Technical Assistance
HYDROLOGY PROJECT

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Training module # WQ -04

How to prepare standard solutions

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with
HALCROW, TAHAL, CES, ORG & JPS

Table of contents

1 Module context

This module describes procedure and a laboratory exercise for preparation of standard solutions. Modules in which prior training is required to complete this module successfully and other available, related modules in this category are listed in the table below.

While designing a training course, the relationship between this module and the others, would be maintained by keeping them close together in the syllabus and place them in a logical sequence. The actual selection of the topics and the depth of training would, of course, depend on the training needs of the participants, i.e. their knowledge level and skills performance upon the start of the course.

No.	Module title	Code	Objectives
1	*Basic water quality concepts*	WQ -01	• Discuss the common water quality parameters • List important water quality issues
2	*Basic chemistry concepts*	WQ -02	• Convert units from one to another • Discuss the basic concepts of quantitative chemistry • Report analytical results with the correct number of significant digits.
3	*The need for good laboratory practices*	WQ -03	• Apply the adopted standard practices in laboratory operations

2 Module profile

Title	:	How to prepare standard solutions
Target group	:	As per training need
Duration	:	One session of 150 min
Objectives	:	After the training the participants will be able to*:* • Select different types of glassware • Use an analytical balance and maintain it. • Prepare standard solutions.
Key concepts	:	• Volumetric glassware • Analytical balance • Primary and secondary standard solutions
Training methods	:	Explanations, demonstration of equipment and practical exercises.
Training tools required	:	• Volumetric glassware: graduated flask, pipettes, burette, graduated cylinder. • Analytical balance with operation manual. • Required chemicals, dried, cooled and stored in desiccator. • Support of a basic chemical laboratory.
Handouts	:	As provided in this module
Further reading and references	:	• Analytical Chemistry: An introduction, D.A. Skoog and D. M. West/1986. Saunders College Publishing • Chemistry for Environmental Engineering, C.N. Sawyer, P.L. McCarty and C.F. Parkin. McGraw-Hill, 1994

3 Session plan

No	Activities	Time	Tools
1	***Preparations*** • Use your standard ToT checklist • Make sufficient copies of supplier's instruction manual for the available balance/s • Dry and cool beforehand sufficient quantity of potassium dichromate and ferrous ammonium sulphate • Collect and arrange glassware mentioned in the text • Obtain ferroin indicator		
2	***Introduction***: • Describe session context and activities • Ask the participants to read the handout	5 min	OHS
3	***Glassware*** • Demonstrate and explain salient features of different type of glassware and their correct use. • Explain: – Temperature specifications – meniscus and how to read – to 'deliver' and to 'contain' specifications – rinsing of pipettes and burettes with solutions to be transferred – holding of burette stopcock	10 min	Required glassware handout OHS
4	***Analytical balance*** • Explain features of the balance • Ask each participant to read the operation manual for the balance and precautions given in the text. • Explain the use of the available balance (two pan, single pan-mechanical or electronic) with the help of the supplier's instruction manual • Demonstrate how to weigh a light object • Discuss results of the exercise • Explain balance maintenance	30 min	Analytical balance Operation manual handout OHS
5	***Standard solutions*** • Explain – the need for heating and cooling of chemicals before weighing – purity and different grades of chemicals	10 min	Handout OHS
6	***Preparing standard solutions*** • Describe the steps involved • Demonstrate the titration procedure for secondary standard	20 min	Handout

7	***Practice:*** • Divide the class in working groups of two persons each. • Let each group prepare the standard solutions	65 min	
8	***Wrap up:*** • Clarify doubts	10 min	

4 Overhead/flipchart masters

OHS format guidelines

Type of text	Style	Setting
Headings:	OHS-Title	Arial 30-36, Bold with bottom border line (not: underline)
Text:	OHS-lev1 OHS-lev2	Arial 26, Arial 24, with indent maximum two levels only
Case:		Sentence case. Avoid full text in UPPERCASE.
Italics:		Use occasionally and in a consistent way
Listings:	OHS-lev1 OHS-lev1-Numbered	Big bullets. Numbers for definite series of steps. Avoid roman numbers and letters.
Colours:		None, as these get lost in photocopying and some colours do not reproduce at all.
Formulas/ Equations	OHS-Equation	Use of a table will ease alignment over more lines (rows and columns) Use equation editor for advanced formatting only

Required glassware

- Volumetric flask
- Watch glass
- Glass funnel
- Reagent bottle
- Pipette
- Burette
- Graduated cylinder
- Conical flask

Analytical balance

- Capacity: 150 – 200 g
- Least count: 0.1 mg
- Care in operation
  - do not exceed capacity
  - beam arresting mechanism
  - use tongs / forceps
  - protect from hot / corrosive substance
  - cleaning after every use
  - vibration free table

