

# REDACTED

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## DETERMINE HYDROXIDE AND CARBONATE CONTENT IN POTASSIUM HYDROXIDE SOLUTION

(mo/yr)

Revisions		Rev:	
Letter	E.O. Number	Description	Date
Used On	Contract#:	<b>Your Company Name</b>	
Prepared By:			
Your Dept:			
Your Dept:		<b>LABORATORY PROCEDURE</b>	
Your Dept:		Your Procedure #	
Your Dept:		Size: <b>A</b>	CAGE: <input type="text"/>
		Your Form# (mo/yr)	1 of 1

Your Logo

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## 1.0 Purpose of Process

This procedure is used to test KOH samples from production and 45% KOH samples from barrels as a receiving/inspection (R&I) operation.

## 2.0 Process Definition

The percent hydroxide concentration and carbonate content of potassium hydroxide and sodium hydroxide solutions can be determined by titrating a solution sample with standard acid. A two step titration is performed on the sample. At the end point of the first titration, the hydroxide is neutralized and the carbonate is converted to bicarbonate. At the end point of the second titration, the bicarbonate is converted to [REDACTED]. The volumes of the standard acids used in the titrations are used to [REDACTED]

## 3.0 Equipment

3.1 Automatic burettes

3.2 [REDACTED]

3.3 Beakers, various sizes

3.4 [REDACTED]

3.5 Disposable pipettor tips

3.6 [REDACTED]

3.7 Glass pipettes

3.8 [REDACTED]

3.9 Stir bars

3.10 [REDACTED]

3.11 Volumetric flasks

## 4.0 Materials

4.1 [REDACTED]

4.2 [REDACTED]

4.3 [REDACTED]

4.4 KOH sample

4.5 [REDACTED]

4.6 [REDACTED]

4.7 [REDACTED]

## 5.0 Preparations

The following solutions may be prepared in advance:

1N HCl, 0.1N HCl, decarbonated water, phenolphthalein indicator and methyl orange indicator.

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## 6.0 Reports

6.1 Form # \_\_\_\_\_, KOH and Carbonate Concentration in Production KOH Samples and Form # \_\_\_\_\_, Receiving/Inspection (R&I) KOH Samples

## 7.0 Safety Requirements

### 7.1 Safety Equipment

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

### 7.2 Safety Precautions

If any of the glassware breaks during the procedure the technician should \_\_\_\_\_

\_\_\_\_\_ All electrical components should \_\_\_\_\_

\_\_\_\_\_ If the technician has any trouble or questions he/she should \_\_\_\_\_

## 8.0 Operator Responsibilities

The technician should understand how to operate all measuring devices used in the procedure. The technician should understand the content of pages 572 through 575 from the 8th edition of the book Reagent Chemicals by the American Chemical Society Specifications. The technician should \_\_\_\_\_

\_\_\_\_\_ The technician should \_\_\_\_\_ The technicians will be responsible for \_\_\_\_\_

\_\_\_\_\_ The technician is responsible for \_\_\_\_\_ The technician performing the analysis should \_\_\_\_\_

