PROCEDURE FOR PREPARING STANDARD REAGENTS, MISCELLANEOUS SOLUTIONS, AND INDICATORS

(mo/yr)

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1.0 Purpose of Process

The purpose of this document is to describe the procedures to produce standard reagents, miscellaneous solutions and indicators.

2.0 Process Definition

This document contains several procedures that describe how to produce each solution.

3.0 Equipment

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4.0 Materials

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5.0 Safety Requirements

5.1 Safety Equipment

The technician performing the analysis must wear the appropriate gloves, lab coat and safety glasses.

5.2 Safety Procedures

If any of the glassware breaks during the procedure, the technician should dispose of the remains in the receptacle in the lab specifically for broken glass. All of the gloves worn to perform the procedure should be disposed in the hazardous waste barrel in production.
All electrical components should be examined to ensure that the components are in good working order to ensure that wires are not touching heat sources and to ensure that all electrical components are not located in or near water. If the technician has any trouble or questions, he/she should [redacted]

6.0 Technician Responsibilities

The technician should understand how to operate all measuring devices used in the procedure. The technician should have been trained to run titrations and to use the automatic titrators if necessary; specifically, the operator should [redacted]
The technician should understand the calculations and ask questions if the calculations seem unclear. The technician is responsible for [redacted]

7.0 Process Controls

The procedures must be performed according to the methods described herein. Any changes to the original document must be approved through the lab supervisor and sent through the signature process to maintain configuration control. All of the required data should be recorded. The lab supervisor should develop [redacted]

8.0 Procedures

8.1 Preparing and standardizing NaOH solutions

8.1.1 Preparing and standardizing 1N NaOH

8.1.1.1 Preparing 1N NaOH from 2N NaOH liquid

8.1.1.1.1 Measure [redacted] of [redacted] NaOH carbonate free liquid in a graduated cylinder and pour it into a [redacted] flask.

8.1.1.1.2 Dilute the solution to volume with Type I water. Place stir bar in the solution and mix for at least 15 minutes on a magnetic stirrer.

8.1.1.1.3 Standardize the solution as described in 8.1.1.4.

8.1.1.2 Preparing 1N NaOH from NaOH crystals

8.1.1.2.1 Weigh [redacted] of low carbonate NaOH pellets in a weigh boat.

8.1.1.2.2 Empty the weigh boat into [redacted] flask.

8.1.1.2.3 Rinse the weigh boat into the [redacted] flask with Type I water.

8.1.1.2.4 Fill [redacted] half way with Type I water and mix until [redacted] Then, dilute to volume with Type I water and add a stir bar. Stir on a magnetic stirrer for 30 minutes.

8.1.1.2.5 Standardize the solution as described in 8.1.1.4.

8.1.1.3 Standardizing 1N NaOH using 1N HCl

8.1.1.3.1 Fill a [redacted] buret with standardized 1N HCl.

8.1.1.3.2 Pipet [redacted] of the prepared NaOH solution into [redacted].

8.1.1.3.3 Place a stir bar in the beaker.

8.1.1.3.4 Add 2 drops of phenolphthalein indicator solution.

8.1.1.3.5 While stirring the beaker with a magnetic stirrer, titrate the prepared NaOH solution with the HCl to a color change. Note the amount of HCl used.

8.1.1.3.6 Calculate the normality of the NaOH solution as follows [redacted]

8.1.1.3.7 Repeat steps 8.1.1.3.1 to 8.1.1.3.6 to titrate 2 additional samples.

8.1.1.3.8 Average the ml's from the 3 titrations to calculate an average normality.

8.1.1.3.9 If it is not within [redacted] of the intended normality, adjust it accordingly and titrate it again starting at step 8.1.1.3.1.

