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PROCEDURE FOR PREPARING STANDARD REAGENTS, MISCELLANEOUS SOLUTIONS, AND INDICATORS

(mo/yr)

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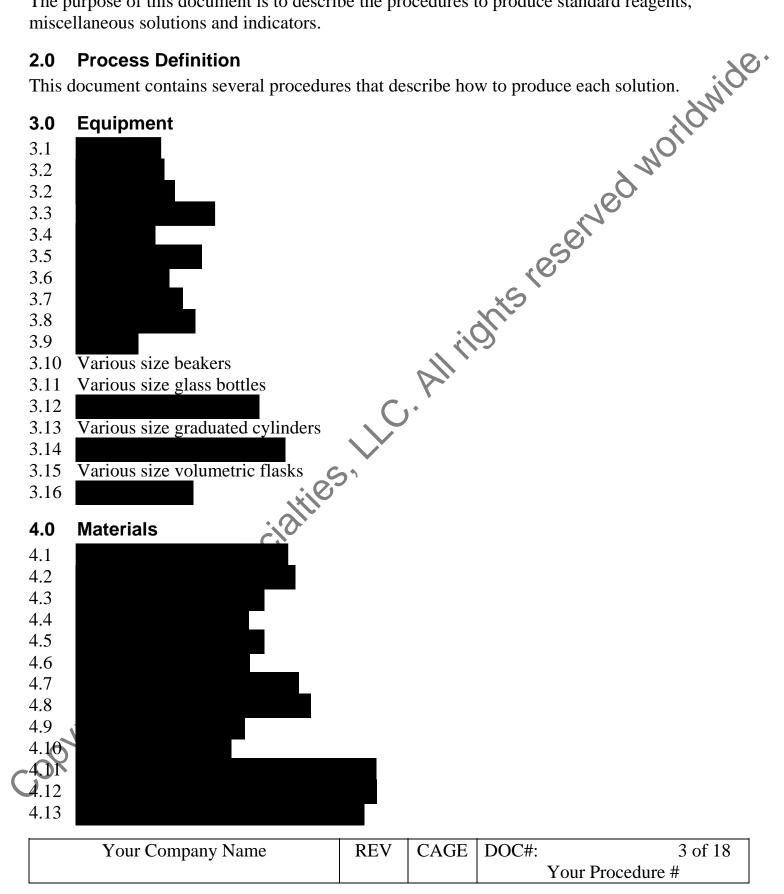
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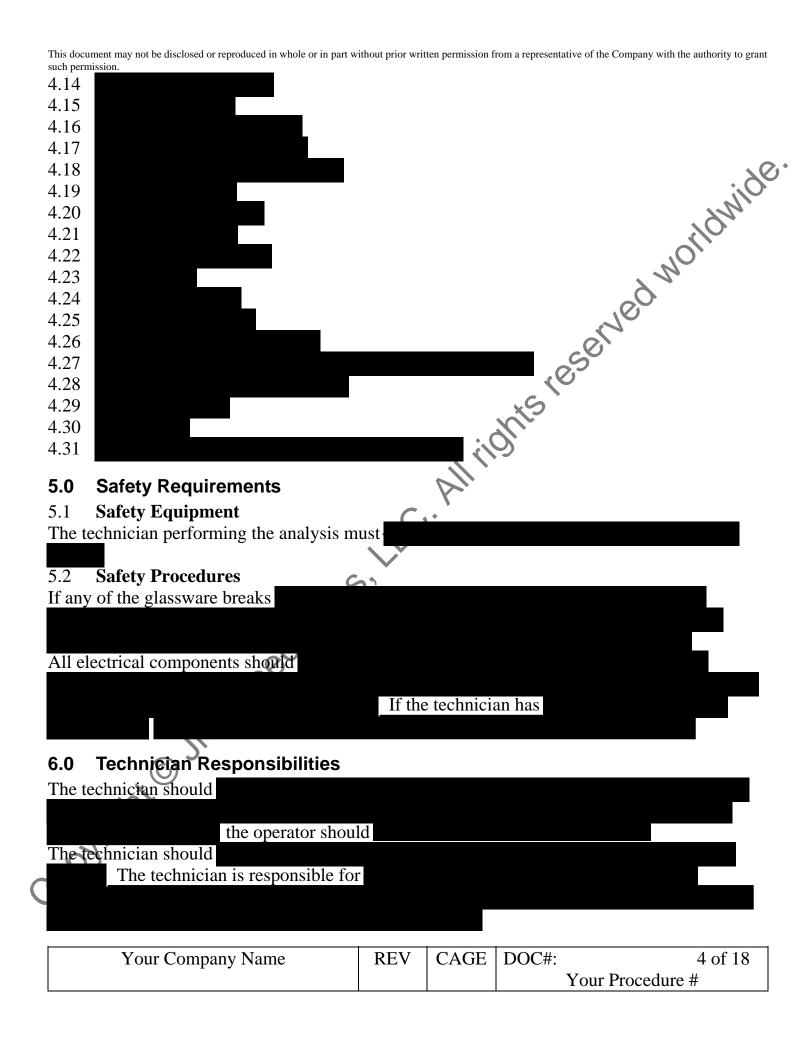
Purpose of Process