## 9.0 Process Controls

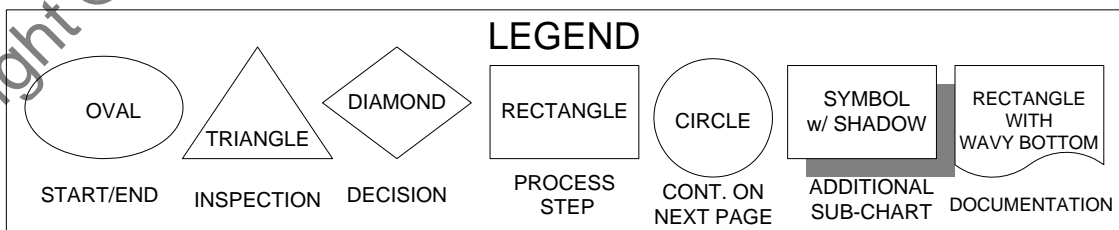
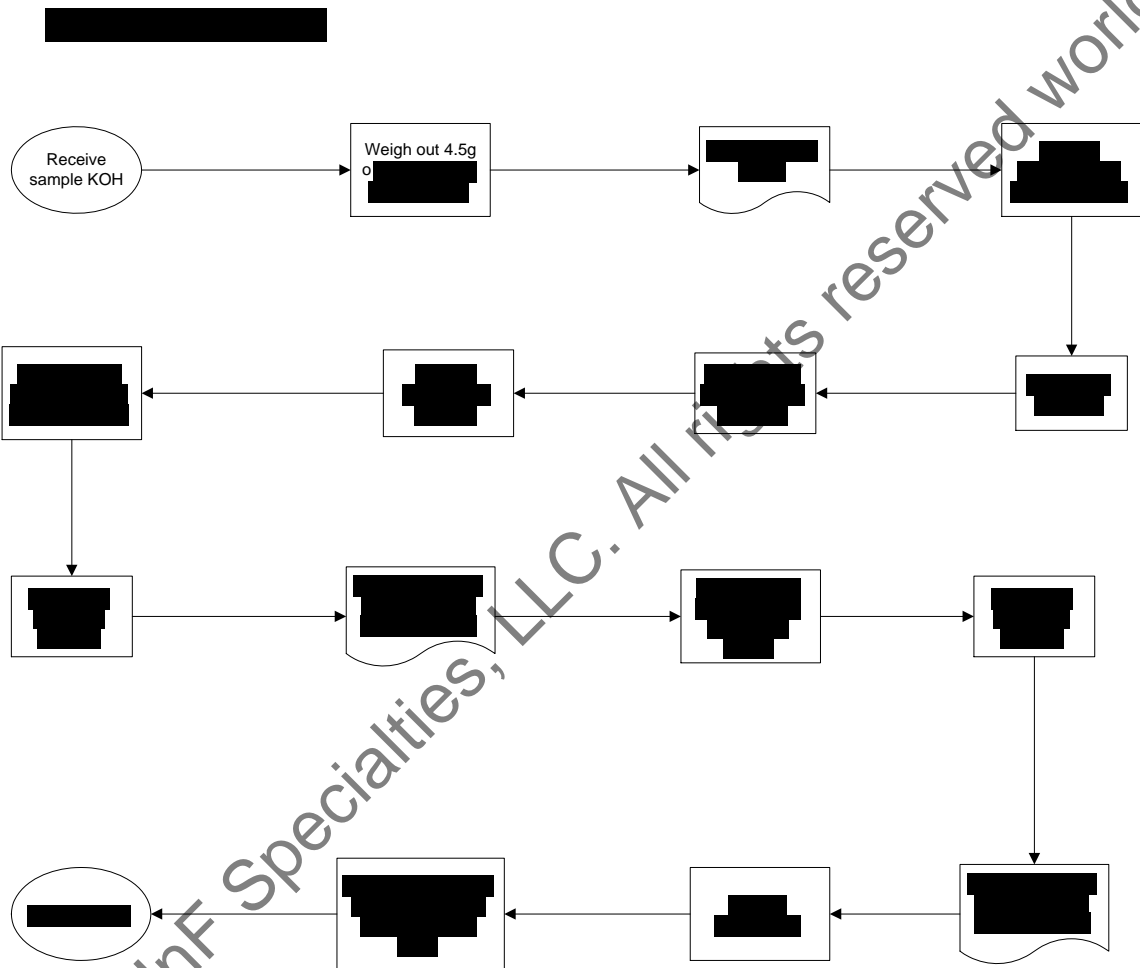
The analysis should be performed according to the procedure described herein. Any changes to the original document should be approved through the lab supervisor and sent through the signature process to maintain configuration control. All of the required data should \_\_\_\_\_

\_\_\_\_\_

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## 10.0 Flowcharts

# PROCEDURE TO DETERMINE HYDROXIDE AND CARBONATE CONTENT IN POTASSIUM HYDROXIDE FLOW CHART





## 11.0 Procedures

**NOTE:** The following procedure is used to determine the hydroxide and carbonate content in KOH solutions from production. This part of the procedure is not to be used for receiving and inspection of KOH solutions. See the Lab Supervisor for direction when a production sample is not large enough to be analyzed.

11.1 Decarbonate Type I water.

11.1.1 [REDACTED]

11.1.2 [REDACTED]

11.1.3 [REDACTED]

11.1.4 [REDACTED]

11.1.5 [REDACTED]

**NOTE:** Special care must be taken in sampling to obtain a representative sample and to avoid absorption of water and carbon dioxide. Each bottle turned into the lab will have [REDACTED]

[REDACTED] For example, the first sample from bottle 1 will be completely analyzed before the second sample will be removed.

11.2 Receive sample KOH.

11.3 Obtain [REDACTED]

11.4 Record sample KOH weight to nearest 0.0001g on Form # [REDACTED]

11.5 Immediately dilute to approximately 150ml in the beaker with decarbonated Type I water.

11.6 [REDACTED]

11.7 [REDACTED]

11.8 Remove the solution from the heat stirrer.

11.9 Add 1-2 drops of phenolphthalein indicator to the beaker.

11.10 Titrate with [REDACTED]

11.11 Record the actual normality of the acid and the volume of acid used to reach the endpoint as V1 on Form # [REDACTED]

11.12 Add 1-2 drops of methyl orange indicator to the beaker.

11.13 Titrate with [REDACTED]

11.14 Record the actual normality of the acid and the volume of acid used to reach the second endpoint as V2 on Form # [REDACTED]

11.15 Perform calculations as shown in Appendix A.

**NOTE:** The procedure should be repeated to ensure there are [REDACTED]

11.16 Report results to Operator requesting the analysis.

**NOTE:** If the results are not what the operator is expecting, a new Sample Submittal Form must be completed for additional analyses.

**NOTE:** The following procedure is to be used to determine the hydroxide and carbonate content in KOH solutions when requested by receiving and inspection (R&I). (Method adapted

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from 8th Edition of Reagent Chemicals, ACS Specifications, pgs 572-575.) This part of the procedure is not to be used for production KOH samples. Two test samples must be analyzed from each sample bottle provided by R&I.

