

**27th INTERNATIONAL CHEMISTRY OLYMPIAD
PRACTICAL EXAMINATION****FRIDAY, JULY 14, 1995**

Please read the entire procedure and the Student's Report before beginning the experiment.

WARNING: You must wear your safety goggles in the Laboratory and use the pipette bulbs provided. Should you remove your goggles, or pipette by mouth, you will be given a warning. A second warning will result in a five point penalty. A third warning will result in removal from the laboratory. Removal from the laboratory will result in a zero mark for the practical examination.

NOTES:

- 1) Write your name, student number (see student's No. on the table) and the delegation at the top of each student's report sheet.
- 2) Begin only after the instructor has given a **START** message.
- 3) You will be given 5 hours to finish the whole practical exam, including writing your report.
- 4) All the answers are to be written on the student's report sheets within the blanks provided. Only those answers given on the correct positions will be taken into consideration.
- 5) Write your report with the ballpen provided.
- 6) Use deionized water except for cooling.
- 7) The significant figures should be used properly.

Ar of some elements

H	1.008	S	32.06
C	12.01	K	39.10
N	14.01	Cu	63.54
O	16.00	I	126
Na	22.99		

PRACTICAL PROBLEM I**Identification of Unknown Solutions****Reagents**

H₂SO₄ (conc.)	H₂SO₄ (6 mol·dm⁻³)
HNO₃(conc.)	HNO₃ (6 mol·dm⁻³)
HCl(conc.)	HCl (6 mol·dm⁻³)
Ba(OH)₂(satd.)	NaOH (6 mol·dm⁻³)
BaCl₂(0.5 mol·dm⁻³)	Ba(NO₃)₂(0.5 mol·dm⁻³)

Desk equipments

One test tube holder
five small test tubes

Problem

You are supplied with five different solutions contained in five test tubes labeled as A,B,C,D and E, respectively. The solution, in each test tube, contains one of the following compounds



Identify these solutions.

NOTES:

- (1) You can only select the provided reagents and use a procedure as simple as possible to complete your task. You are getting a mark not only according to the correct identification, but also to the number of steps you have taken.
- (2) You have to carry out the whole analysis by using the provided amount of these unknown solutions. Supplement of them will be available, but it will reduce the mark you obtain.

PRACTICAL PROBLEM II

Preparation of cis-Copper-bis-Glycinate Hydrate [Cu(gly)₂·xH₂O]

Copper(II) amino acidate coordination compounds are monomeric units for synthesizing important biopolymers such as metalloenzymes like ceruloplasmin, on which every living organism depends. In laboratory cis-copper-bisglycinate hydrate can be produced by the reaction of cupric hydroxide with glycine at a temperature of *ca.* 70°C.

Reagents:

CuSO₄·5H₂O(s)

NH₃·H₂O(3 mol·dm⁻³)

glycine(s)

95% ethanol,

acetone

NaOH(2 mol·dm⁻³)

BaCl₂(0.5 mol·dm⁻³)

Desk equipments

beaker 250 cm³ ×4

graduated cylinder 100cm³ ×1

filter flask (shared)

Buchner funnel 60mm ×1

watch glass ×2

dropper ×2

spatula ×1

glass stirring rod ×1

aspirator

thermometer (at least 100° C)

Procedure:

1. Preparation of Cu(OH)₂

(1) Dissolve your pre-weighted sample of CuSO₄·5H₂O (6.0 g) in 40 cm³ of water with a 250 cm³ beaker as a container.

(2) Add slowly 3 mol·dm⁻³ ammonia solution to the CuSO₄ solution, gently stirring, until the precipitate is completely dissolved and the solution is turning blue-violet.

(3) Add 2 mol·dm⁻³ NaOH solution to the above solution until no more precipitate formed.

(4) Filter the precipitate over a Buchner funnel under reduced pressure. Wash the precipitate with water until no SO_4^{2-} ion is detected in the filtrate.