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

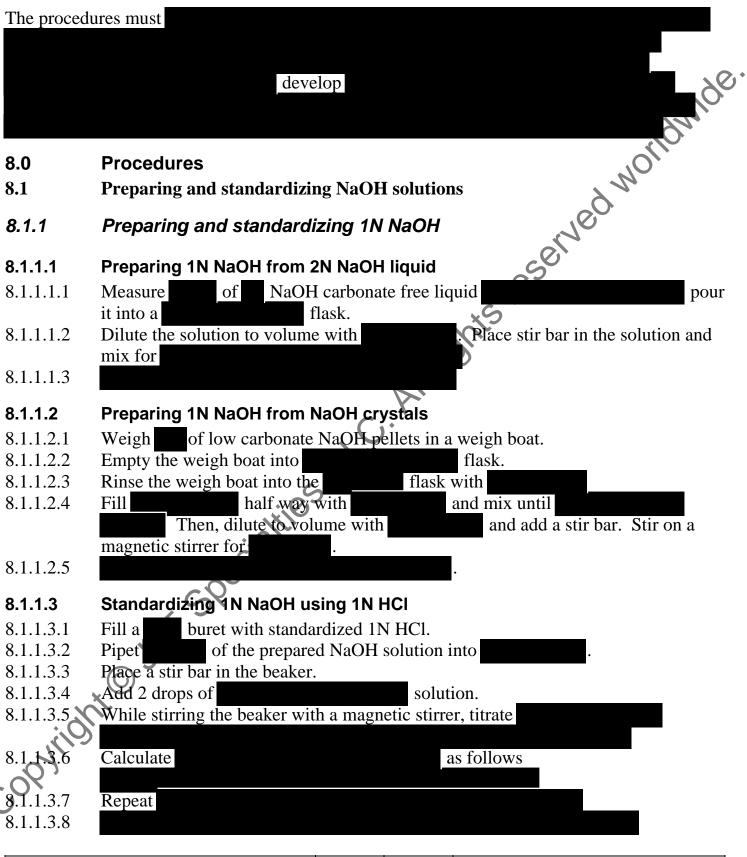
Process Definition 2.0

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

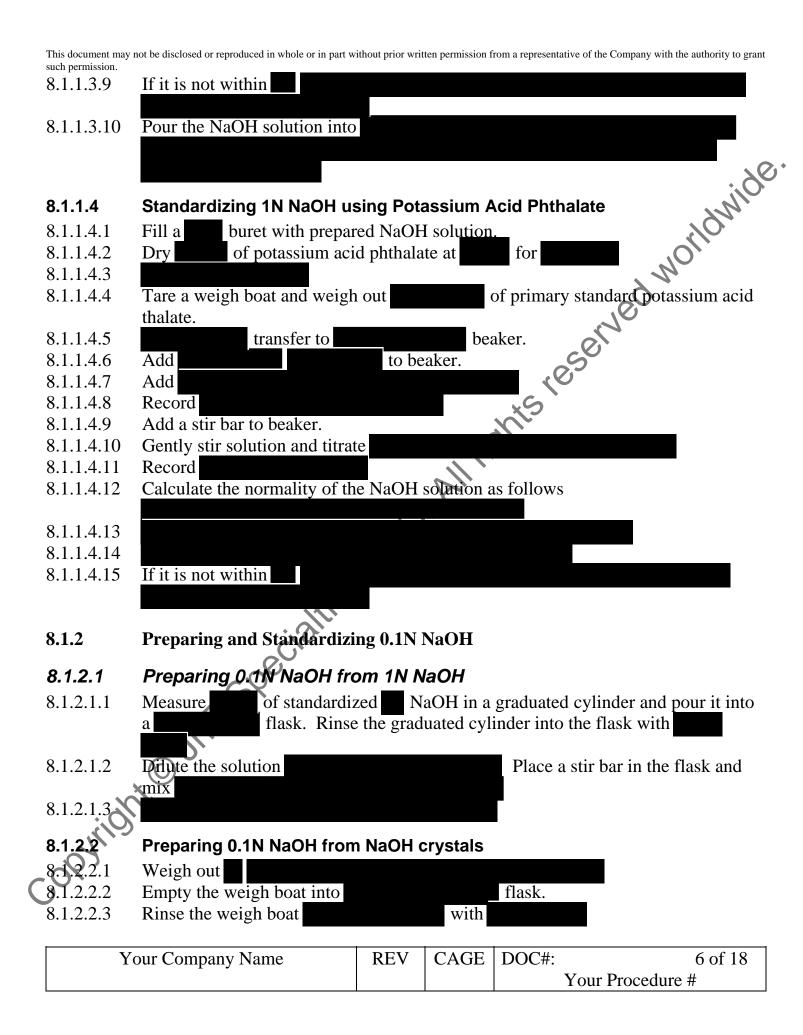




7.0 Process Controls



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8.1.2.2.4	Fill and mix until
	Dilute to volume with and add a stir bar. Stir on
	a magnetic stirrer for
8.1.2.2.5	
8.1.2.3	Standardizing 0.1N NaOH using 0.1N HCI Fill a buret with standardized 0.1N HCI. Pipet of the prepared NaOH solution into beaker. Place stir bar in the beaker. Add
8.1.2.3.1	Fill a buret with standardized 0.1N HCl.
8.1.2.3.2	Pipet of the prepared NaOH solution into beaker.
8.1.2.3.3	Place stir bar in the beaker.
8.1.2.3.4	Add
8.1.2.3.5	While stirring the beaker with a magnetic stirrer,
0.1.0.0.6	
8.1.2.3.6	Calculate the normality of the NaOH solution as follows:
8.1.2.3.7	
8.1.2.3.8	
8.1.2.3.9	If it is not within
0.1.2.3.7	If it is not within
8.1.2.3.10	Pour the NaOH solution into
0404	Standardining 0.4N NoOLI voir a Potogoium Acid Dhtholata
