

Activity: Standard Operating Procedures (SOPs)

Student Guide

Scenario:

You are a chemist in a Quality Control (QC) lab. Develop an SOP for preparing a standard acid solution for titration.

Instructions:

Fill in the blanks with the most appropriate words or phrases to complete the SOP.

Tasks to be completed:

- 1) Read the SOP
- 2) Identify all the possible HAZARDS in the tasks listed in the SOP
 - a. Chemical, reactions, products, equipment, storage, disposal, security
- 3) Identify the possible CONTROLS to mitigate or reduce the hazards
 - a. Administrative, operational, engineering, PPE
- 4) Fill in the blanks to complete the SOP

Hazards in this SOP		Controls
Preparation of solutions		
Equipment		
Waste/disposal		
Security		

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Standard Operating Procedure-Titration Reagents

Chemical Safety and Security Training

Scope and Application

The purpose is to provide a standard operating procedure for preparing standardized reagents to be used in titrations. This procedure will insure that all solutions are uniform to accurately and repetitively perform titrations.

Definitions

Deionized (DI) Water: Water that has been through a deionization system to produce water similar to ASTM Type I reagent with 16.7 Mohms resistivity (see ASTM in Reference section).

CO₂-free Water: deionized water that has been boiled to expel the dissolved CO₂ gas.

Specific Chemical(s) of Concern and Hazard Assessment(s)

- | | | |
|---|---------------------------------|----------------|
| 1) Sodium carbonate | Na ₂ CO ₃ | CAS# 297-19-8 |
| a. Hazard(s): Irritant, may be harmful is swallowed _____ | | |
| _____ | | |
| 2) Sulfuric Acid | H ₂ SO ₄ | CAS# 7664-93-9 |
| a. Hazard(s): _____ | | |
| _____ | | |

Hazard Communication and Control

- A.) Administrative Controls: When working with potentially hazardous materials, follow all mandated health, safety, and security procedures. Be prepared in case of an emergency (e.g., _____, _____, and _____). Review all _____ before handling chemicals or performing tasks. For questions consult your _____.
- B.) Engineering Controls: 1) _____
2) _____
3) _____
- C.) Personal Protective equipment
- a. Head (e.g., hard hat, etc.): _____
 - b. Face (e.g., goggles, face shield, etc.): _____
 - c. Hand (e.g., Butyl, Nitrile, etc.): _____
 - d. Body (e.g., lab coat, Butyl apron, silver shield, etc.): _____
 - e. Feet (e.g. Rubber, Butyl, etc.): _____

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Equipment and Supplies

Calibrated Balance and weighing material	Magnetic stirring hot plate and stir bar
Graduated cylinder or pipette	Glassware (volumetric flasks, beakers)
Oven	Sample/Reagent bottles and glassware
Desiccator	pH meter
50mL Buret	

Reagent and Standard Preparation

Preparation of Reagents:

1) Sodium carbonate (CAS# 297-19-8, ex. Sigma S2127)

Weigh out a few grams of sodium carbonate and dry in an oven at 140C. Place dry sodium carbonate in a desiccator to cool. Boil deionized water to expel CO₂ and allow to cool. Add 1.06g of the anhydrous sodium carbonate to a 1000mL volumetric flask and dilute to volume with CO₂-free water. The solution must be prepared with in a few hours of preparation. Store in pyrex bottle with ground glass stopper

2) Sulfuric Acid (CAS# 7664-93-9, ex. Sigma 258105)

Dilute 2.8mL of sulfuric acid to 1000mL with CO₂-free water in a volumetric flask. This solution is approximately 0.1N. Dilute 200mL of 0.1N sulfuric acid to 1000mL with CO₂-free water in a separate volumetric flask.

Standardization of Acid Titrant:

- 1) Add 10mL of 0.02N standard sodium carbonate solution to a 250mL beaker
- 2) Add 90 mL of CO₂-free water and add stir bar
- 3) Using a calibrated pH meter, begin titration with sulfuric acid solution to and end point of pH 4.5. When nearing the end point, slow down the titration rate and be sure that pH equilibrium is reached before adding more titrant
- 4) Calculate the normality of the sulfuric acid solution (See calculations). Should be approximately 0.02N.
- 5) Store in a dated and labeled bottle (see storage handling)

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Sample Handling and Storage

All reagents and solutions should be properly labeled with the full name, date, concentration, lab/room number and preparer. All reagents should be tightly sealed and stored in a cool, dry area. Unless otherwise noted, all reagents should not be stored or used for longer than one year after the date of preparation.

Calibration and Standardization

All equipment and instrumentation should be properly calibrated before using. Be sure to check and note calibration dates before beginning.

Calculations and Equations

Calculating Normality:

$$NV=N'V'$$

Where, N is normality of the standard, V is the volume of the standard (mL), N' is the normality of the titrant, and V' is the volume of the titrant (mL)

Documentation and Data Handling

Balance and pH meter calibration, and Standardized concentrations must be recorded in the appropriate log books (ex: pH Meter Calibration Log and Standard Stock Solution Log).

Emergency Procedures

Contact with eyes – _____

Contact with skin – _____

Ingestion – _____

Inhalation – _____

Spill response- _____

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Waste Management

Security

References and Resources

ASTM. American Society for Testing and Materials. Standard Specifications for Reagent Water. D1193-77 (Reapproved 1983). Annual Book of ASTM Standards, Vol. 11.01. ASTM: Philadelphia, PA, 1991.

MSDS(s)

I have thoroughly read and I understand the Standard Operating Procedure above. I agree to implement all safety procedures and precautions as noted. If I have any questions in the future regarding this procedure I will discuss them with my supervisor.

Date: _____

Employee/Student Name and Signature

Institution/Dept: _____

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I discussed the company Chemical Safety & Security Plan with this employee and answered any questions he had regarding it.

Date: _____

Supervisor Name and Signature