(5) Collect $\text{Cu}(\text{OH})_2$ for the preparation of $\text{Cu}(\text{gly})_2 \cdot x\text{H}_2\text{O}$.

Write the equations for the main chemical reactions having taken place in the above procedure.

2. Preparation of cis-copper-bisglycinate hydrate

(1) Dissolve a pre-weighted sample of glycine (3.6 g) in 130 cm^3 of water and then warm the solution in a hot water bath (70°C). Add the $\text{Cu}(\text{OH})_2$ to the solution, gently stirring until the precipitate is dissolved. Perform a hot filtration and add 10 cm^3 of 95% ethanol.

(2) Cool the solution and then needle-like crystals appear, place it in the ice water bath for 10 min.

(3) Filter the crystals over a Buchner funnel under reduced pressure, wash once with 10 cm^3 of ethanol-water mixing solvent and then twice with 10 cm^3 acetone, squeeze the crystals as dry as possible on the funnel.

(4) Collect the crystals to a watch glass and dry it (consult your supervisor).

(5) Half an hour later weigh the product. Write the mass of product and the percentage of yield on your student's report. Give the expressions for calculation to show how you calculate.

PRACTICAL PROBLEM III

Determination of copper(II) content in $\text{Cu}(\text{gly})_2 \cdot x\text{H}_2\text{O}$

The Cu(II) content in $\text{Cu}(\text{gly})_2 \cdot x\text{H}_2\text{O}$ crystals prepared yourself can be determined by iodometry with starch solution as indicator. Based on the data obtained one can calculate the moles of hydrates of crystals in $\text{Cu}(\text{gly})_2 \cdot x\text{H}_2\text{O}$.

Reagents

Standard KIO_3 (see the label on the bottle to get the accurate concentration)

H_2SO_4 (1.0 mol · dm⁻³) as indicator.

KI (0.6 mol · dm⁻³)

KSCN (2 mol · dm⁻³)

Starch (0.5%)

$\text{Na}_2\text{S}_2\text{O}_3$ (to be standardized)

Desk equipments

buret 50cm³ ×1

pipette 25cm³ ×1

pipette bulb

beakers (dry) 100cm³ ×2

volumetric flask 100cm³ ×1

Erlenmeyer flask 250 cm³ ×3

graduated cylinder 10cm³ ×3, 100cm³ ×1

wash bottle

single pan balance (shared)

hot water bath (shared)

Procedure

1. Standardization of $\text{Na}_2\text{S}_2\text{O}_3$ solution

(1) Transfer 25.00 cm³ of standard KIO_3 solution to an Erlenmeyer flask.

(2) Add 5 cm³ of water, 10 cm³ of KI solution and 5 cm³ of H_2SO_4 (1.0 mol · dm⁻³) to the flask.

(3) Titrate immediately with $\text{Na}_2\text{S}_2\text{O}_3$ solution.

(4) Add 2 cm³ starch solution when the colour of the titrand turns pale yellow.

(5) Continue titrating until the blue color of the solution disappears.

(6) Proceed with step (1)—(5) twice parallelly.

2. Determination of Cu(II) content in $\text{Cu}(\text{gly})_2 \cdot x\text{H}_2\text{O}$

- (1) Weigh 1.0—1.2 g (precision of ± 0.0002 g) of $\text{Cu}(\text{gly})_2 \cdot x\text{H}_2\text{O}$ with a dry 100 cm^3 beaker as the container.**
- (2) Dissolve it with 40 cm^3 of water and 8 cm^3 of H_2SO_4 ($1.0 \text{ mol} \cdot \text{dm}^{-3}$).**
- (3) Transfer the above solution quantitatively to a 100 cm^3 volumetric flask and dilute to the mark.**
- (4) Transfer 25.00 cm^3 of the Cu(II) Solution to an Erlenmeyer flask, add 50 cm^3 of water and 10 cm^3 of KI solution to the flask.**
- (5) Titrate immediately with standardized $\text{Na}_2\text{S}_2\text{O}_3$ solution.**
- (6) Add 2 cm^3 of starch solution and 3 cm^3 of KSCN solution to the flask when the colour of the titrand turns from brown to pale yellow.**
- (7) Titrate continuously until the blue color of the solution disappears.**
- (8) Proceed with steps (4)—(7) twice parallelly.**

Name

Student's No.