8.1.2.4	Standardizing 0.1N NaOH using Potassium Acid Phthalate
8.1.2.4.1	Fill a buret with the prepared NaOH solution.
8.1.2.4.2	Dry of potassium acid phthalate at
8.1.2.4.3	Weigh of the dried potassium acid phthalate in a weigh boat. Note the weight
8.1.2.4.4	Empty the weigh boat into beaker.
8.1.2.4.5	Rinse the weighing boat into the beaker with Add
	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
8.1.2.4.7	Add
8.1.2.4.8	While continuing to stir the beaker,
94940	Calculate the normality of the NaOII solution as follows:
8.1.2.4.9	Calculate the normality of the NaOH solution as follows:
8.1.2.4.10	
0.1.2.7.1	
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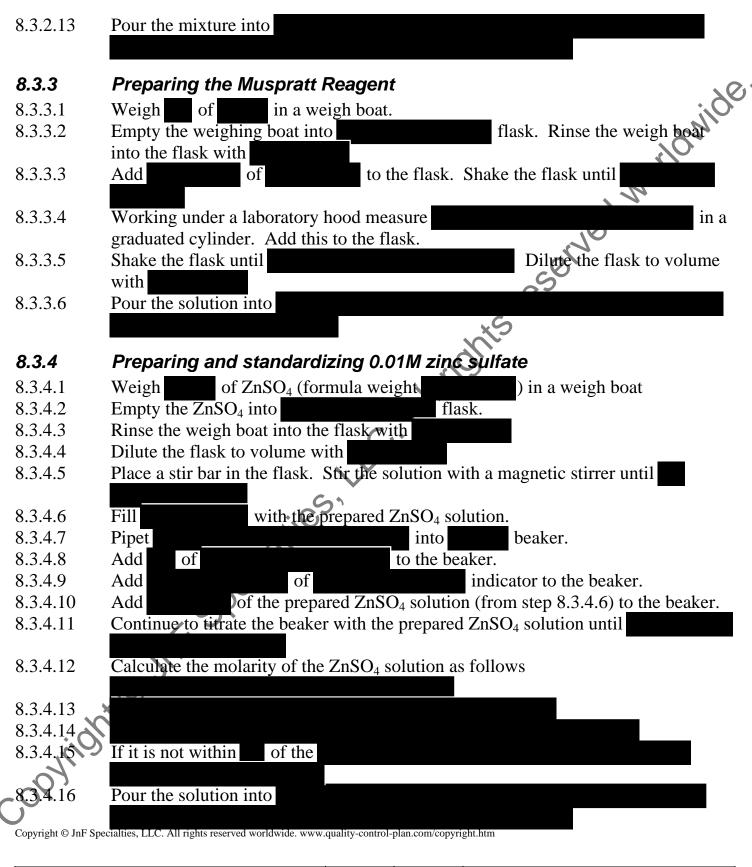
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8.1.2.4.11				
8.1.2.4.12	If it is not within			
	_			_
8.1.2.4.13	Pour the NaOH solution into			
				>0
				flaskdwide
8.1.3	Prep <u>aring</u> and Standard	<u> </u>	_	
8.1.3.1	Pipet of standardized		OH into	
8.1.3.2	Dilute the flask to volume w		,	Stopper the flask and shake it to
	thoroughly mix the NaOH so			7 12
8.1.3.3	Fill buret with standa			