Primary standard: preparation

- Potassium dichromate solution 0•0417 M
  - Use primary grade / analytical reagent grade chemical.
  - Molecular Weight (MW) of $K_2Cr_2O_7$ = 294g.
  - weight of reagent for 1L of 0.0417M solution

$$= 294 \ \frac{g}{\cancel{mole}} \times 0{\bullet}0417 \ \frac{\cancel{mole}}{\cancel{L}} \times \cancel{1\ L} = \mathbf{12{\bullet}2598\ g}$$

Primary standard: procedure

1. Chemical dried at 103-105 ^{o}C for 2 h & cooled in desiccator.
2. Weigh 12•2598 g in a clean, oven dried, cooled & tared watch glass.
3. Carefully transfer weighed chemical to a funnel placed on 1L volumetric flask.
4. Wash the watch glass with a small amount of water into the funnel.
5. Add about 0.5L distilled water, swirl to dissolve & make up to 1L mark.
6. Store the reagent in a reagent bottle (not in volumetric flask).

Secondary standard: preparation

- Ferrous ammonium sulphate solution 0•25 M
  - Use primary grade / analytical reagent grade chemical.
  - Molecular Weight (MW) of $Fe(NH_4)2(SO_4)2.6H_2O$ = 392 g.
  - weight of reagent for 1L of 0.25M solution

$$= 392 \ \frac{g}{\cancel{mole}} \times 0{\bullet}25 \ \frac{\cancel{mole}}{\cancel{L}} \times \cancel{1\ L} = \mathbf{98\ g}$$

Secondary standard: procedure

1. Weigh 98g in a clean, oven dried, cooled & tared watch glass.
2. Carefully transfer weighed chemical to funnel placed on a 1L volumetric flask.
3. Wash watch glass & funnel with distilled water into flask using wash bottle.
4. Add about 0.5 L distilled water & 20 ml concentrated H_2SO_4, cool and dilute to 1L mark.
5. Store reagent in reagent bottle (not in volumetric flask).

Standardisation

- Standardise Ferrous ammonium sulphate solution against standard potassium dichromate solution, 0.0417M as follows:

1. Dilute 10mL standard $K_2Cr_2O_7$ to about 100 mL in a conical flask.
2. Add 30 mL conc. H_2SO_4 and cool.
3. Add (2 to 3 drops) ferroin indicator.
4. Titrate with FAS titrant using 0.10 to 0.15mL
5. Read volume when solution turns red at end point
6. Calculate molarity

Calculate molarity: Ferrous ammonium sulphate

$$\text{Molarity} = \frac{\text{Vol. of } 0.0417\text{M } K_2Cr_2O_7 \text{ solution tritrated, mL}}{\text{Vol. of FAS used in titration, mL}} \times 0.25$$

Exercise

Prepare standard solutions

- Work in groups of two
- Time 65 min

5 Evaluation sheets

6 Handout

Required glassware

- Volumetric flask
- Watch glass
- Glass funnel
- Reagent bottle
- Pipette
- Burette
- Graduated cylinder
- Conical flask

Analytical balance

- Capacity: 150 – 200 g
- Least count: 0.1 mg
- Care in operation
  - do not exceed capacity
  - beam arresting mechanism
  - use tongs / forceps
  - protect from hot / corrosive substance
  - cleaning after every use
  - vibration free table

Primary standard: preparation

- Potassium dichromate solution 0.0417 M
  - Use primary grade / analytical reagent grade chemical.
  - Molecular Weight (MW) of $K_2Cr_2O_7$ = 294g.
  - weight of reagent for 1L of 0.0417M solution

$$= 294 \ \frac{g}{\cancel{mole}} \times 0.0417 \ \frac{\cancel{mole}}{\cancel{L}} \times \cancel{1\ L} = 12.2598\ g$$

Procedure

1. Chemical dried at 103-105 ^{o}C for 2 h & cooled in desiccator.
2. Weigh 12.2598 g in a clean, oven dried, cooled & tared watch glass.
3. Carefully transfer weighed chemical to a funnel placed on 1L volumetric flask.
4. Wash the watch glass with a small amount of water into the funnel.
5. Add about 0.5L distilled water, swirl to dissolve & make up to 1L mark.
6. Store the reagent in a reagent bottle (not in volumetric flask).

Secondary standard: preparation

- Ferrous ammonium sulphate solution 0.25 M
- Use primary grade / analytical reagent grade chemical.
- Molecular Weight (MW) of $Fe(NH_4)2(SO_4)2.6H_2O$ = 392 g.
- weight of reagent for 1L of 0.25M solution

$$= 392 \ \frac{g}{\cancel{mole}} \times 0.25 \ \frac{\cancel{mole}}{\cancel{L}} \times \cancel{1\ L} = 98\ g$$

Secondary standard: procedure

1. Weigh 98g in a clean, oven dried, cooled & tared watch glass.
2. Carefully transfer weighed chemical to funnel placed on a 1L volumetric flask.
3. Wash watch glass & funnel with distilled water into flask using wash bottle.
4. Add about 0.5 L distilled water & 20 ml concentrated H_2SO_4, cool and dilute to 1L mark.
5. Store reagent in reagent bottle (not in volumetric flask).

