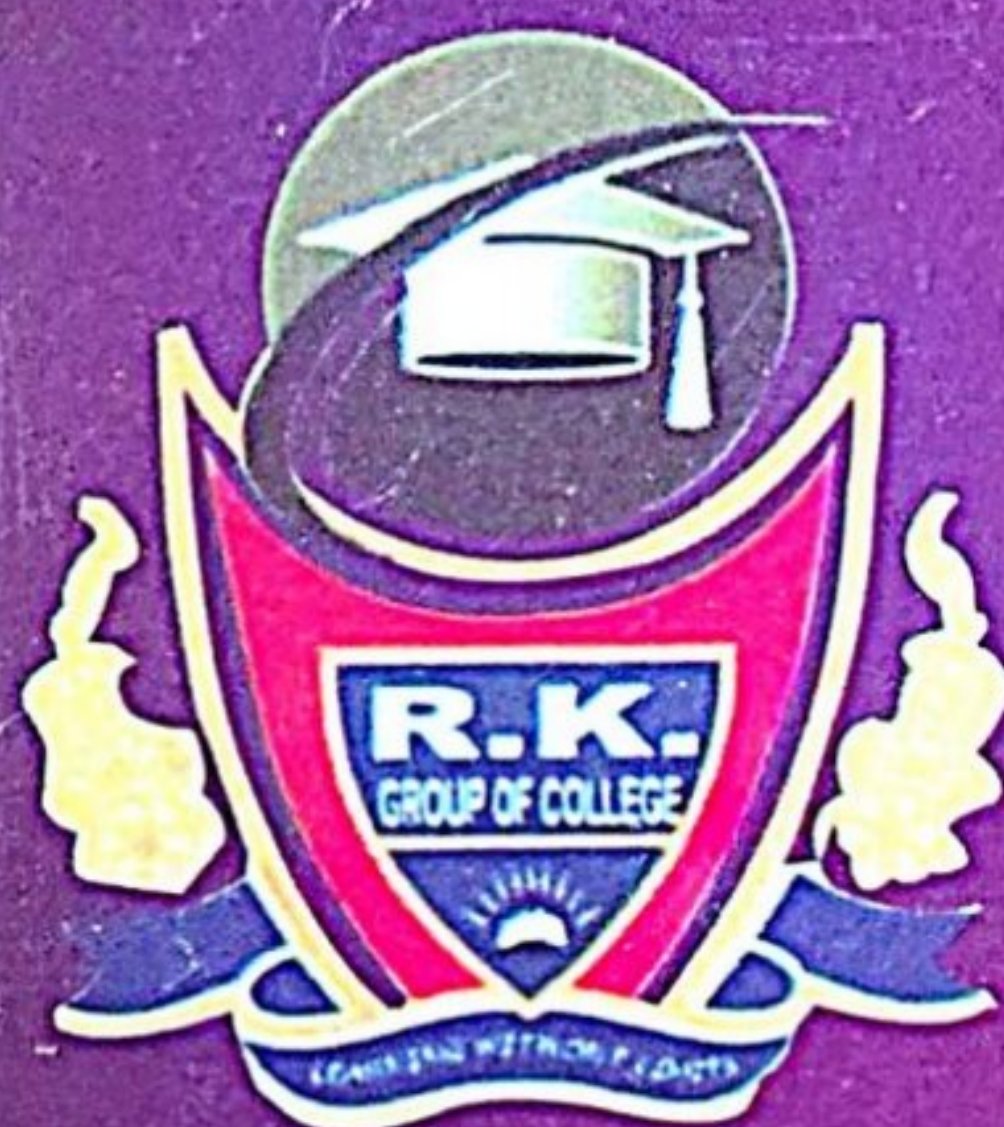
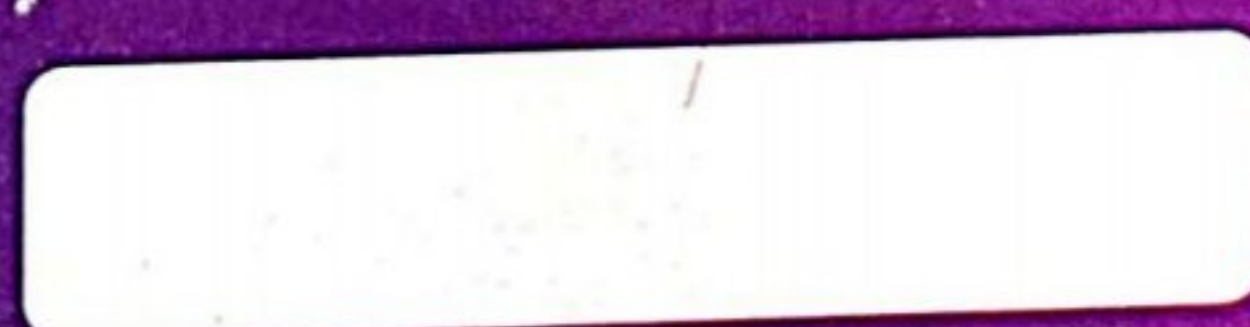




R. K. GROUP OF COLLEGE

BEHIND KALWAR POLICE STATION, KALWAR, JAIPUR (RAJ.)



CERTIFICATE

Name: Radha Yadav

Class: B.Sc. IVth Sem.

Roll No.:

Exam No.:

Institution R.K. Vigyan College

This is certified to be the bonafide work of the student in the

Laboratory during the academic

year 20 /20

No of practicals certified out of in the

subject of Chemistry

Examiner's Signature

Teacher in-charge

Principal

Date :

institution Rubber stamp

university of rajasthan

Chemistry practical Exam

B.Sc. Semester-4th(chemistry)Practical-(a)

duration _4hrs

Max Marks-10*40.

Min marks 4*+16

Ex1 preparation of Tetraamminecopper(II) sulphate (10)

Ex2 synthesis of iodoform from ethanol and acetone (10)

Ex3 Determine of transition temperature of given substance
($\text{MnCl}_2 \cdot 4\text{H}_2\text{O}$). (10)

4. viva-voice 5

5 .Record. 5

university of rajasthan

Chemistry practical Exam

B.Sc. Semester-4 th (chemistry)Practical-(B)

duration _4hrs

Max Marks-10*40.

Min marks 4*+16

**Ex1 preparation of cis and trans-potassium diaquadioxalatochromate (III)
- (10)**

Or

Synthesis of sodium trioxalatoferrate (III)

Ex2 synthesis of Aspirin from salicylic acid (acetylation).-(10)

Or

Synthesis of phthalimide from phthalic anhydride.

Ex3 To construct the phase diagram of two component system like phenol-H₂O System and determine the CST(Critical solution temperature) and composition of the solution at CST. (10)

Ex4 viva-voice. (5)

Ex5 Record. (5)

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Chemistry practical Exam

B.Sc. Semester-4 (chemistry) Practical-(C)

duration _4hrs

Max Marks-10*40.