8.1.3.4	Pipet of the prepare	d NaOH	solution i	nto beaker.
8.1.3.5	Place stir bar in the beaker.			
8.1.3.6	Add			3
8.1.3.7	While stirring the beaker			
8.1.3.8	Calculate the normality of th		colution o	A Howar
0.1.3.0	Calculate the normality of th	e NaOn	solution a	is tollows.
8.1.3.9				
8.1.3.10				
8.1.3.11	If it is not within			
0.1.5.11				
8.1.3.12	Pour the NaOH solution into			
8.1.4	Preparing and Standard	izing 0.0	0159N N	aOH
8.1.4.1	Pour of	into	_	flask.
NOTE:	it	is not nec	cessary	
	R			
8.1.4.2		into a gra	duated cy	linder and pour it into the
	volumetric.	1		
8.1.4.3	Dilute the volumetric to volu			Place a stir bar in the flask.
	(01)1	n a magn	etic stirre	er to thoroughly mix the NaOH
0111	solution.	1' 10	131 1101	
8.1.4.4	Fill a buret with standa			
8.1.4.5	Pipet of the prepare	d NaOH	solution i	nto beaker.
8.1.4.6	Place stir bar in the beaker.			
8.1.4. /	Add While stiming the healten with	h o	otic stime	
8.1.4.8	While stirring the beaker wit	ıı a magn	euc stirre	er,
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i		i	1	1 0 0 1 1 0 0 0 0 0 1