8.1.1.3.10 Pour the NaOH solution into [redacted]

8.1.1.4 Standardizing 1N NaOH using Potassium Acid Phthalate

8.1.1.4.1 Fill a [redacted] buret with prepared NaOH solution.

8.1.1.4.2 Dry [redacted] of potassium acid phthalate at [redacted] for 1-2 hours.

8.1.1.4.3 Cool it in a desiccator.

8.1.1.4.4 Tare a weigh boat and weigh out [redacted] of primary standard potassium acid thalate.

8.1.1.4.5 Quantitatively transfer to [redacted] beaker.

8.1.1.4.6 Add [redacted] Type I water to beaker.

8.1.1.4.7 Add 2 drops of phenolphthalein indicator solution.

8.1.1.4.8 Record initial volume of NaOH in buret.

8.1.1.4.9 Add a stir bar to beaker.

8.1.1.4.10 Gently stir solution and titrate with NaOH to a permanent color change.

8.1.1.4.11 Record volume of titrant used.

8.1.1.4.12 Calculate the normality of the NaOH solution as follows [redacted]

8.1.1.4.13 Repeat steps 8.1.1.4.1 to 8.1.1.4.12 to titrate 2 additional samples.

8.1.1.4.14 Average the NaOH normality values from the 3 titrations.

8.1.1.4.15 If it is not within [redacted] of the intended number, adjust it accordingly and titrate it again starting at step 8.1.1.4.1.

8.1.2 Preparing and Standardizing 0.1N NaOH

8.1.2.1 Preparing 0.1N NaOH from 1N NaOH

8.1.2.1.1 Measure [redacted] of standardized [redacted] NaOH in a graduated cylinder and pour it into a [redacted] flask. Rinse the graduated cylinder into the flask with Type I water.

8.1.2.1.2 Dilute the solution to volume with Type I water. Place a stir bar in the flask and mix for at least 15 minutes on a magnetic stirrer.

8.1.2.1.3 Standardize the solution as described in 8.1.2.4.

8.1.2.2 Preparing 0.1N NaOH from NaOH crystals

8.1.2.2.1 Weigh out [redacted] of low carbonate NaOH pellets into weigh boat.

8.1.2.2.2 Empty the weigh boat into [redacted] flask.

8.1.2.2.3 Rinse the weigh boat [redacted] with Type I water.

8.1.2.2.4 Fill [redacted] half way with Type I water and mix until [redacted] Dilute [redacted] to volume with Type I water and add a stir bar. Stir on a magnetic stirrer for 30 minutes.

8.1.2.2.5 Standardize the solution as described in 8.1.2.4.

8.1.2.3 Standardizing 0.1N NaOH using 0.1N HCl

8.1.2.3.1 Fill a [redacted] buret with standardized 0.1N HCl.

8.1.2.3.2 Pipet [redacted] of the prepared NaOH solution into [redacted] beaker.

8.1.2.3.3 Place stir bar in the beaker.

8.1.2.3.4 Add 2 drops phenolphthalein indicator solution.

8.1.2.3.5 While stirring the beaker with a magnetic stirrer, titrate the prepared NaOH solution with the HCl solution to a color change. Note the amount of HCl titrant used.

8.1.2.3.6 Calculate the normality of the NaOH solution as follows:

[redacted]

8.1.2.3.7 Repeat steps 8.1.2.3.1-8.1.2.3.6 to titrate 2 additional samples.

8.1.2.3.8 Average the ml's from the 3 titrations to calculate an average normality.

8.1.2.3.9 If it is not within [redacted] of the intended number, adjust it accordingly and titrate it again starting at step 8.1.2.3.1.

8.1.2.3.10 Pour the NaOH solution into [redacted]

8.1.2.4 Standardizing 0.1N NaOH using Potassium Acid Phthalate

8.1.2.4.1 Fill a [redacted] buret with the prepared NaOH solution.

8.1.2.4.2 Dry [redacted] of potassium acid phthalate at 100°C for 1-2 hours. Cool it in a desiccator.

8.1.2.4.3 Weigh [redacted] of the dried potassium acid phthalate in a weigh boat. Note the weight to the nearest 0.0001g.

8.1.2.4.4 Empty the weigh boat into [redacted] beaker.

8.1.2.4.5 Rinse the weighing boat into the beaker with Type I water. Add [redacted] Type I water to the beaker.

8.1.2.4.6 Place a stir bar in the beaker. Stir with a magnetic stirrer to dissolve the potassium acid phthalate. The beaker may have to be heated slightly to get the entire solid into solution.

8.1.2.4.7 Add 2 drops of phenolphthalein indicator solution.

8.1.2.4.8 While continuing to stir the beaker, titrate the potassium acid phthalate solution with the prepared NaOH solution to a color change. Note the amount of NaOH titrant used.