Min marks 4*+16

Ex1 preparation of bis (glyoxamato) nickel (II). (10)

Ex2. Synthesis of succinic anhydride. (10)

Ex3 To study the effect of solution NaCl and succinic acid etc on the CST(critical solution temperature) of two partially missible liquid (phenol H₂O system and determine the concentration of the solute in the given partially missible liquid system. (10)

Ex4 viva-voice. (5)

Ex5 Record. (5)



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S. No.	Name of Experiment	Page No.	Date of Experiment	Date of Submission	Remarks
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2.	Purification of iodobenzene from acetone.				
3.	To determine transition temp. of $MnCl_2 \cdot 4H_2O$ by Thermometric method.				
4.	Formation of potassium dioxalato diaqua chromate $[K_2Cr(C_2O_4)_2(H_2O)_2]$ [cis]				
5.	Preparation potassium di- oxalato aqua chromate III $K_2[Cr(C_2O_4)_2(H_2O)_2] \cdot 2H_2O$ formation [trans]				
6.	Sodium dioxalatofermate (III) $Na_2(FeC_2O_4)_3$ formation.				
7.	Purification of aspirin from Salicylic acid.				

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S. No.	Name of Experiment	Page No.	Date of Experiment	Date of Submission	Remarks
8.	To determine critical sol ⁿ temp. of phenol water system.				
9.	Nickel dimethylglyoxime complex $[Ni(DMG)_2]$				
10.	Synthesis of phthamide from phthalic anhydride				

Experiment No. 1

Object $\rightarrow$ Tetra-amine copper sulphate $(\text{Cu}(\text{NH}_3)_4\text{SO}_4)$ formed.

Necessary Equipment $\rightarrow$

- (i) Copper Sulphate - 5 gm
- (ii) Ammonia beaker - 10 ml
- (iii) Alcohol - 40 ml
- (iv) dilute H_2SO_4 - 1-2 drops

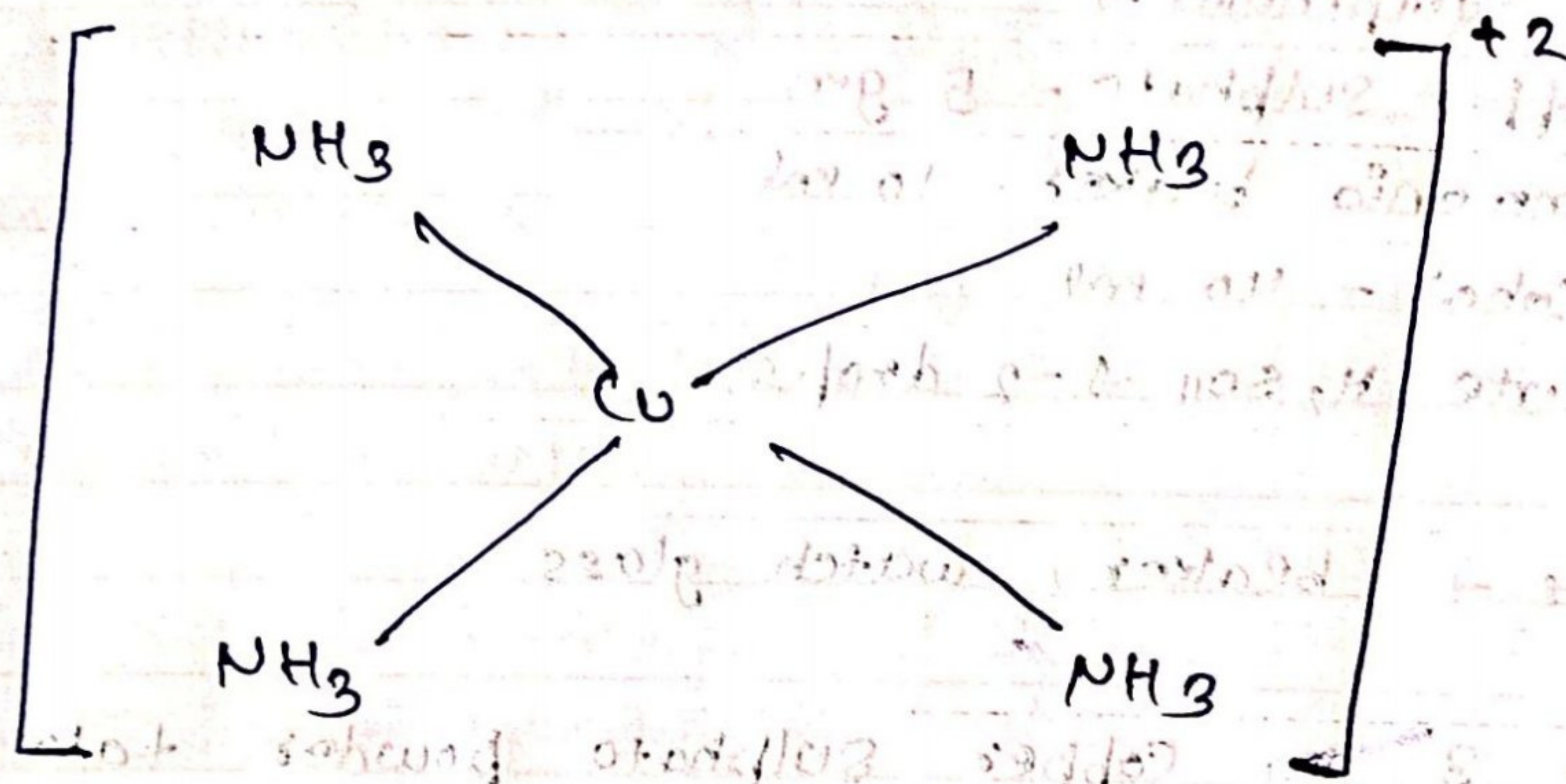
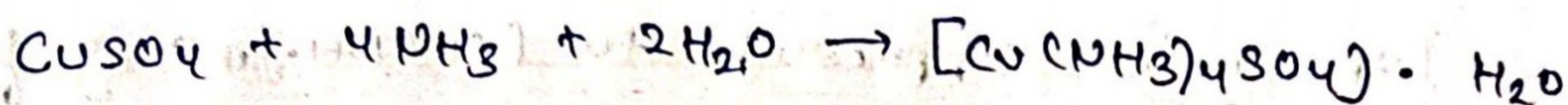
Equipments $\rightarrow$ beaker, watch glass

Method $\rightarrow$ 3 gm Copper Sulphate powder take in beaker and solved in low amount of water and some drops (mixed) of H_2SO_4 .

Now, In this NH_3 solution (i) moved regularly because cupric hydroxide blue the solution becomes dark blue colour and smell of NH_3 comes.

Now, this blue colour solution moved regularly and put in beaker. after this heat for 10-15 minutes on 60°C - 70°C beaker cool and in this needle shaped crystals are separated. and dried in the filter paper and separated.