Delegation

**Student's Report for Practical
Problem I**

1. Identification of unknown solutions

labels of solutions	A	B	C	D	E
chemical formulae					

2. Equations for chemical reactions taking place in each experimental step

step one (with the first reagent you choose)

step two (with the second reagent you choose)

step three (with the third reagent you choose)

If you proceed with more steps, write the corresponding chemical equations continuously.

Name

Student's No.

Delegation

**Student's Report for Practical
Problem II**

1. Equations for the main chemical reactions having taken place in the preparation of $\text{Cu}(\text{OH})_2$.

2. Mass of $\text{Cu}(\text{gly})_2 \cdot x\text{H}_2\text{O} =$ (g)

Percent yield

The expression for calculation

Name

Student's No.

Delegation

Student's Report for Practical Problem III

1. Standardization of Na₂S₂O₃ Solution

1) The two equations for chemical reactions taking place during the standardization of Na₂S₂O₃.

i

ii.

(2) Volumes of Na₂S₂O₃ Solution

$V_1 =$ cm^3 $V_2 =$ cm^3
 $V_3 =$ cm^3

$V(\text{mean value}) =$ cm^3

(3) Concentration of Na₂S₂O₃ = $\text{mol}\cdot\text{dm}^{-3}$

Expression for the calculation:

2. Determination of Cu(II) in Cu(gly)₂·xH₂O

(1) Chemical equation for the reaction between Cu²⁺ and I⁻

(2) Mass of Cu(gly)₂·xH₂O = g

3) Volumes of Na₂S₂O₃ solution

$V_1 =$ cm^3 $V_2 =$ cm^3 $V_3 =$ cm^3

$V(\text{mean value}) = \quad \text{cm}^3$

(4) Mass % of Cu(II) in $\text{Cu}(\text{gly})_2 \cdot x\text{H}_2\text{O} = \quad \%$
Expression for the calculation:

(5) X Value in $\text{Cu}(\text{gly})_2 \cdot x\text{H}_2\text{O}$

$X = \quad$ **(with precision of 0.01)**

Expression for the calculation:

**Results key for practical problem I
(10 pts)**

1. Identification of unknown solutions (5 pts)

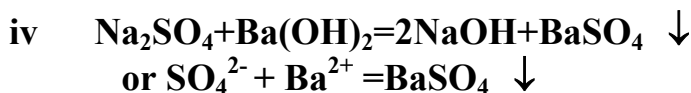
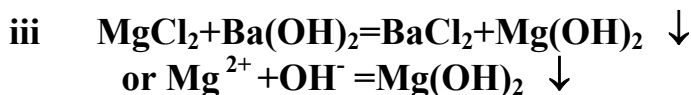
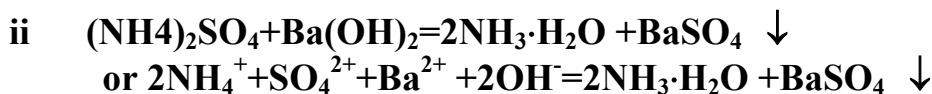
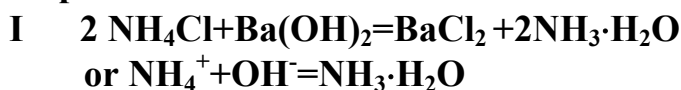
labels of solutions	A	B	C	D	E
chemical formulae					

1 pt for one correct identification

2. Equations for chemical reactions taking place in each experimental step

5 pts

Step one



Step two



5 pts for using Ba(OH)₂ and HCl or Ba(OH)₂ only

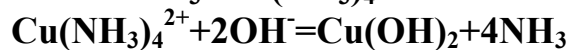
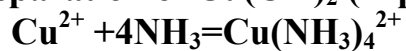
3 pts for using more than these two reagents

for one incorrect equation (-1pt)