8.1.2.4.9 Calculate the normality of the NaOH solution as follows:

[redacted]

8.1.2.4.10 Repeat steps 8.1.2.4.1 to 8.1.2.4.9 to titrate 2 additional samples.

8.1.2.4.11 Average the NaOH normality values from the 3 titrations.

8.1.2.4.12 If it is not within [redacted] of the intended number, adjust it accordingly and titrate it again starting at step 8.1.1.4.1.

8.1.2.4.13 Pour the NaOH solution into [redacted]

8.1.3 Preparing and Standardizing 0.1N NaOH

8.1.3.1 Pipet [redacted] of standardized [redacted] NaOH into [redacted] flask.

8.1.3.2 Dilute the flask to volume with Type I water. Stopper the flask and shake it to thoroughly mix the NaOH solution.

8.1.3.3 Fill [redacted] buret with standardized 0.1N HCl.

8.1.3.4 Pipet [redacted] of the prepared NaOH solution into [redacted] beaker.

8.1.3.5 Place stir bar in the beaker.

8.1.3.6 Add 2 drops of phenolphthalein indicator to the beaker.

8.1.3.7 While stirring the beaker with a magnetic stirrer, titrate the prepared NaOH solution with the HCl to a color change. Note the amount of HCl used.

8.1.3.8 Calculate the normality of the NaOH solution as follows:

[redacted]

8.1.3.9 Repeat steps 8.1.3.3-8.1.3.8 to titrate two additional samples.

8.1.3.10 Average the ml's from the 3 titrations. Calculate the average normality.

8.1.3.11 If it is not within [redacted] of the intended value, adjust it accordingly and titrate it again starting at step 8.1.3.3.

8.1.3.12 Pour the NaOH solution into [redacted]

8.1.4 Preparing and Standardizing 0.0159N NaOH

8.1.4.1 Pour [redacted] of Type I water into [redacted] flask.

NOTE: 16ml is an approximation and it is not necessary to be completely accurate with the initial volume measurement.

8.1.4.2 Measure [redacted] of 1N NaOH into a graduated cylinder and pour it into the volumetric.

8.1.4.3 Dilute the volumetric to volume with Type I water. Place a stir bar in the flask. Stopper the flask and stir it on a magnetic stirrer to thoroughly mix the NaOH solution.

8.1.4.4 Fill a [redacted] buret with standardized 0.1N HCl.

8.1.4.5 Pipet [redacted] of the prepared NaOH solution into [redacted] beaker.

8.1.4.6 Place stir bar in the beaker.

8.1.4.7 Add 2 drops of phenolphthalein indicator solution to the beaker.

8.1.4.8 While stirring the beaker with a magnetic stirrer, titrate the prepared NaOH solution with the HCl to a color change. Note the amount of titrant used.

8.1.4.9 Calculate the normality of the NaOH solution as follows [redacted]

8.1.4.10 The normality of the NaOH solution should be 0.0159. If it is not 0.0159N, add small amounts of [redacted] or [redacted], mix thoroughly, and titrate the solution again as described in steps 8.1.4.4-8.1.4.8.

8.1.4.11 When the solution is 0.0159N, repeat steps 8.1.4.4-8.1.4.8 to titrate 2 additional samples. The average normality of the 3 titrations should be 0.0159N.

8.1.4.12 Pour the NaOH solution into [redacted]

8.2 Preparing and Standardizing HCl Solutions

8.2.1 Preparing 1N HCl and Standardizing 1N HCl with sodium carbonate

8.2.1.1 Dilute [redacted] of hydrochloric acid to [redacted] and mix thoroughly.

8.2.1.2 Weigh accurately [redacted] sodium carbonate, Alkalimetric Standard that has previously been heated at [redacted] for [redacted].