Standardisation

- Standardise Ferrous ammonium sulphate solution against standard potassium dichromate solution, 0.0417M as follows:

1. Dilute 10mL standard $K_2Cr_2O_7$ to about 100 mL in a conical flask.
2. Add 30 mL conc. H_2SO_4 and cool.
3. Add (2 to 3 drops) ferroin indicator.
4. Titrate with FAS titrant using 0.10 to 0.15mL
5. Read volume when solution turns red at end point
6. Calculate molarity

Calculate molarity: Ferrous ammonium sulphate

$$\text{Molarity of FAS solution} = \frac{\text{Vol. of 0.0417M } K_2Cr_2O_7 \text{ solution tritrated, mL}}{\text{Vol. of FAS used in titration, mL}} \times 0.25$$

Exercise: Prepare standard solutions

- Work in groups of two
- Time 65 min

Add copy of Main text in chapter 8, for all participants.

7 Additional handout

These handouts are distributed during delivery and contain test questions, answers to questions, special worksheets, optional information, and other matters you would not like to be seen in the regular handouts.

It is a good practice to pre-punch these additional handouts, so the participants can easily insert them in the main handout folder.

8 Main text

How to prepare standard solutions

1. Aim

2. To become familiar with basic glassware and equipment
3. To learn use and care of the chemical balance
4. To prepare standard solutions
   (a) Primary standard 0.0417 M $K_2Cr_2O_7$
   (b) Secondary standard ferrous ammonium sulphate (FAS), approximately 0.25 M

2. Basic glassware

Volumetric or graduated flask. Pear shaped flat bottom vessel with a long neck. A line etched around the neck indicates the specified volume it contains when filled to the mark. Used for preparation of standard solutions and exact dilutions.

Pipettes. There are two kinds: (1) those which have one mark and deliver a small constant volume; (2) those in which the stem is graduated and deliver various small volumes at the user's discretion. Used to transfer exact volumes.

Burette. Long cylindrical tube of uniform bore throughout the graduated length, terminating at the lower end in a stopcock. Used to add increasing amounts of solutions in exact volumes as in titration.

Graduated cylinder. Graduated cylindrical tubes used to measure and transfer liquid volumes where a high level of accuracy is not required.

3. Analytical balance

Used for weighing chemicals for the preparation of solutions. Common balances have a weighing capacity of 150 and 200 g and can read down to 0.1 mg.

The maximum weighing capacity should not be exceeded. Be certain that the arresting mechanism of the beam is engaged whenever the loading of the balance is being changed and when the balance is not in use. Tongs or forceps should be used to place or remove articles from the pans. Never weigh hot or corrosive substance that is likely to damage the balance. Clean the balance after every use.

4. Glassware required for practical exercise

Volumetric flask, watch glass, glass funnel, reagent bottle, pipette, burette, graduated cylinder, conical flask.

5. Preparation of primary standard, potassium dichromate solution, 0.0417M

1. Use primary grade or analytical reagent grade chemical for the preparation of the standard solution. The chemical should be dried at 103-105 ^{o}C for 2 h and cooled in a desiccator.
2. Molecular Weight (MW) of $K_2Cr_2O_7$ = 294g. Therefore weight of reagent for 1L of 0.0417M solution = 294g/mole x 0.0417 mole/L x 1L = 12.2598 g.
3. Weigh the above amount into a clean, oven dried, cooled and tared watch glass.
4. Carefully transfer the weighed chemical to a funnel placed on a 1L volumetric flask.
5. Wash the watch glass with a small amount of water into the funnel.
6. Add about 0.5L distilled water, swirl to dissolve and make up to 1L mark.

7. Store the reagent in a reagent bottle (not in the volumetric flask).

6. Preparation of secondary standard, ferrous ammonium sulphate, approximately 0.25M

1. Molecular Weight (MW) of Fe(NH4)2(SO4)2.6H2O = 392 g. Therefore weight of reagent for 1L of 0.25M solution = 392 g/mole x 0.25 mole/L x 1L = 98 g.
2. Weigh the above amount into a clean, oven dried, cooled and tared watch glass.
3. Carefully transfer the weighed chemical to a funnel placed on a 1L volumetric flask.
4. Wash the watch glass and the funnel with distilled water into the flask using a wash bottle.
5. Add about 0.5 L distilled water and 20 ml concentrated H2SO4, cool and dilute to 1L mark.
6. Store the reagent in a reagent bottle (not in the volumetric flask).
7. Standardise this solution against standard potassium dichromate solution, 0.0417M as follows:

Standardization

Dilute 10mL standard $K_2Cr_2O_7$ to about 100 mL in a conical flask. Add 30 mL conc. H_2SO_4 and cool. Titrate with FAS titrant using 0.10 to 0.15mL (2 to 3 drops) ferroin indicator. The solution turns red at the end point.

$$\text{Molarity of FAS solution} = \frac{\text{Vol. Of 0.041M } K_2Cr_2O_7 \text{ solution tritrated, mL}}{\text{Vol. of FAS used in titration, mL}} \times 0.25$$