Result $\rightarrow$ By above method diamine copper sulphate (II) obtained which colour is blue.



tetramine copper(II)

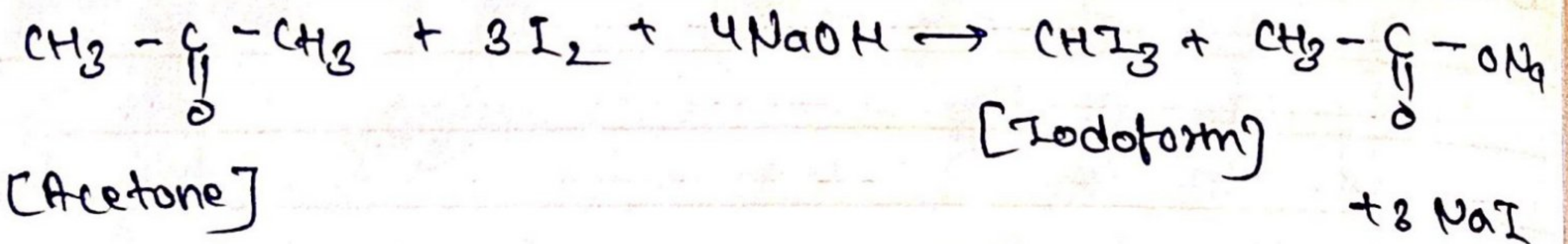
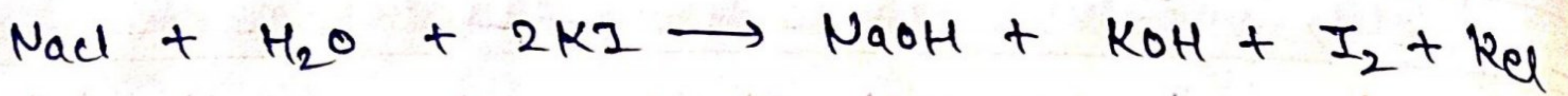
Gain - 4 gm

Precaution - beaker should be clean and dried.

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Teacher's Signature.....



[Acetone]

Experiment No- 2

object $\rightarrow$ Preparation of Iodoform from acetone

Useful Reagent $\rightarrow$ (1) acetone - 20 ml

(2) KI - 6 gm

(3) 5% Na-Hypochloride - solⁿ - 10 ml

Equipment $\rightarrow$ 250 ml flask, keep

Method $\rightarrow$ In a 250 ml flask take 20 ml acetone 6 gm potassium iodide put and it mixed in 80-100 ml water. Now this moved continue and 5% Na-hypochloride - solⁿ of 70 ml on this moved iodoform make yellow crystal in which hypochloride solution mixed and iodoform yellow crystal be ~~sp~~ separated the flask put for 15-20 min. Now iodoform be separated by funnel and washed with water and dry with filter paper this solid yellow substance are crystallization by ethanol and methanol spirit.

Result $\rightarrow$ About reagent make formation of iodoform.

Physical state - solid yellow colour solid crystal obtained

gained - 8-4 gm

m.p - 120-122 °C

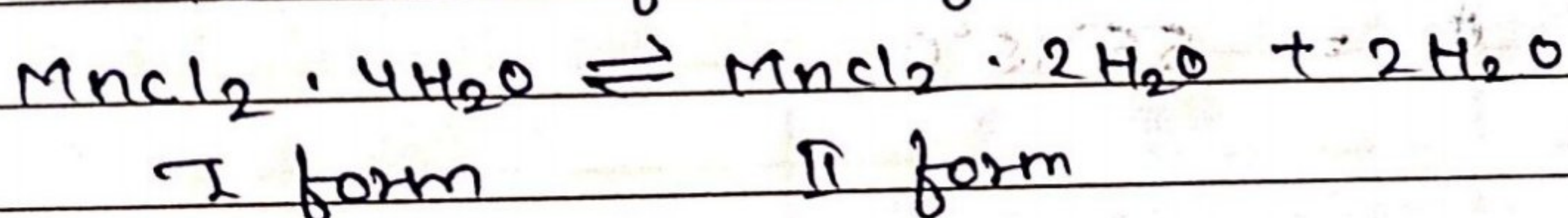
Experiment No- 3

object $\rightarrow$ To determine transition temp. of $\text{MnCl}_2 \cdot 4\text{H}_2\text{O}$ by Thermometric method.

Apparatus $\rightarrow$ Boiling tube, thermometer, two stirrers, beaker.

chemical $\rightarrow \text{MnCl}_2 \cdot 4\text{H}_2\text{O}$ (Manganese chloride)

Principle $\rightarrow$ This procedure is based on the fact that on heating there is absorption of heat and one form changes to other form on cooling there is loss of heat changing the second form to the first form.



On heating manganese chloride there is continuous rise of temperature. At transition point there is no rise in temperature when absorption of heat changes one form (i) to second form (ii) when the change is complete, the temperature start rising on cooling II form changes to form I.

Observation Table

Teacher's Signature.....

Calculation -

Transition temp. = Average of heating & cooling temp.

$$t = \frac{t_1 + t_2}{2}$$

$$t = \frac{55.7 + 54.6}{2}$$

$$t = 55.15^\circ\text{C}$$

Time in minutes

Temperature °C

Heating

Cooling

1	51	60.9
2	51.6	59.9
3	52.4	58.6
4	52.9	57.7
5	53.7	56.7
6	54.3	55.6
7	54.9	55.6
8	55.7	54.6
9	55.7	54.6
10	55.7	54.0
11	56.5	53.6
12	57.3	53.2
13	58.0	53.2
14	59.5	52.7
15	60.2	52.2
16	60.2	51.8
17	60.2	51.4

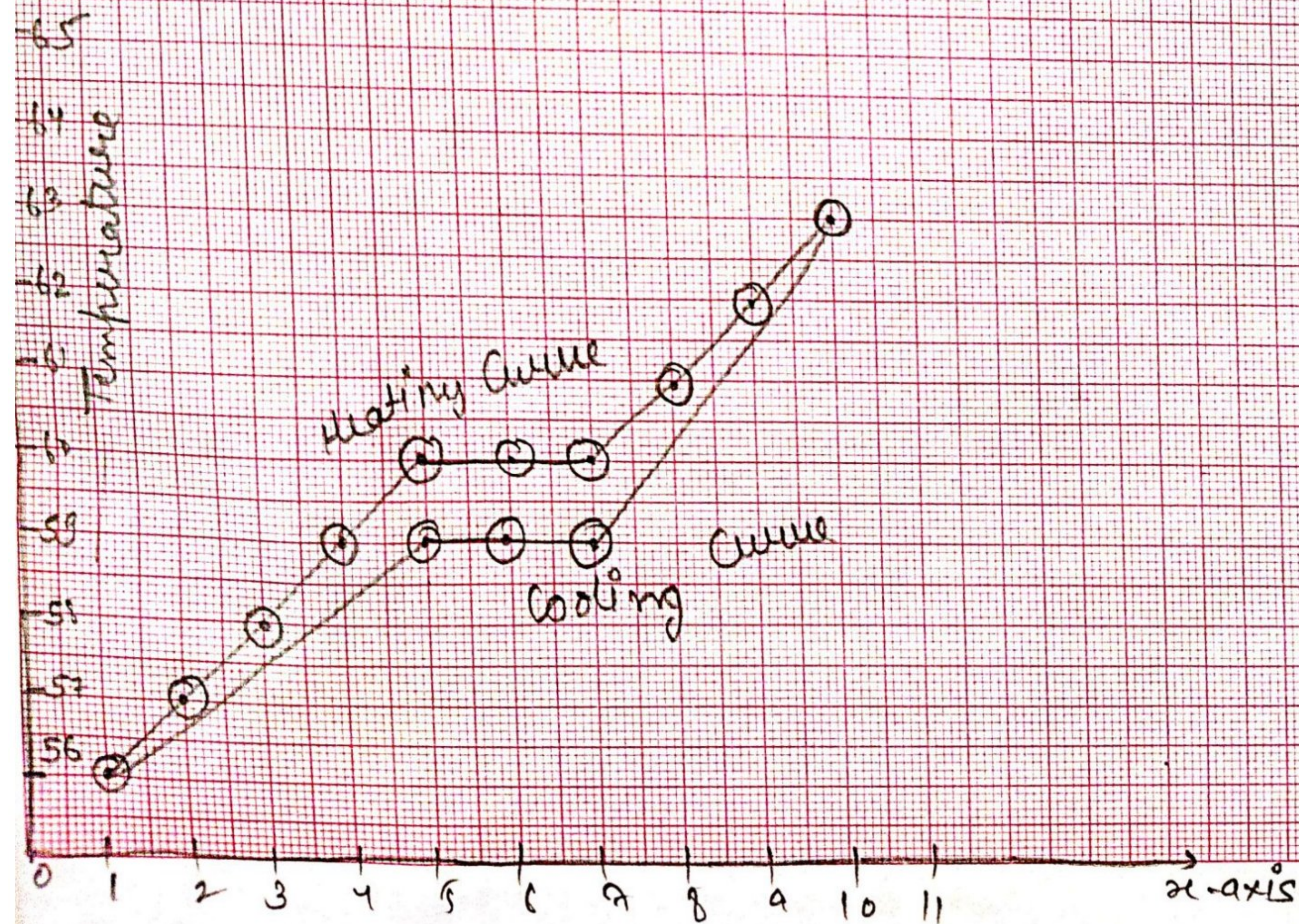
Result - Transition temp. of given $\text{MnCl}_2 \cdot 4\text{H}_2\text{O}$ is
55.15°C.