8.2.1.3 Dissolve the sodium carbonate in [redacted] if Type I water.

8.2.1.4 Add 0.1ml of methyl red indicator solution.

8.2.1.5 Add a stir bar to the solution.

8.2.1.6 While stirring the solution, add the acid slowly from a buret until [redacted]

8.2.1.7 Heat the solution to boiling. Cool the solution to room temperature. Titrate again until [redacted]

8.2.1.8 Repeat this procedure until [redacted]

8.2.1.9 Repeat steps 8.2.1.2-8.2.1.8 for two additional samples.

8.2.1.10 Calculate the normality of the HCl solution as follows [redacted]

8.2.1.11 Average the three samples' normalities. If the average is not within [redacted] of the intended normality, adjust it accordingly and titrate again starting at step 8.2.1.2.

8.2.2 Preparing and standardizing 0.1N HCl

8.2.2.1 Dilute [redacted] of hydrochloric acid to [redacted] and mix thoroughly.

8.2.2.2 Weigh accurately [redacted] of sodium carbonate, Alkalimetric Standard that has previously been heated at [redacted] for [redacted].

8.2.2.3 Dissolve the sodium carbonate in [redacted] if Type I water.

8.2.2.4 Add [redacted] l of methyl red indicator solution.

8.2.2.5 Add a stir bar to the solution.

8.2.2.6 While stirring the solution, add the acid slowly from a buret until [redacted]

8.2.2.7 Heat the solution to boiling. Cool the solution to room temperature. Titrate again until [redacted]

8.2.2.8 Repeat this procedure until [redacted]

8.2.2.9 Repeat steps 8.2.2.2-8.2.2.8 for two additional samples.

8.2.2.10 Calculate the normality of the HCl solution as follows [redacted]

8.2.2.11 Average the three samples' normalities. If the average is not within [redacted] of the intended normality, adjust it accordingly and titrate again starting at step 8.2.2.2.

8.2.3 Preparing and standardizing 0.001N HCl

8.2.3.1 Obtain [redacted] of previously standardized 0.1N HCl.

8.2.3.2 Transfer to a [redacted] flask.

8.2.3.3 Dilute the flask to volume with Type I water.

8.2.3.4 Obtain [redacted] of the diluted solution.

8.2.3.5 Transfer to [redacted] beaker.

8.2.3.6 Add 1 drop of phenolphthalein indicator to the solution.

8.2.3.7 Titrate with previously standardized 0.01N NaOH to a color change.

8.2.3.8 Calculate the normality with the following equation: [redacted]

8.3 Preparing and Standardizing Miscellaneous Solutions

8.3.1 Preparing and standardizing 0.05M EDTA

8.3.1.1 Preparing [redacted] EDTA

8.3.1.1.1 Dry approximately [redacted] of EDTA (disodium salt form) at [redacted] for [redacted] hour. Cool to room temperature in a desiccator for approximately 1 hour.

8.3.1.1.2 Weigh [redacted] of the EDTA in a weigh boat.

8.3.1.1.3 Empty the weigh boat into [redacted] flask.

8.3.1.1.4 Rinse the weigh boat into the flask with Type I water.

8.3.1.1.5 Fill the flask to volume with Type I water.

8.3.1.1.6 Place a stir bar in the flask and stir with a magnetic stirrer until [redacted]

8.3.1.1.7 Standardize the EDTA mixture.

8.3.1.2 Standardizing the EDTA with Calcium carbonate

8.3.1.2.1 Fill a [redacted] buret with the prepared EDTA solution.

8.3.1.2.2 Weigh [redacted] in a weigh boat. Note the weight to the nearest 0.0001g.

8.3.1.2.3 Pour the calcium carbonate into [redacted] beaker. Rinse the weigh boat into the beaker with [redacted] Type I water.

8.3.1.2.4 Swirl the beaker to form slurry.

8.3.1.2.5 Cover the beaker with a watch glass. Add 4-5 drops of concentrated HCl via a pipet inserted between the lip of the beaker and the edge of the watch glass.

8.3.1.2.6 Swirl the beaker to dissolve the calcium carbonate.

8.3.1.2.7 Rinse the sides of the beaker, the outer surface of the pipet, and the watch glass with Type I water. Dilute the beaker with Type I water to [redacted].

8.3.1.2.8 Place a stir bar in the beaker.

8.3.1.2.9 While stirring the solution with a magnetic stirrer, add [redacted] of the EDTA being standardized from 8.3.1.2.1.