11.17 Decarbonate Type I water.

11.17.1 [REDACTED]

11.17.2 [REDACTED]

11.17.3 [REDACTED]

11.17.4 [REDACTED]

11.17.5 [REDACTED]

**NOTE:** Special care must be taken in sampling to obtain a representative sample and to avoid absorption of water and carbon dioxide. Each sample taken from the barrels will [REDACTED]

[REDACTED] For example, the first sample from barrel 1 will be completely analyzed before the second sample will be removed.

11.18 Prepare barium chloride reagent solution.

11.18.1 Dissolve 120g of barium chloride dihydrate,  $BaCl_2 \cdot 2 H_2O$ , in [REDACTED]

11.18.2 Filter if necessary

11.18.3 Transfer solution to 1000ml volumetric flask

11.18.4 Dilute to volume with [REDACTED]

11.19 Receive or obtain samples.

11.20 Obtain [REDACTED]

11.21 Record sample KOH weight to nearest 0.0001g on Form # [REDACTED]

11.22 Add 750ml of decarbonated Type I water to the Erlenmeyer flask.

11.23 Add a stir bar to the Erlenmeyer flask.

11.24 Set up a nitrogen purge.

11.25 Place Erlenmeyer flask on heat plate/stirrer.

11.26 Gently heat and stir to thoroughly mix the KOH solution for one hour.

11.27 Remove the solution from the heat plate/stirrer.

11.28 Cool solution and maintain the nitrogen purge.

11.29 Once cool, [REDACTED]

11.30 Remove system from nitrogen purge.

11.31 Transfer the solution to [REDACTED]

11.32 Rinse [REDACTED] with [REDACTED]

11.33 Transfer the rinsings to [REDACTED]

11.34 Dilute to volume with [REDACTED]

11.35 With a glass pipette, transfer [REDACTED]

11.36 Transfer [REDACTED]

11.37 With a glass pipette add [REDACTED]

11.38 Mix thoroughly and let solution stand for a few minutes.

11.39 Add 1-2 drops of phenolphthalein indicator to the flask.

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1. Titrate with [REDACTED]
2. Record the actual normality of the acid and the volume of acid used to reach the endpoint as V1 on Form # [REDACTED].
3. Add 1-2 drops of methyl orange indicator to the flask.
4. Titrate with [REDACTED]
5. Record the actual normality of the acid and the volume of acid used to reach the second endpoint as V2 on Form # [REDACTED].
6. Perform calculations as shown in Appendix A.

**NOTE:** Procedure must be repeated to ensure [REDACTED]

11.40 Report results to Operator requesting the analysis.

**NOTE:** If the results are not what the Operator is expecting, a new Sample Submittal Form must be completed for any additional analyses.

## 12.0 Documentation

The form that must be completed for this analysis is Form # [REDACTED]: KOH and Carbonate Concentration in Production KOH Samples and when requested for Receiving/Inspection KOH Samples.

Form # [REDACTED] requires the following:

[REDACTED]

Sample weight, V1, V2, KOH wt %,  $K_2CO_3$  wt %, 1N HCl actual normality and standardization date, 0.1N HCl actual normality and standardization weight

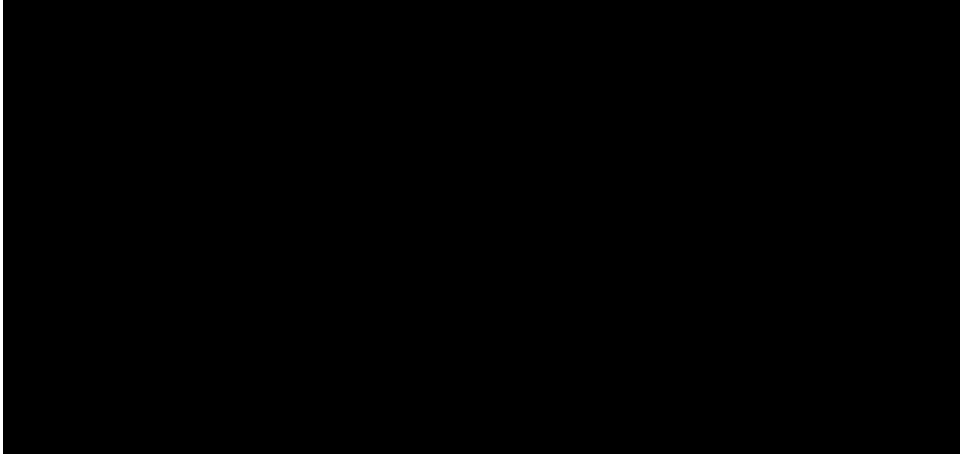
### Appendix A: Sample Calculations

Calculate wt% KOH and wt%  $K_2CO_3$  for Production KOH samples

[REDACTED]

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Calculate wt% KOH and wt% K<sub>2</sub>CO<sub>3</sub> for R&I KOH samples



The equivalents above are taken from the ACS Specifications.

### 13.0 WORKMANSHIP

Adherence to applicable federal, state, local and environmental, health and safety requirements is mandatory.

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