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Ans. (3)

Scale

x axis - 1 square = 1 unit
y axis - 1 square = 1 unit



Experiment No. - 4

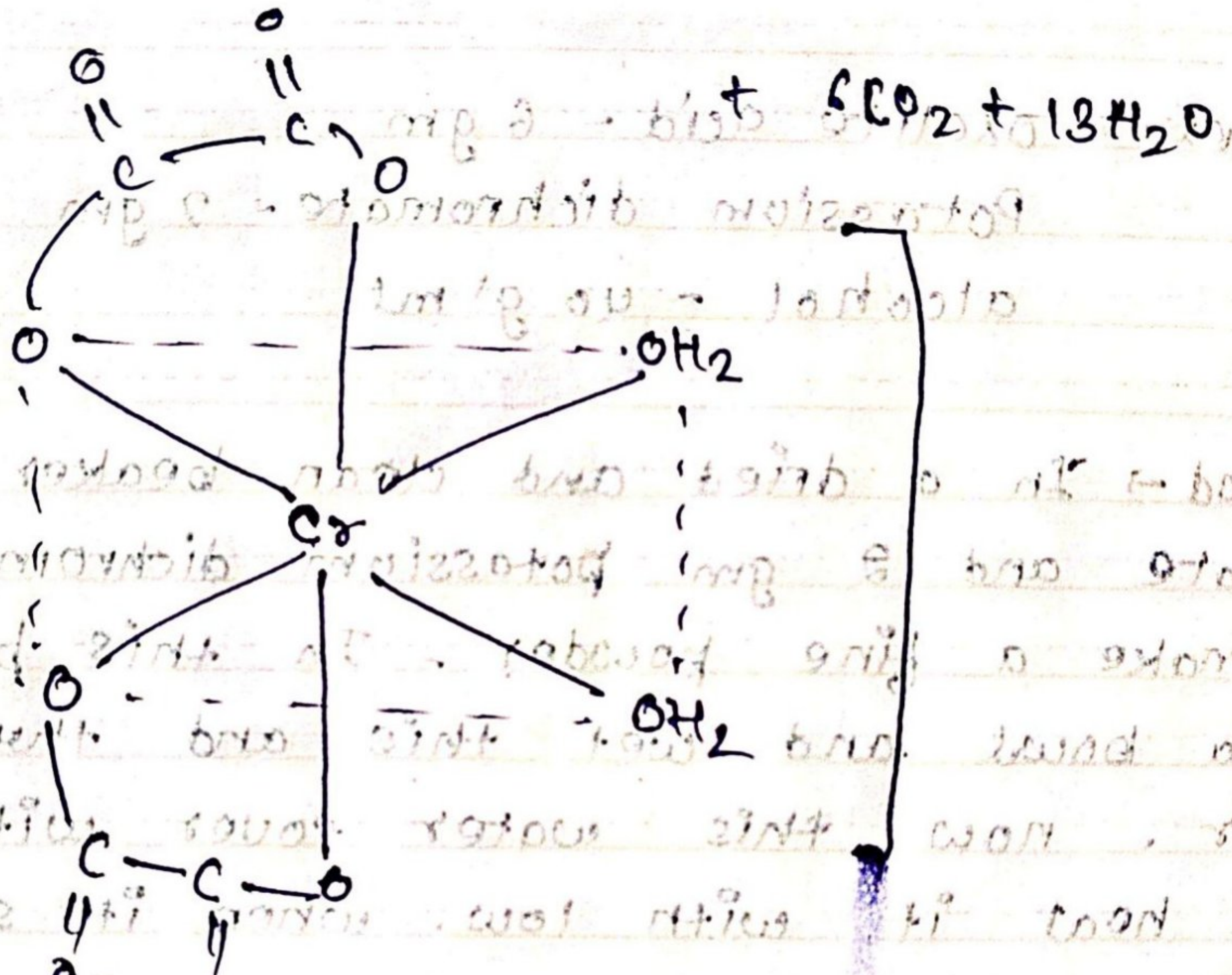
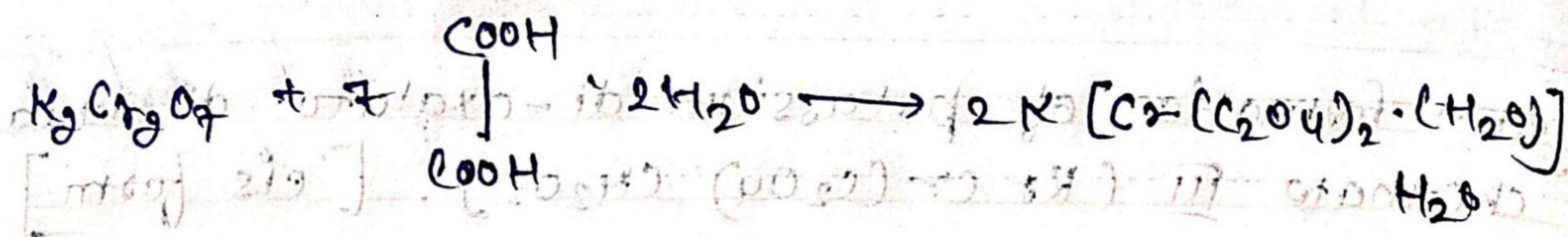
Object $\rightarrow$ formation of potassium di-oxalato di aqua chromate III ($K_2Cr(C_2O_4)_2 \cdot 2H_2O$) [els form]

Reagent $\rightarrow$ oxalite acid - 6 gm
Potassium dichromate - 2 gm
alcohol - 40 g/ml

Method $\rightarrow$ In a dried and clean beaker we take 6 gm oxalate and 2 gm potassium dichromate and crushed to make a fine powder. In this powder take a china bowl and wet this and throw the more water. now this water cover with watch glass and heat it with low. when it substance heat then reaction is fastly and with high smell the CO_2 gas removed. and this mixture converted into liquid dark colour. and this put out to burner and mix 20 ml alcohol the spatula then to move it makes a solid substance if solid substances are not formed then this make liquid filtered and remaining soln put 20 ml alcohol and mixed it in spatula. for this substance make solid and this moved and heat it.