8.3.1.2.10 Add [redacted] sodium hydroxide ([redacted]) and [redacted] of hydroxyl naphthol blue indicator.

8.3.1.2.11 Continue to titrate the solution with the EDTA until [redacted]

8.3.1.2.12 Calculate the molarity of the EDTA solution as follows

[redacted]

8.3.1.2.13 Repeat steps 8.3.1.2.1-8.3.1.2.11 to titrate 2 additional samples.

8.3.1.2.14 Average the ml's from the 3 titrations to calculate an average molarity.

8.3.1.2.15 If it is not within [redacted] of the intended molarity, adjust it accordingly and titrate again starting at step 8.3.1.2.1.

8.3.1.2.16 Pour the mixture into [redacted]

8.3.2 Preparing and Standardizing 0.05M Magnesium Sulfate

8.3.2.1 Weigh [redacted] of $MgSO_4*7H_2O$ in a weigh boat.

8.3.2.2 Empty the weigh boat into [redacted] flask. Rinse the weigh boat into the flask with DI water.

8.3.2.3 Dilute the solution to volume with Type I water. Place a stir bar in the flask and stir with a magnetic stirrer until [redacted]

8.3.2.4 Fill [redacted] burette with the prepared 0.05M $MgSO_4$ solution.

8.3.2.5 Pipet [redacted] of standardized 0.05M EDTA solution into [redacted] beaker.

8.3.2.6 Add [redacted] of muspratt reagent to the beaker.

8.3.2.7 Add approximately [redacted] of Eriochrome Black T indicator to the beaker.

8.3.2.8 While stirring the beaker with a magnetic stirrer, titrate it with the $MgSO_4$ solution (from 8.3.2.4) until [redacted]. Note the amount of titrant used.

8.3.2.9 Calculate the molarity of the $MgSO_4$ solution as follows

[redacted]

8.3.2.10 Repeat steps 8.3.2.4-8.3.2.8 for 2 additional samples.

8.3.2.11 Average the mls from the 3 titrations to calculate an average molarity.

8.3.2.12 If it is not within [redacted] of the intended molarity, adjust it accordingly and titrate again starting at step 8.3.2.4.

8.3.2.13 Pour the mixture into [redacted]

8.3.3 Preparing the Muspratt Reagent

8.3.3.1 Weigh [redacted] of NH_4Cl in a weigh boat.

8.3.3.2 Empty the weighing boat into [redacted] flask. Rinse the weigh boat into the flask with Type I water.

8.3.3.3 Add [redacted] of Type I water to the flask. Shake the flask until [redacted]

8.3.3.4 Working under a laboratory hood measure [redacted] in a graduated cylinder. Add this to the flask.

8.3.3.5 Shake the flask until [redacted] Dilute the flask to volume with Type I water.

8.3.3.6 Pour the solution into [redacted]

8.3.4 Preparing and standardizing 0.01M zinc sulfate

8.3.4.1 Weigh [redacted] of $ZnSO_4$ (formula weight: [redacted]) in a weigh boat

8.3.4.2 Empty the $ZnSO_4$ into [redacted] flask.

8.3.4.3 Rinse the weigh boat into the flask with Type I water.

8.3.4.4 Dilute the flask to volume with Type I water.

8.3.4.5 Place a stir bar in the flask. Stir the solution with a magnetic stirrer until [redacted]

8.3.4.6 Fill [redacted] with the prepared $ZnSO_4$ solution.

8.3.4.7 Pipet [redacted] into [redacted] beaker.

8.3.4.8 Add [redacted] of Muspratt reagent (buffer) to the beaker.

8.3.4.9 Add [redacted] of Eriochrome Black T indicator to the beaker.

8.3.4.10 Add [redacted] of the prepared $ZnSO_4$ solution (from step 8.3.4.6) to the beaker.

8.3.4.11 Continue to titrate the beaker with the prepared $ZnSO_4$ solution until [redacted]

8.3.4.12 Calculate the molarity of the $ZnSO_4$ solution as follows [redacted]

8.3.4.13 Repeat steps 8.3.4.6-8.3.4.11 to titrate 2 additional samples.

8.3.4.14 Average the ml's from the 3 titrations to calculate an average molarity.

8.3.4.15 If it is not within [redacted] of the intended molarity, adjust it accordingly and titrate again starting at step 8.3.4.6.

