

REDACTED

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PROCEDURE FOR PREPARING STANDARD REAGENTS, MISCELLANEOUS SOLUTIONS, AND INDICATORS

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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

- 3.1 [REDACTED]
- 3.2 [REDACTED]
- 3.2 [REDACTED]
- 3.3 [REDACTED]
- 3.4 [REDACTED]
- 3.5 [REDACTED]
- 3.6 [REDACTED]
- 3.7 [REDACTED]
- 3.8 [REDACTED]
- 3.9 [REDACTED]
- 3.10 Various size beakers
- 3.11 Various size glass bottles
- 3.12 [REDACTED]
- 3.13 Various size graduated cylinders
- 3.14 [REDACTED]
- 3.15 Various size volumetric flasks
- 3.16 [REDACTED]

4.0 Materials

- 4.1 [REDACTED]
- 4.2 [REDACTED]
- 4.3 [REDACTED]
- 4.4 [REDACTED]
- 4.5 [REDACTED]
- 4.6 [REDACTED]
- 4.7 [REDACTED]
- 4.8 [REDACTED]
- 4.9 [REDACTED]
- 4.10 [REDACTED]
- 4.11 [REDACTED]
- 4.12 [REDACTED]
- 4.13 [REDACTED]

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[Redacted content]

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5.0 Safety Requirements

5.1 Safety Equipment

The technician performing the analysis must

5.2 Safety Procedures

If any of the glassware breaks

All electrical components should

If the technician has

6.0 Technician Responsibilities

The technician should

the operator should

The technician should

The technician is responsible for

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7.0 Process Controls

The procedures must

develop

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 of NaOH carbonate free liquid pour it into a flask.

8.1.1.1.2 Dilute the solution to volume with . Place stir bar in the solution and mix for

8.1.1.1.3

8.1.1.2 Preparing 1N NaOH from NaOH crystals

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

8.1.1.2.2 Empty the weigh boat into flask.

8.1.1.2.3 Rinse the weigh boat into the flask with

8.1.1.2.4 Fill half way with and mix until . Then, dilute to volume with and add a stir bar. Stir on a magnetic stirrer for .

8.1.1.2.5

8.1.1.3 Standardizing 1N NaOH using 1N HCl

8.1.1.3.1 Fill a buret with standardized 1N HCl.

8.1.1.3.2 Pipet of the prepared NaOH solution into .

8.1.1.3.3 Place a stir bar in the beaker.

8.1.1.3.4 Add 2 drops of solution.

8.1.1.3.5 While stirring the beaker with a magnetic stirrer, titrate

8.1.1.3.6 Calculate as follows

8.1.1.3.7 Repeat

8.1.1.3.8

- 8.1.1.3.9 If it is not within [REDACTED]
- 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 [REDACTED]
- 8.1.1.4.3 [REDACTED]
- 8.1.1.4.4 Tare a weigh boat and weigh out [REDACTED] of primary standard potassium acid thalate.
- 8.1.1.4.5 [REDACTED] transfer to [REDACTED] beaker.
- 8.1.1.4.6 Add [REDACTED] to beaker.
- 8.1.1.4.7 Add [REDACTED]
- 8.1.1.4.8 Record [REDACTED]
- 8.1.1.4.9 Add a stir bar to beaker.
- 8.1.1.4.10 Gently stir solution and titrate [REDACTED]
- 8.1.1.4.11 Record [REDACTED]
- 8.1.1.4.12 Calculate the normality of the NaOH solution as follows [REDACTED]
- 8.1.1.4.13 [REDACTED]
- 8.1.1.4.14 [REDACTED]
- 8.1.1.4.15 If it is not within [REDACTED]

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 [REDACTED]
- 8.1.2.1.2 Dilute the solution [REDACTED] Place a stir bar in the flask and mix [REDACTED]
- 8.1.2.1.3 [REDACTED]

8.1.2.2 Preparing 0.1N NaOH from NaOH crystals

- 8.1.2.2.1 Weigh out [REDACTED]
- 8.1.2.2.2 Empty the weigh boat into [REDACTED] flask.
- 8.1.2.2.3 Rinse the weigh boat [REDACTED] with [REDACTED]

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

8.1.2.2.5 [redacted]

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 [redacted]

8.1.2.3.5 While stirring the beaker with a magnetic stirrer, [redacted]

8.1.2.3.6 Calculate the normality of the NaOH solution as follows: [redacted]

8.1.2.3.7 [redacted]

8.1.2.3.8 [redacted]

8.1.2.3.9 If it is not within [redacted]

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 [redacted]

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

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 [redacted]. Add [redacted] 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 [redacted]

8.1.2.4.7 Add [redacted]

8.1.2.4.8 While continuing to stir the beaker, [redacted]

8.1.2.4.9 Calculate the normality of the NaOH solution as follows: [redacted]

8.1.2.4.10 [redacted]

8.1.2.4.11 [REDACTED]

8.1.2.4.12 If it is not within [REDACTED]

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 [REDACTED]. 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 [REDACTED]

8.1.3.7 While stirring the beaker [REDACTED]

8.1.3.8 Calculate the normality of the NaOH solution as follows:

8.1.3.9 [REDACTED]

8.1.3.10 [REDACTED]

8.1.3.11 If it is not within [REDACTED]

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 [REDACTED] into [REDACTED] flask.