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8.1.4.9	Calculate the normality of th	e NaOH	solution a	as follows
0.4.4.10				
8.1.4.10				
8.1.4.11	When the solution is			
0.1.7.11	when the solution is			
8.1.4.12	Pour the NaOH solution into			
				,,0,
				SQ MO.
8.2 Prepa	ring and Standardizing HCl	Solution	S	,e ^O
8.2.1	Preparing 1N HCl and St	tandard	izing 1N	HCl with sodium carbonate
8.2.1.1	Dilute of hydrochloric	acid to	and mix	k thoroughly
8.2.1.2	Weigh accurately		m carbon	ate, Alkalimetric Standard that has
	previously been heated at	for	<u> </u>	*S *
8.2.1.3	Dissolve the sodium carbona	ite in	if	
8.2.1.4	Add			9)
8.2.1.5	Add a stir bar to the solution			
8.2.1.6	While stirring the solution, a	dd	•	until
8.2.1.7	Heat the solution to boiling.		•	Titrate again
0.2.1.7	until			Titute again
8.2.1.8	Repeat this procedure until			
	0			<u> </u>
8.2.1.9				
8.2.1.10	Calculate the normality of the	e HCl so	lution as t	follows
0 0 1 11				
8.2.1.11				
8.2.2	Preparing and standard	izing 0.1	N HCI	
8.2.2.1	Dilute of hydrochloric	acid to	and mi	x thoroughly.
8.2.2.2	Weigh accurately	of sodiu	m carbon	ate, Alkalimetric Standard that has
	previously been heated at	for	<u> </u>	
8.2.2.3	Dissolve the sodium carbona	ite in	if	
8.2.2.4	Add			
8.2.3.5	Add a stir bar to the solution	•		4:1
8 .2.2.6	While stirring the solution,			until
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8.2.2.7	Heat the solution to boiling.				Titrate again
	until				
8.2.2.8	Repeat this procedure until				
8.2.2.9					>
8.2.2.10	Calculate the normality of the	e HCl so	lution as t	follows	Silve
8.2.2.11					Ť
8.2.3	Preparing and standard	izing 0.0	001N HC	I ICI.	91/4
8.2.3.1	Obtain of previously st	•	ed 0.1N F	ICl.	180
8.2.3.2	Transfer to a	flask			
8.2.3.3	Dilute the flask to volume w			S)
8.2.3.4	Obtain of the diluted so			,0 3	
8.2.3.5	Transfer to beaker.			·6)	
8.2.3.6	Add				
8.2.3.7	Titrate with				
8.2.3.8	Calculate the normality with	the follo	wing equ	ation:	
8.3 Prepa	ring and Standardizing Misc	ellaneou	Solution	ns	
8.3.1	Preparing and standard	izing 0.0	5M EDT	A	
8.3.1.1	Preparing E	TA			
8.3.1.1.1	Dry approximately of E	DTA (dis		It form) at	for hour.
02112	Cool to room temperature in Weigh of the ED				iloui.
8.3.1.1.2		IA III a '	weigh boa		
8.3.1.1.3	Empty the weigh boat into	o fleate wi	th	flask.	
8.3.1.1.4	Rinse the weigh boat into the Fill the flask to volume with		ull		
8.3.1.1.5 8.3.1.1.6			th a macr	natia atiman um	411
8.3.1.1.0	Place a stir bar in the flask a	na sur wi	ın a magı	ieuc surrer un	ull
8.3.1.1.7	Standardize the EDTA mixtu	ıre.			
8.3.1.2	Standardizing the EDTA wit				
8.3.1.2.1	Fill a buret with the pro		OTA solu	tion.	
8.3.1.2.2	Weigh				in a weigh boat.
0X,	Note the weight				-
8.3.1.2.3		nto	beake	r. Rinse the w	eigh boat into the
,	beaker with				<i>G</i>
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such permission.	
8.3.1.2.4	Swirl the beaker to
8.3.1.2.5	Cover the beaker with a watch glass. Add
	pipet inserted between
8.3.1.2.6	Swirl the beaker to dissolve
8.3.1.2.7	Rinse
	with Dilute the beaker with to .
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
	being standardized from 8.3.1.2.1.
8.3.1.2.10	Add () and of
8.3.1.2.11	Continue to titrate the solution with the EDTA until
8.3.1.2.12	Calculate the molarity of the EDTA solution as follows
8.3.1.2.13	
8.3.1.2.14	
8.3.1.2.15	If it is not within of the intended molarity,
8.3.1.2.16	Pour the mixture into
8.3.2	Preparing and Standardizing 0.05M Magnesium Sulfate
8.3.2.1	Weigh of a weigh boat.
8.3.2.2	Empty the weigh boat into flask. Rinse the weigh boat into
0.3.2.2	the flask with
8.3.2.3	Dilute the solution to volume with Place a stir bar in the flask and
0.3.2.3	stir with a magnetic stirrer until
8.3.2.4	Fill burette with the prepared 0.05M MgSO ₄ solution.
8.3.2.5	Pipet of standardized solution into beaker.
8.3.2.6	Add of to the beaker.
8.3.2.7	Add approximately of indicator to the beaker.
8.3.2.8	While stirring the beaker with a magnetic stirrer, titrate it with the MgSO ₄ solution
0.2.2.0	(from 8.3.2.4) until . Note the amount of titrant used.
8.3.2.9	Calculate the molarity of the MgSO ₄ solution as follows
\$10.2N	emodiate the initiality of the right of the right
8.3.2.10	
8.3.2.11	
8.3.2.12	If it is not within of the intended molarity,
ر	
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Stir until