8.3.4.16 Pour the solution into [redacted]

8.3.5 Preparing 0.1M Dimethylglyoxime (DMG)

8.3.5.1 Weigh [redacted] of DMG in weigh boat.
8.3.5.2 Empty the DMG into [redacted] flask.
8.3.5.3 Rinse the weigh boat into the flask with [redacted] l of denatured ethyl alcohol.
8.3.5.4 Dilute the flask to volume with Type I water.
8.3.5.5 Place a stir bar in the flask. Stir with a magnetic stirrer to dissolve the DMG. It may be necessary to heat the flask slightly to get it all into solution.
8.3.5.6 Place a stopper in the flask. Label the flask with [redacted]

8.3.6 Preparing decarbonated Type I water

8.3.6.1 Place [redacted] of Type I water in [redacted] beaker. Bubble nitrogen through the Type I water at [redacted] SCFH for [redacted] to remove the dissolved gasses from the Type I water. Cover the beaker.
8.3.6.2 As an alternative method for removing the dissolved gasses, [redacted]

8.3.7 Preparing pH 1.68 buffer solution

8.3.7.1 Use SRM 189a Potassium Tetroxalate salt
8.3.7.2 Measure [redacted] of salt (un-dried) for each [redacted] solution desired.
8.3.7.3 Record the temperature and resistivity of the Type I water.
8.3.7.4 Rinse a volumetric flask appropriate to the volume of solution desired with Type I water.
8.3.7.5 Pour the crystals into the volumetric flask and fill to the mark with Type I water. Add a clean stir bar and stir the solution until [redacted]
8.3.7.6 Use a new nalgene bottle or the bottle returned from production that previously contained the 1.68 buffer solution to contain the new solution. Rinse the bottle with a small amount of the new solution. Fill the bottle with the buffer and label appropriately. Solutions expire [redacted] from preparation.
8.3.7.7 Record data in appropriate log book or log form.

8.3.8 Preparing 6.86 buffer solution

8.3.8.1 Measure [redacted] KCl .
8.3.8.2 Pour KCl into [redacted] volumetric.
8.3.8.3 Rinse weigh boat with Type I water into the volumetric.
8.3.8.4 Dilute to volume with [redacted] buffer.
8.3.8.5 Stir until [redacted]
8.3.8.6 Label appropriately.
8.3.8.7 Solution has [redacted] shelf life.

8.3.9 Preparing saturated KCl solution for production

8.3.9.1 Determine the molarity of the solution that production needs. This molarity will=X in the following equation:
[redacted]

8.3.9.2 Pour KCl into [redacted] volumetric and dilute to volume with Type I water. Stir until [redacted] Be patient...this will take time. The flask will be cold to the touch. This is normal.

8.3.9.3 Label appropriately.

8.3.9.4 Shelf life is [redacted]

8.3.10 Preparing pH 11.70 buffer solution

8.3.10.1 Pour [redacted] of commercially prepared pH 10 buffer into a small beaker.

8.3.10.2 Place a stir bar in the beaker and stir with a magnetic stirrer.

8.3.10.3 Suspend a digital pH meter probe in the beaker.

8.3.10.4 While continuing to stir the beaker, add drops of [redacted] until [redacted]

8.3.10.5 Place the buffer solution in a plastic bottle. Label the bottle with [redacted]

8.3.11 Preparing 50% by weight sulfuric acid solution

8.3.11.1 Place a [redacted] glass bottle with funnel on a scale. Tare the scale.

8.3.11.2 Add Type I water to fill the bottle approximately half full. Note the weight of the Type I water.

8.3.11.3 Slowly add the same weight of concentrated sulfuric acid to the bottle. Add the sulfuric acid very slowly as the solution will heat up.

8.3.11.4 After the sulfuric acid has been added, cap the bottle loosely. After the solution has cooled down, tighten the cap.

8.3.11.5 Label the bottle with [redacted]

8.3.12 Preparing Nochromix

8.3.12.1 Pour one package of Nochromix crystals into [redacted]
Use safety coated glass bottles or heavy wall polyethylene containers only.