**Results key for practical problem II
(12 pts)**

1. Equations for the main chemical reactions having taken place in the

preparation of Cu(OH)₂ (2 pts)



one pt for one correct equation

2. Mass of Cu(gly)₂·xH₂O = (g)

mass ≥ 2.6g (8 pts)

2.6 > mass ≥ 2.2 (6 pts)

2.2 > mass ≥ 1.5 (4 pts)

1.5 > mass ≥ 1.1 (2 pts)

1.0 > mass ≥ 0.5 (1 pt)

dripping (0 pt)

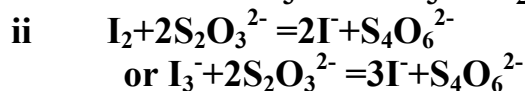
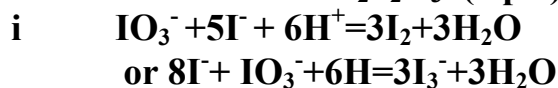
Percent yield

The expression for calculation

Results key for practical problem III (20 pts)

1. Standardization of Na₂S₂O₃ solution (10 pts)

(1) The two equations for chemical reactions taking place during the standardization of Na₂S₂O₃. (2 pts)



1 pt for one correct equation. (incorrect balance -0.5 pt)

(2) Volumes of Na₂S₂O₃ solution (4 pts)

$$V_1 = \quad \text{cm}^3 \quad V_2 = \quad \text{cm}^3$$

$$V_3 = \quad \text{cm}^3$$

$$V(\text{mean value}) = \quad \text{cm}^3$$

1 pt for correct significant figures

3 times of titration (1 pt)

two or one titration (0 pt)

$$\text{precision} \leq 0.04 \text{ cm}^3 \quad (2 \text{ pts})$$

(maximum-minimum)

$$0.04 \text{ cm}^3 < \text{precision} \leq 0.08 \text{ cm}^3 \quad (1 \text{ pt})$$

(3) Concentration of Na₂S₂O₃ mol·dm⁻³ (4 pts)

correct significant figures (1 pt)

$$\text{error} \quad \pm 0.00015 \quad \text{—} \quad \pm 0.00020 \text{ mol}\cdot\text{dm}^{-3} \quad (3 \text{ pts})$$

$$\pm 0.00021 \quad \text{—} \quad \pm 0.00025 \text{ mol}\cdot\text{dm}^{-3} \quad (2 \text{ pts})$$

$$\pm 0.00026 \quad \text{—} \quad \pm 0.00030 \text{ mol}\cdot\text{dm}^{-3} \quad (1 \text{ pt})$$

2. Determination of Cu(II) in Cu(gly)₂·xH₂O (10 pts)

(1) Chemical equation for the reaction between Cu²⁺ and I⁻



(2) Mass of Cu(gly)₂·xH₂O = g (1 pt)

(3) Volumes of Na₂S₂O₃ (4 pts)
correct significant figures (1 pt)

3 times of titration (1 pt)

$$\text{precision} \leq 0.04 \text{ cm}^3 \quad (2 \text{ pts})$$

$$0.04 \text{ cm}^3 < \text{precision} \leq 0.08 \text{ cm}^3 \quad (1 \text{ pt})$$

(4) Mass % of Cu²⁺ in Cu(gly)₂·xH₂O = 27.66% (Theo.) (1 pt)

mass % > 29% or mass % < 26% (0 pt pts)

(5) Calculation of x in Cu(gly)₂·xH₂O (4 pts)

$x = 1.00$ (Theo.)

the ranges of x :

- | | |
|---|----------------|
| $1.00 \pm (0.00 \text{ — } 0.10)$ | (4 pts) |
| $1.00 \pm (0.11 \text{ — } 0.15)$ | (3 pts) |
| $1.00 \pm (0.16 \text{ — } 0.20)$ | (2 pts) |
| $1.00 \pm (0.21 \text{ — } 0.25)$ | (1 pts) |