In which crystals are formed this crystals are dark pink black coloured.

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Result $\rightarrow$ By above method the potassium dioxalato diqua chromate (III) obtained.

This is black coloured solⁿ. 8.2 gm produced.

Precaution - Beaker and flask both are dried and clean and heated the solution of china dish slowly by which it does not blast.

In hot solⁿ the alcohol mixed time the burner should be closed by which there is no risk of burn.

Teacher's Signature.....

Experiment No - 5

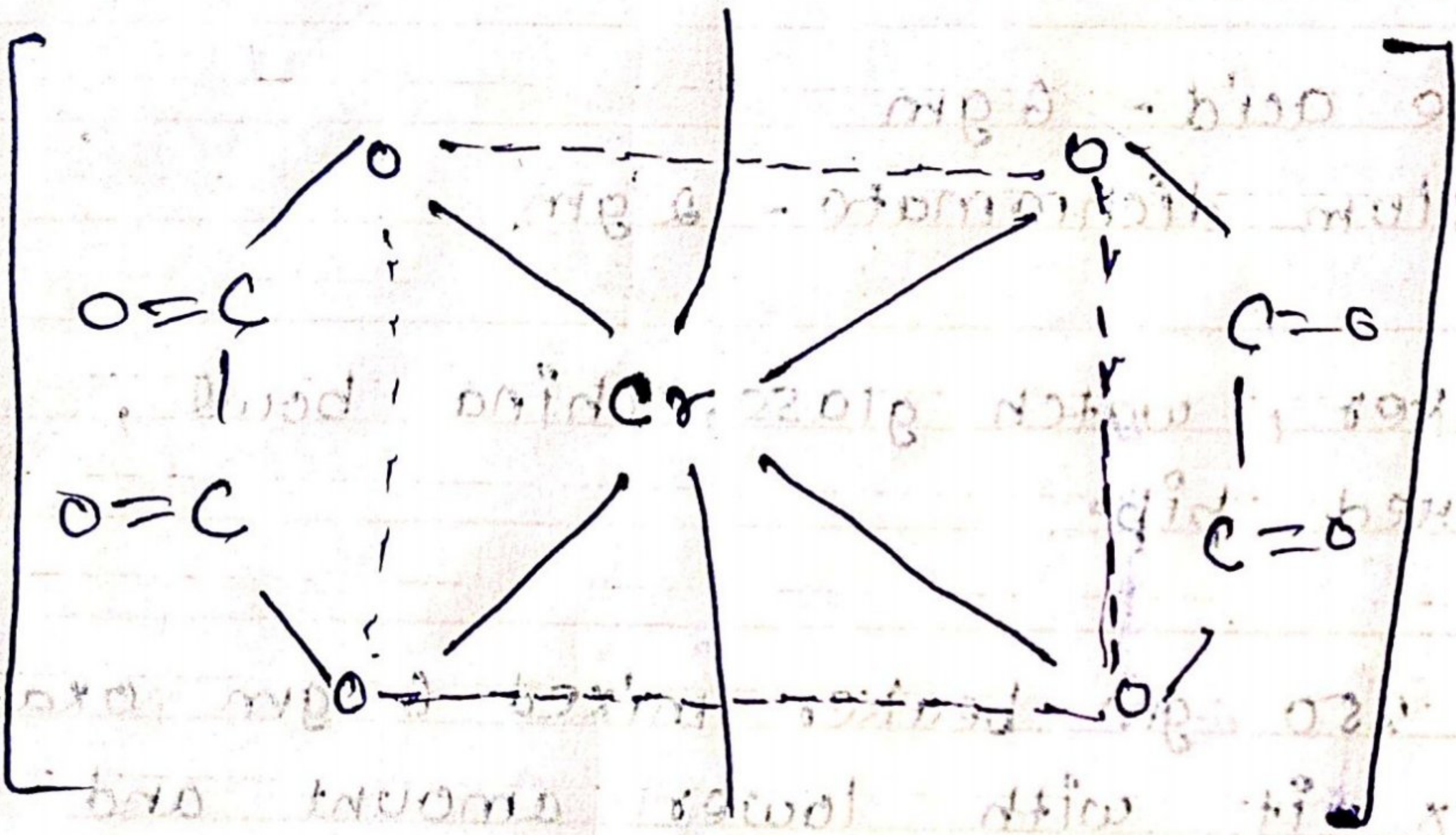
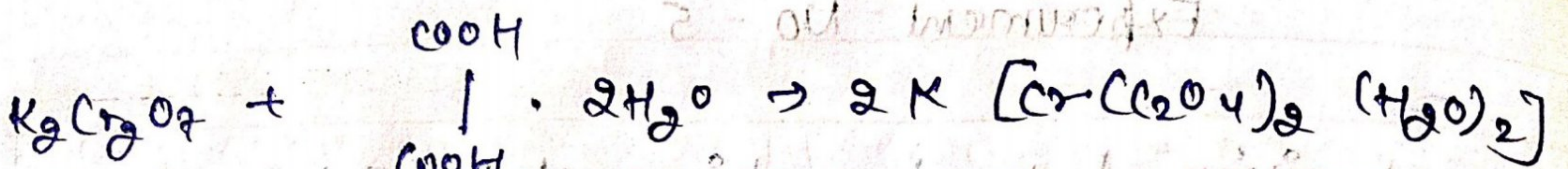
Object → Preparation of potassium di-oxalato aqua chromate (III) $K_2 [Cr(C_2O_4)_2 (H_2O)_2] \cdot 2H_2O$ formation.
[trans form].

Reagent → oxalic acid - 6 gm
potassium dichromate - 2 gm.

Equipment → Beaker, watch glass, china bowl, filtered pipe.

Method → In a 250 gm beaker mixed 6 gm oxalic acid and mix it with lower amount and heat it. and by this method in filtered tube we take 2 gm potassium dichromate put in and heat it now this solⁿ put it in beaker and cover with watch glass because reaction is highly now both solution then mixed and make dark colour liquid. and put it on china bowl then in one third heat it. and cold it on room temperature. and crystals are formed in china bowl. and wash this crystals with cold water and in last alcohol washed and crystals are formed of pink colour.