Label appropriately.

Solution has shelf life.

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8.3.9	Preparing saturated KCI solution for production
8.3.9.1	Determine the molarity of the solution This molarity
	will=X in the following equation:
8.3.9.2	Pour KCl into volumetric and dilute to volume with
	Stir until Be patientthis 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
8.3.10	Stir until Be patientthis will take time. The flask will be cold to the touch. This is normal. Label appropriately. Shelf life is Preparing pH 11.70 buffer solution
8.3.10.1	Pour 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 meter probe in the beaker.
8.3.10.4	While continuing to stir the beaker, add drops of until
8.3.10.5	Place the buffer solution in a plastic bottle. Label the bottle with
8.3.11	Preparing 50% by weight sulfuric acid solution
8.3.11.1	Place a glass bottle with funnel on a scale. Tare the scale.
8.3.11.2	Add to fill the bottle approximately half full. Note the weight of the
8.3.11.3	Slowly add the same weight of concentrated sulfuric acid to the bottle. Add the
	sulfuric acid
8.3.11.4	After the sulfuric acid has been added, After the solution
00117	has
8.3.11.5	Label the bottle with
8.3.12	Preparing Nochromix
8.3.12.1	Pour one package of Nochromix crystals into
. ~	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

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8.3.12.2.4	Save the rest of the crystals f	or future	use.		
8.3.12.3	Place cap loosely on bottle.				
8.3.12.4	Let stand overnight.				
NOTE: Do	not				
8.3.12.5	Label the bottle.			•	7(
8.3.12.6	For best results, use				
8.3.12.7				ed, it can be regenerated with	12.
8.3.13	Preparing 2Molar cadmi	um nitra	ite	20	
8.3.13.1	Clean a 5 gallon graduated p	re-weigh	ed bucket	t equipped with a spigot and lice	d.
8.3.13.2	Have production weight		of cadmin	um nitrate into the bucket.	
8.3.13.3	Get exact weight of cadmiun				
8.3.13.4	Subtract bucket weight to ge	t cadmiui	n nitrate	weight.	
8.3.13.5	Divide cadmium nitrate weig	ght by	to get to	otal volume.	
8.3.13.6	Dilute with				
8.3.13.7	Set up a mixer and let the sol	lution mix	x for		
NOTE: The	e mixing should be done under	r a hood i	n the lab.		
8.3.13.8	Dilute the solution according	to the fo	llowing:		
8.3.13.8.1					
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 wa				
8.3.13.9.2	Set the slit width to for the	cadmiur	n progran	n.	
8.3.13.9.3	Set the wavelength to	ım.			
8.3.13.9.4	Use cadmium standards with	the follo	wing con	centrations:	
8.3.12.9.4.1	•				
8.3.12.9.4.2					
8.3.12.9.4.3					
8.3.13.9.5	Calculate the molarity of the	solution	using the	following:	
1/100					
8.3.13.10	If the solution is not				
8.3.13.11	Pour the cadmium nitrate sol	ution into	O		
ノ					
			<u> </u>	1	
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8.3.14	Preparing 3.5% Methoce	el Solutio	on			