NOTE: [REDACTED] it is not necessary [REDACTED]

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 [REDACTED]. 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 [REDACTED]

8.1.4.8 While stirring the beaker with a magnetic stirrer, [REDACTED]

8.1.4.9 Calculate the normality of the NaOH solution as follows

8.1.4.10

8.1.4.11 When the solution is

8.1.4.12 Pour the NaOH solution into

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 of hydrochloric acid to and mix thoroughly.

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

8.2.1.3 Dissolve the sodium carbonate in if

8.2.1.4 Add

8.2.1.5 Add a stir bar to the solution.

8.2.1.6 While stirring the solution, add until

8.2.1.7 Heat the solution to boiling. Titrate again until

8.2.1.8 Repeat this procedure until

8.2.1.9

8.2.1.10 Calculate the normality of the HCl solution as follows

8.2.1.11

8.2.2 *Preparing and standardizing 0.1N HCl*

8.2.2.1 Dilute of hydrochloric acid to and mix thoroughly.

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

8.2.2.3 Dissolve the sodium carbonate in if

8.2.2.4 Add

8.2.2.5 Add a stir bar to the solution.

8.2.2.6 While stirring the solution, add until

8.2.2.7 Heat the solution to boiling. [REDACTED] Titrate again until [REDACTED]

8.2.2.8 Repeat this procedure until [REDACTED]

8.2.2.9 [REDACTED]

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

8.2.2.11 [REDACTED]

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 [REDACTED]

8.2.3.4 Obtain [REDACTED] of the diluted solution.

8.2.3.5 Transfer to [REDACTED] beaker.

8.2.3.6 Add [REDACTED]

8.2.3.7 Titrate with [REDACTED]

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 [REDACTED]

8.3.1.1.5 Fill the flask to volume with [REDACTED]

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 [REDACTED]

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 [REDACTED]

8.3.1.2.3 Pour the [REDACTED] into [REDACTED] beaker. Rinse the weigh boat into the beaker with [REDACTED]

- 8.3.1.2.4 Swirl the beaker to [REDACTED]
- 8.3.1.2.5 Cover the beaker with a watch glass. Add [REDACTED] via a pipet inserted between [REDACTED]
- 8.3.1.2.6 Swirl the beaker to dissolve [REDACTED]
- 8.3.1.2.7 Rinse [REDACTED] with [REDACTED]. Dilute the beaker with [REDACTED] 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] ([REDACTED]) and [REDACTED] of [REDACTED]
- 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 [REDACTED]
- 8.3.1.2.14 [REDACTED]
- 8.3.1.2.15 If it is not within [REDACTED] of the intended molarity, [REDACTED]
- 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 [REDACTED] in a weigh boat.
- 8.3.2.2 Empty the weigh boat into [REDACTED] flask. Rinse the weigh boat into the flask with [REDACTED]
- 8.3.2.3 Dilute the solution to volume with [REDACTED]. 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 MgSO₄ solution.
- 8.3.2.5 Pipet [REDACTED] of standardized [REDACTED] solution into [REDACTED] beaker.
- 8.3.2.6 Add [REDACTED] of [REDACTED] to the beaker.
- 8.3.2.7 Add approximately [REDACTED] of [REDACTED] indicator to the beaker.
- 8.3.2.8 While stirring the beaker with a magnetic stirrer, titrate it with the MgSO₄ solution (from 8.3.2.4) until [REDACTED]. Note the amount of titrant used.
- 8.3.2.9 Calculate the molarity of the MgSO₄ solution as follows [REDACTED]
- 8.3.2.10 [REDACTED]
- 8.3.2.11 [REDACTED]
- 8.3.2.12 If it is not within [REDACTED] of the intended molarity, [REDACTED]

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8.3.2.13 Pour the mixture into [REDACTED]

8.3.3 **Preparing the Muspratt Reagent**

8.3.3.1 Weigh [REDACTED] of [REDACTED] in a weigh boat.

8.3.3.2 Empty the weighing boat into [REDACTED] flask. Rinse the weigh boat into the flask with [REDACTED]

8.3.3.3 Add [REDACTED] of [REDACTED] 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 [REDACTED]

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 ZnSO₄ (formula weight [REDACTED]) in a weigh boat

8.3.4.2 Empty the ZnSO₄ into [REDACTED] flask.

8.3.4.3 Rinse the weigh boat into the flask with [REDACTED]

8.3.4.4 Dilute the flask to volume with [REDACTED]

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 ZnSO₄ solution.