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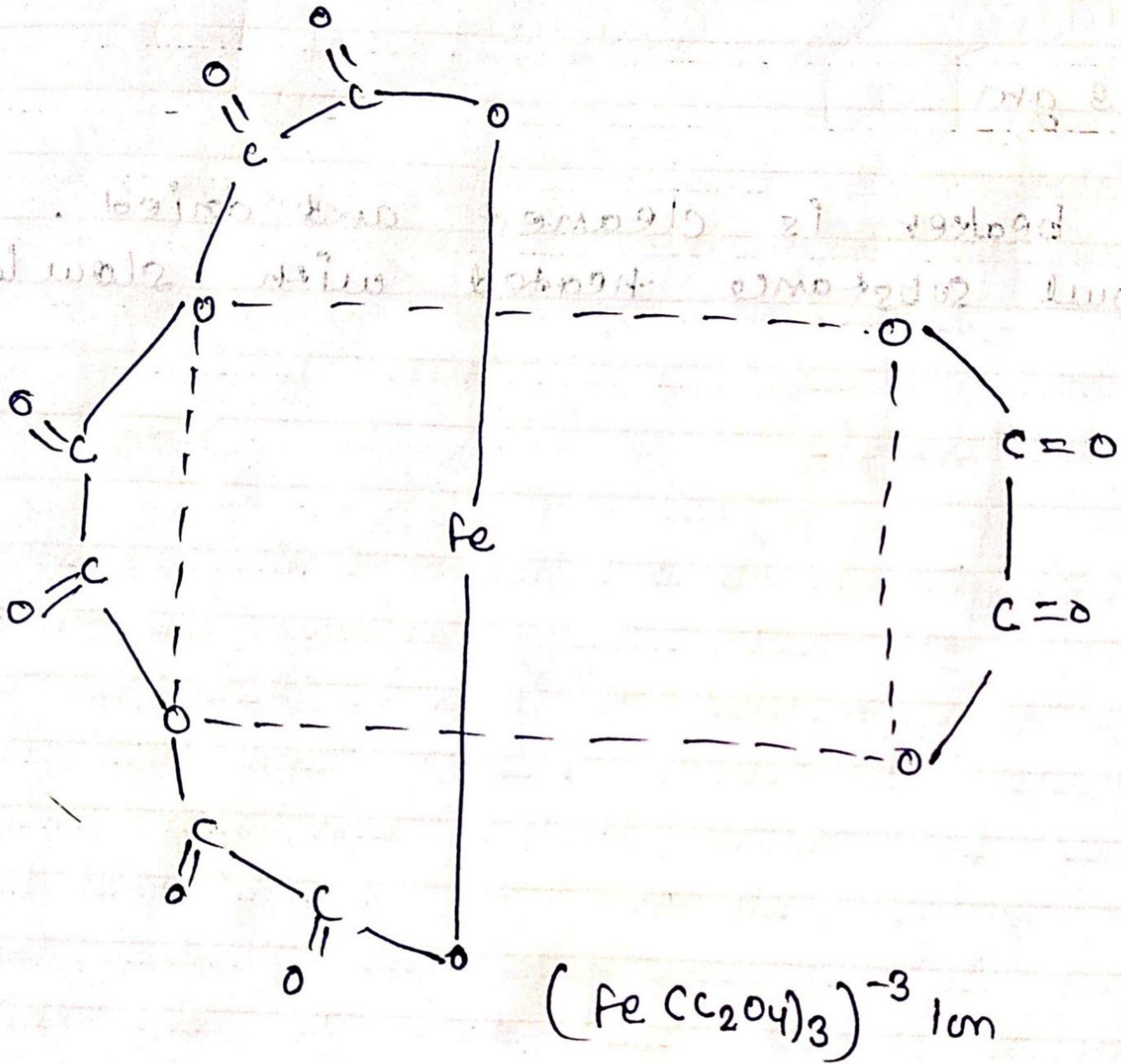
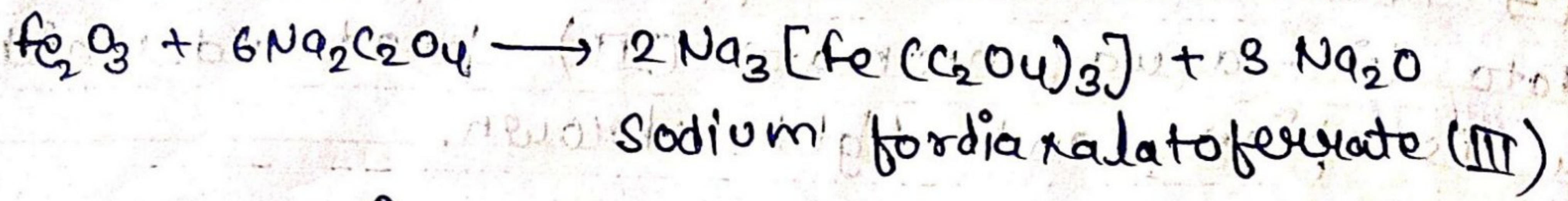


Result $\rightarrow$ Above method the opposition potassium di-oxalato di-aqua chromate (III) produced which colour is pink and light brown.

Grain = 5.3 gm

Precaution $\rightarrow$ beaker is cleaned and dried.
china bowl substance heated with slow low.

Teacher's Signature.....



Experiment No- 6

Object $\rightarrow$ Sodium dioxalatoferriate (III) $\text{Na}_2(\text{FeC}_2\text{O}_4)_3$ formation

Necessary Equipment - 1) Ferric chloride 10 gm

2) NaOH - 12 gm

3) oxalic acid - 12 gm

Beaker watch glass.

Method $\rightarrow$ In a beaker 10 gm FeCl_3 10 ml water 3 gm NaOH 5 ml H_2O mixed now ferric chloride solⁿ mixed in few about of NaOH solⁿ then ferric hydroxide is produce. Now one another beaker mixed 12 gm oxalic acid in this solution two mixed 4 gm NaOH oxilate solution produced. Now with the help of glass rod the water solⁿ mixed in ferric hydroxide solution and heat it. Now this solution filtered and cooled and filtered the ~~for~~ water and alcohol put for 2-3 days.

Result - Above method the crystal of Sodium dioxalatoferriate green colour produced.

Yield - 15 gm.

Precaution - The beaker should be clean and clear.

Teacher's Signature.....

Experiment No - 7

Object $\rightarrow$ Preparation of aspirin from salicylic acid.

Acid Reaction used $\rightarrow$ Aspirin is formed by acetylation of salicylic acid with acetic anhydride.

(I) salicylic acid : 5.0 mm 25

(II) acetic anhydride acid : 7.0 ml, 22-5

(III) Concentrated H_2SO_4 : 0.5 ml.

Required equipment $\rightarrow$ 100 ml. volume round bottom flask, thermometer and beaker.

Method $\rightarrow$ Take 5.0 gram salicylic acid, 7.0 ml acetic anhydride and 0.5 ml conc. H_2SO_4 in a round bottom flask of 100 ml capacity. shake the mixture well and heat it on a water bath at 50-60°C for 15-20 minutes. cool this mixture and mix it with about 100 ml water taken in another bath. The solid substance gets separated. Filter it and dissolve it in a minimum amount of ethyl alcohol. On keeping it at room temp. needle like colorless crystals are obtained.

Physical State - Colorless needle like crystals

duration $\rightarrow$ 5.5 g 2.5 en

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Experiment No $\rightarrow$ 8

Object $\rightarrow$ To determine critical solⁿ temp. of phenol water system.

