagent in DI Water:

0.5 g/L Calmagite

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Historically, the Calmagite reagent also contained the buffer, with the EDTA solution being separate. The Calmagite & Buffer reagent also included SDS. To improve the long-term stability of the reagents without compromising method performance, the EDTA reagent was combined with the buffer, while the Calmagite was kept separate in deionized water. Additionally, SDS was removed, as it was suspected to contribute to reagent degradation.

Tip: Due to the absence of SDS, it is recommended that technicians run a thorough cleaning solution through the system following Total Hardness analysis to prevent reagent buildup and ensure the continued accuracy and longevity of the instrumentation.

Ca^{2+}+Mg-EDTA→ Ca-EDTA+Mg^{2+}

Mg^{2+}+Calmagite→ Mg-Calmagite Complex (wine-red)

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