8.3.12.2 A half batch of Nochromix may be made.

8.3.12.2.1 Weigh a full package of Nochromix crystals.

8.3.12.2.2 Divide the weight by 2.

8.3.12.2.3 Add half the weight of a whole package to a half bottle of concentrated sulfuric acid.

8.3.12.2.4 Save the rest of the crystals for future use.
8.3.12.3 Place cap loosely on bottle.
8.3.12.4 Let stand overnight.
NOTE: Do not close or store the container tightly capped!!
8.3.12.5 Label the bottle.
8.3.12.6 For best results, use [redacted]
8.3.12.7 When the cleaning solution is stale or discolored, it can be regenerated with [redacted]

8.3.13 Preparing 2Molar cadmium nitrate

8.3.13.1 Clean a 5 gallon graduated pre-weighed bucket equipped with a spigot and lid.
8.3.13.2 Have production weight [redacted] of cadmium nitrate into the bucket.
8.3.13.3 Get exact weight of cadmium nitrate and bucket.
8.3.13.4 Subtract bucket weight to get cadmium nitrate weight.
8.3.13.5 Divide cadmium nitrate weight by [redacted] to get total volume.
8.3.13.6 Dilute with DI water to the volume obtained in 8.3.13.5.
8.3.13.7 Set up a mixer and let the solution mix for a minimum of 30 minutes.
NOTE: The mixing should be done under a hood in the lab.
8.3.13.8 Dilute the solution according to the following:
8.3.13.8.1 [redacted]
8.3.13.8.2
8.3.13.8.3
8.3.13.8.4
8.3.13.8.5
8.3.13.8.6
8.3.13.9 Set up the AA to analyze the solution.
8.3.13.9.1 Turn on the equipment to warm up for 10 minutes.
8.3.13.9.2 Set the slit width to [redacted] for the cadmium program.
8.3.13.9.3 Set the wavelength to [redacted] nm.
8.3.13.9.4 Use cadmium standards with the following concentrations:
8.3.12.9.4.1 250ppm
8.3.12.9.4.2 500ppm
8.3.12.9.4.3 1000ppm
8.3.13.9.5 Calculate the molarity of the solution using the following:
[redacted]
8.3.13.10 If the solution is not with 2M ± [redacted], adjust it accordingly.
8.3.13.11 Pour the cadmium nitrate solution into [redacted]

[redacted]

8.3.14 Preparing 3.5% Methocel Solution

8.3.14.1 Check the conductivity of the laboratory DI water. Do not prepare the methocel solution unless the DI water conductivity is [redacted] ohm-cm.
8.3.14.2 Measure [redacted] of DI water into [redacted] flask.
8.3.14.3 Use a hot plate to heat the water in the flask to [redacted].
8.3.14.4 Measure [redacted] of DI water into another [redacted] flask.
8.3.14.5 Place this flask in an ice water bath until [redacted]
8.3.14.6 Weigh out [redacted] of type [redacted] methocel into a weigh boat.

NOTE: Methocel powder must always be kept in a tightly closed container. The container should have a green tag. If the powder does not have a green tag, do not use and notify QC.

8.3.14.7 Remove the flask from the hot plate and slowly add the methocel to the hot water, a little bit at a time. Agitate the solution while adding the methocel by rotating the flask. Agitate the solution until [redacted] If a gel forms, stop adding the Methocel until [redacted]
8.3.14.8 Remove the other flask from the ice bath and slowly add the cold water to the hot solution, a little bit at a time. If a gel forms, stop adding the cold water until the gel dissolves.
8.3.14.9 After all the cold water has been added, the solution should [redacted]
8.3.14.10 Seal the Erlenmeyer flask tightly with a synthetic rubber stopper or a cork that has been covered with [redacted].
8.3.14.11 Label the flask with [redacted]

8.4 Preparing indicator solutions

8.4.1 Preparing phenolphthalein indicator for process use

NOTE: Phenolphthalein can be prepared as either an aqueous or alcohol solution.

8.4.1.1 Aqueous solution: Dissolve [redacted] of phenolphthalein disodium salt in Type I water in a [redacted] volumetric. Dilute [redacted] volumetric to volume with Type I water.
8.4.1.2 Alcohol solution: Dissolve [redacted] of phenolphthalein disodium salt in [redacted] of denatured ethyl alcohol in [redacted] volumetric. Dilute [redacted] volumetric to volume with Type I water.
8.4.1.3 The phenolphthalein solution should be within [redacted] range. The titration end points are clear (8.2) to red-violet (9.8).