Principle $\rightarrow$ Take a weighed amount of two liquid in a well cleaned pipette. Two layers of the mixture are formed as is shown by the turbidity. Stir w the mixture. Now, heat the mixture & note the temp. (t_1) at which turbidity of two solⁿ disappears. Now, the miscible solution is cooled & the temp. at which turbidity again reappear is noted (t_2)

Average of temp. t_1 & t_2 is the miscibility temp.

If a graph is plotted between the composition (x-axis) and the temp. of the mixture (y-axis) a curve is obtained. Highest point in the curve is obtained. Highest point in the curve is the critical solⁿ temp.

Required apparatus - Boiling tubes, thermometer, stirrer, Calibrated pipette.

Required chemicals - Pure phenol and double distilled water.

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observation - Amount of phenol in test tube = 4gm
density of water = 1 gm/ml

observation table-

S.NO.	weight of Phenol (gm)	weight of H ₂ O (gm)	% of phenol in mixture	Temp. at which Turbidity disappears	Temp. at which Turbidity reappears	Average Temp. T _c $= \frac{T_1 + T_2}{2}$
1	4	4	50%	62.1	59.2	60.65
2	4	5	44%	62.3	61.2	61.75
3	4	6	40%	62.5	61.9	62.20
4	4	7	36%	62.5	62.2	62.85
5	4	8	33.33%	62.8	62.4	63.10
6	4	9	30.9%	62.6	61.4	62.20
7	4	10	28.5%	61.6	60.2	60.90
8	4	11	27.5%	60.8	59.6	59.90

Result → critical solⁿ Temp. of phenol water system = 63.1°C
concⁿ of phenol at critical solⁿ temp. = 33.33%

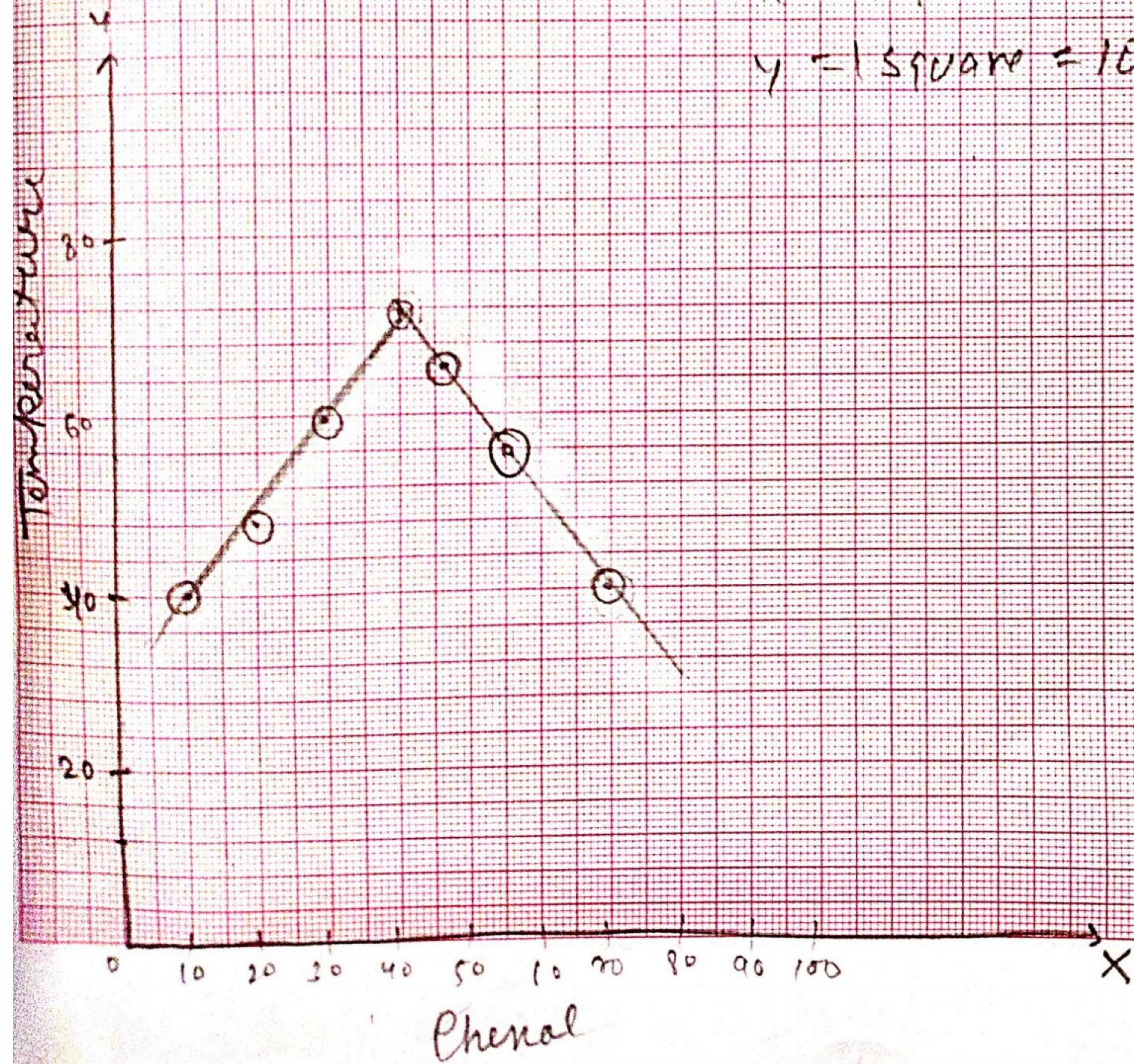
Teacher's Signature.....

Ans (8)

Scale

X = 1 square = 10 unit

Y = 1 square = 10 unit



Experiment No. 9

Object $\rightarrow$ nickel dimethylglyoxime complex $[\text{Ni}(\text{DMG})_2]$

Necessary Equipment $\rightarrow$

- (1) Nickel
- (2) Dimethylglyoxime - 10%
- (3) Ammonia solution $\rightarrow$ 1:1