8.3.14.1	Check the conductivity			Do not p	repare the	e methocel
	solution unless the	conductiv	vity is	ohm-o		"MIL
8.3.14.2	Measure of	into		\mathbf{f}	lask.	107
8.3.14.3	Use a hot					Olli
8.3.14.4	Measure of		to anothe	r	•	lask.
8.3.14.5	Place this flask in		ntil			
8.3.14.6	Weigh out of type	methoc	el into a v	weigh boat.	<u> </u>	
	ethocel powder must				The o	container
should have						
8.3.14.7	Remove the flask			add the meth		
	a little bit at a time. Agitate		on while	adding the me		
	. Agitate the solution un				If a g	gel forms,
	stop adding the Methocel un)		
8.3.14.8	Remove the other flask from			owly add		
		If a gel	torms, sto	op adding		
0.2.14.0	A.C. 11.1	G.	* .1 1	1 11		
8.3.14.9	After all the has b	een adde	d, the sol	ution should		
0 2 1 4 10	Sool the Enlangeary of floor tie	مادات ساداد	o ozza 41. od	.; o		
8.3.14.10	Seal the Erlenmeyer flask tig	gnuy with	a synthe	ne rubber stoj	oper or a c	cork that has
8.3.14.11	been covered with Label the flask with					
0.3.14.11	Label the Hask with					
	Citor					
8.4	Preparing indicator solution	nc				
0.4	Treparing indicator solution	1115				
8.4.1	Preparing phenolphthale	ein indic	ator for	process us	e	
NOTE: Ph	enolphthalein can be prepared	<u>l as</u> either	an aqueo	ous or alcohol	solution.	
8.4.1.1	Agueous solution: Dissolve	of phe	enolphtha	lein diso <u>dium</u>	salt in	in
	a volumetric. Dilute	<u>vo</u> lumetri			<u>. </u>	
8.4.1.2	Alcohol solution: Dissolve	of phe	nolphthal	ein disodium	salt in	of
$(Q)_{i}$	denatured ethyl alcohol in	volum	etric. Di	lute volum	netric to v	olume with
all.			ı		_	
8.4.1.3	The phenolphthalein solution	n should b	e within		range.	The titration
	end points are					
_						
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8.4.7.3	Rinse the weigh boat into the volumetric flask with
8.4.7.4	Pipet 1 of buffer into the flask.
8.4.7.5	Dilute the flask to volume with
8.4.7.6	Place a stir bar in the flask. Stir with a magnetic stirrer for
8.4.7.7	Pour the solution into a plastic bottle. Label the bottle with
8.4.7.8	Store the indicator in Discard the indicator after
<i>8.5</i>	Preparing Standards for the AA spectrometer
NOTE: The	is recipe is not used when analyzing the production solutions. The technician
	use this recipe for the environmental sample analysis.
8.5.1 Prepa	ring stock standard mix
8.5.1.1	Make a batch of fresh The glassware that is to be used should be
8.5.1.2	Clean a bottle, and the necessary
	number of the thoroughly. The number of is dependent upon the
0 - 1 0	number of elements that will be mixed.
8.5.1.3	Pipet of each element into flask:
	C).
8.5.1.4	Add of concentrated to the flask.
8.5.1.5	Dilute to volume with
8.5.1.6	Shake the flask to
8.5.1.7	Pour a small amount of the standard just made into the plastic bottle.
0.3.1.7	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 standard mix into
	(C)
,	X.
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