8.4.2 Preparing phenolphthalein indicator for leak tests

8.4.2.1 Dissolve 1g phenolphthalein disodium salt in 100ml of 95% ethyl alcohol.

8.4.2.2 Dilute 1ml of the solute in 500ml of Type I water.

[redacted]

8.4.2.3 Adjust the pH of the solution to [redacted] with [redacted] potassium hydroxide, or [redacted] sodium hydroxide.

8.4.2.4 If the pH drops below [redacted] readjust it to [redacted] with [redacted] potassium hydroxide or [redacted] sodium hydroxide.

8.4.3 Preparing methyl orange indicator

8.4.3.1 Dissolve [redacted] methyl orange powder in Type I water. Dilute to [redacted] with Type I water.

8.4.3.2 Methyl orange should be in [redacted] range. The titration end points are pink (3.1) to yellow (4.4).

8.4.4 Preparing methyl red indicator

8.4.4.1 Dissolve [redacted] methyl red powder in Type I water. Dilute to [redacted] with Type I water.

8.4.4.2 Methyl red indicator should be [redacted] pH range. The titration end points are pink (4.2) to yellow (6.2).

8.4.5 Preparing methyl red indicator for production use

8.4.5.1 Dissolve 1g methyl red powder in Type I water.

8.4.5.2 Dilute to [redacted] with Type I water, as production always uses [redacted].

8.4.6 Preparing methyl red-methylene blue indicator

8.4.6.1 Weigh [redacted] of methyl red indicator powder in a weigh boat.

8.4.6.2 Empty the weigh boat into [redacted] flask.

8.4.6.3 Rinse the weigh boat with [redacted] into the volumetric.

8.4.6.4 Dilute the flask to volume with [redacted].

8.4.6.5 Pour the solution into [redacted].

8.4.6.6 Weigh [redacted] of the methylene blue indicator powder in a weigh boat.

8.4.6.7 Empty the weigh boat into [redacted] flask.

8.4.6.8 Rinse the weigh boat with [redacted] into the volumetric.

8.4.6.9 Dilute the flask to volume with 95% alcohol.

8.4.6.10 Pipette 50ml of the methylene blue into the plastic bottle and shake.

8.4.7 Preparing Eriochrome Black T indicator

8.4.7.1 Weigh [redacted] of Eriochrome Black T powder in a weigh boat.

8.4.7.2 Empty the weigh boat into [redacted] flask.

8.4.7.3 Rinse the weigh boat into the volumetric flask with [redacted] of Type I water.

8.4.7.4 Pipet [redacted] l of [redacted] buffer into the flask.

8.4.7.5 Dilute the flask to volume with [redacted].

8.4.7.6 Place a stir bar in the flask. Stir with a magnetic stirrer for 30 minutes.

8.4.7.7 Pour the solution into a plastic bottle. Label the bottle with [redacted]

8.4.7.8 Store the indicator in [redacted] Discard the indicator after [redacted].

8.5 Preparing Standards for the AA spectrometer

NOTE: This recipe is not used when analyzing the production solutions. The technician should only use this recipe for the environmental sample analysis.

8.5.1 Preparing [redacted] stock standard mix

8.5.1.1 Make a batch of fresh Nochromix. The glassware that is to be used should be as analytically clean as possible.

8.5.1.2 Clean a [redacted] flask, a [redacted] bottle, and the necessary number of [redacted] thoroughly. The number of [redacted] is dependent upon the number of elements that will be mixed.

8.5.1.3 Pipet [redacted] of each element into [redacted] flask:

[redacted]

8.5.1.4 Add [redacted] of concentrated nitric acid (trace metal grade) to the flask.

8.5.1.5 Dilute to volume with Type I water.

8.5.1.6 Shake the flask to [redacted].

8.5.1.7 Pour a small amount of the [redacted] standard just made into the plastic bottle. Screw on the cap and shake the bottles.

8.5.1.8 Discard that small amount of solution.

8.5.1.9 Pour the remaining [redacted] standard mix into [redacted]