8.3.4.7 Pipet [REDACTED] into [REDACTED] beaker.

8.3.4.8 Add [REDACTED] of [REDACTED] to the beaker.

8.3.4.9 Add [REDACTED] of [REDACTED] indicator to the beaker.

8.3.4.10 Add [REDACTED] of the prepared ZnSO₄ solution (from step 8.3.4.6) to the beaker.

8.3.4.11 Continue to titrate the beaker with the prepared ZnSO₄ solution until [REDACTED]

8.3.4.12 Calculate the molarity of the ZnSO₄ solution as follows

[REDACTED]

8.3.4.13 [REDACTED]

8.3.4.14 [REDACTED]

8.3.4.15 If it is not within [REDACTED] of the [REDACTED]

8.3.4.16 Pour the solution into [REDACTED]

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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 [REDACTED]
- 8.3.5.4 Dilute the flask to volume with [REDACTED]
- 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 [REDACTED]
- 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 [REDACTED] 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 [REDACTED] 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 [REDACTED]
- 8.3.7.4 Rinse a volumetric flask appropriate to the volume of solution desired with [REDACTED]
- 8.3.7.5 Pour the crystals into the volumetric flask and fill to the mark with [REDACTED]. Add a clean stir bar and stir the solution until [REDACTED]
- 8.3.7.6 Use a new [REDACTED] bottle or the bottle [REDACTED]. 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] .
- 8.3.8.2 Pour [REDACTED] 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.

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8.3.9 Preparing saturated KCl solution for production

- 8.3.9.1 Determine the molarity of the solution [REDACTED] This molarity will=X in the following equation:
[REDACTED]
- 8.3.9.2 Pour KCl into [REDACTED] volumetric and dilute to volume with [REDACTED]. 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 [REDACTED] 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 [REDACTED] to fill the bottle approximately half full. Note the weight of the [REDACTED].
- 8.3.11.3 Slowly add the same weight of concentrated sulfuric acid to the bottle. Add the sulfuric acid [REDACTED].
- 8.3.11.4 After the sulfuric acid has been added, [REDACTED]. After the solution has [REDACTED].
- 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].
[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 [REDACTED].
[REDACTED]

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 [REDACTED]

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 [REDACTED]

8.3.13.7 Set up a mixer and let the solution mix for [REDACTED]

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 [REDACTED]
- 8.3.13.8.3 [REDACTED]
- 8.3.13.8.4 [REDACTED]
- 8.3.13.8.5 [REDACTED]
- 8.3.13.8.6 [REDACTED]

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 [REDACTED]

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 [REDACTED]
- 8.3.12.9.4.2 [REDACTED]
- 8.3.12.9.4.3 [REDACTED]

8.3.13.9.5 Calculate the molarity of the solution using the following:

- 8.3.13.10 If the solution is not [REDACTED]
- 8.3.13.11 Pour the cadmium nitrate solution into [REDACTED]

8.3.14 Preparing 3.5% Methocel Solution

8.3.14.1 Check the conductivity [redacted] Do not prepare the methocel solution unless the [redacted] conductivity is [redacted] ohm-cm.

8.3.14.2 Measure [redacted] of [redacted] into [redacted] flask.

8.3.14.3 Use a hot [redacted].

8.3.14.4 Measure [redacted] of [redacted] into another [redacted] flask.

8.3.14.5 Place this flask in [redacted] until [redacted].

8.3.14.6 Weigh out [redacted] of type [redacted] methocel into a weigh boat.

NOTE: Methocel powder must [redacted]. The container should have [redacted]. If the powder does not have [redacted].

8.3.14.7 Remove the flask [redacted] and slowly add the methocel to [redacted] a little bit at a time. Agitate the solution while adding the methocel by [redacted]. Agitate the solution until [redacted]. If a gel forms, stop adding the Methocel until [redacted].

8.3.14.8 Remove the other flask from [redacted] and slowly add [redacted]. If a gel forms, stop adding [redacted].

8.3.14.9 After all the [redacted] 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 [redacted] in a [redacted] volumetric. Dilute [redacted] volumetric to volume with [redacted].

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 [redacted].

8.4.1.3 The phenolphthalein solution should be within [redacted] range. The titration end points are [redacted].

8.4.2 Preparing phenolphthalein indicator for leak tests

8.4.2.1 Dissolve 1g phenolphthalein disodium salt in 100ml of [REDACTED]

8.4.2.2 Dilute 1ml of the solute in 500ml of [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 [REDACTED] Dilute to [REDACTED] with [REDACTED]

8.4.3.2 Methyl orange should be in [REDACTED] range. The titration end points are [REDACTED]

8.4.4 Preparing methyl red indicator

8.4.4.1 Dissolve [REDACTED] methyl red powder in [REDACTED] Dilute to [REDACTED] with [REDACTED]

8.4.4.2 Methyl red indicator should be [REDACTED] pH range. The titration end points are [REDACTED]

8.4.5 Preparing methyl red indicator for production use

8.4.5.1 Dissolve 1g methyl red powder in [REDACTED]

8.4.5.2 Dilute to [REDACTED] with [REDACTED] 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 [REDACTED]

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 [REDACTED]
- 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 [REDACTED]
- 8.4.7.7 Pour the solution into a plastic bottle. Label the bottle with [REDACTED]
[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 [REDACTED] The glassware that is to be used should be [REDACTED]
- 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 [REDACTED] to the flask.
- 8.5.1.5 Dilute to volume with [REDACTED]
- 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]
[REDACTED]

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