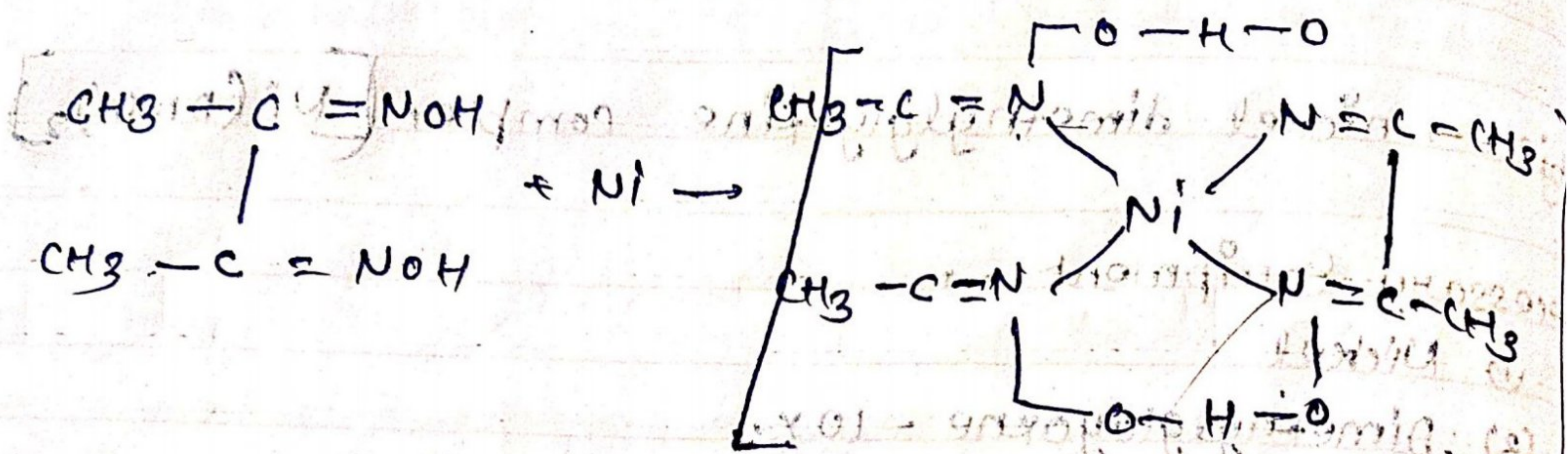
Equipments $\rightarrow$ Beaker, watch glass.

Method $\rightarrow$ One 400 ml. Beaker given nickel sulphate 6 gm Heat at $72-80^\circ\text{C}$ and this solution put in 42-50 ml. Di-methylglyoxime after this in solⁿ 1:1 ammonium solution drop by drop in watch glass. In Beaker the water Heat at 10-30 min. the down dropped used colour sum di-methylglyoxime reagent mixed and test it. That the solⁿ is complete or not. Beaker put for Half-Hours. this is separated by funnel and this is washed cooled water two three time and in last the solution of washed 5 ml alcohol, and dried it

Result $\rightarrow$ Above method $\text{Ni}(\text{DMG})_2$ produced Red Colour Solⁿ.

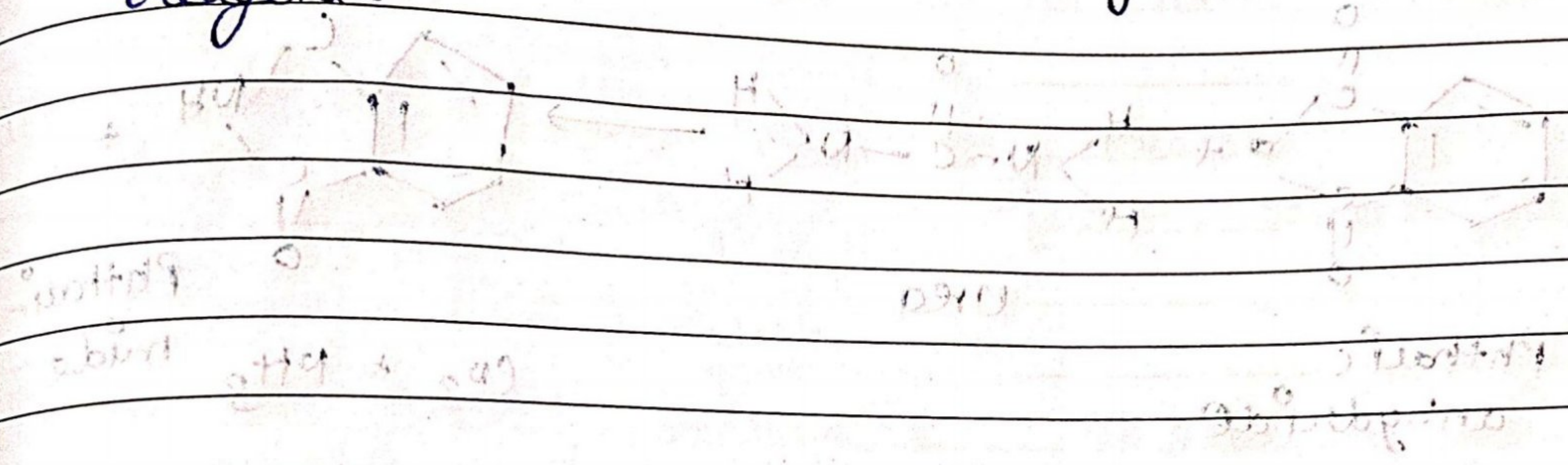
gained $\sim$ 3.4 gm.

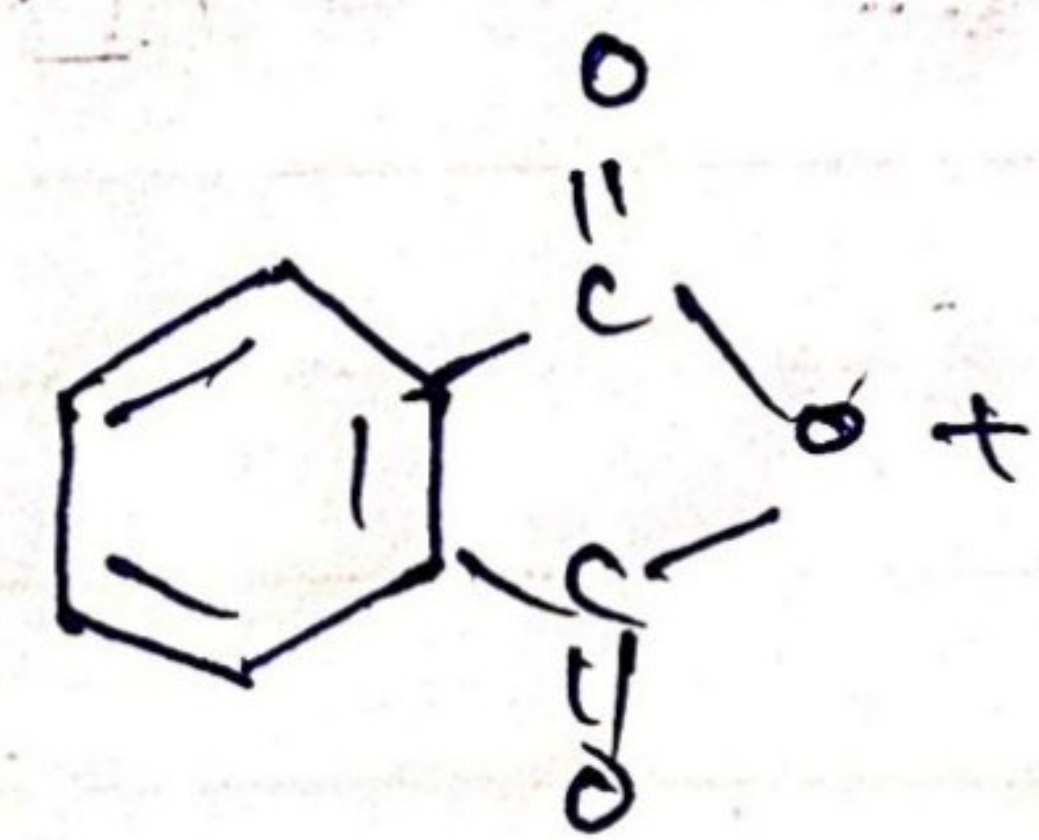
Teacher's Signature.....



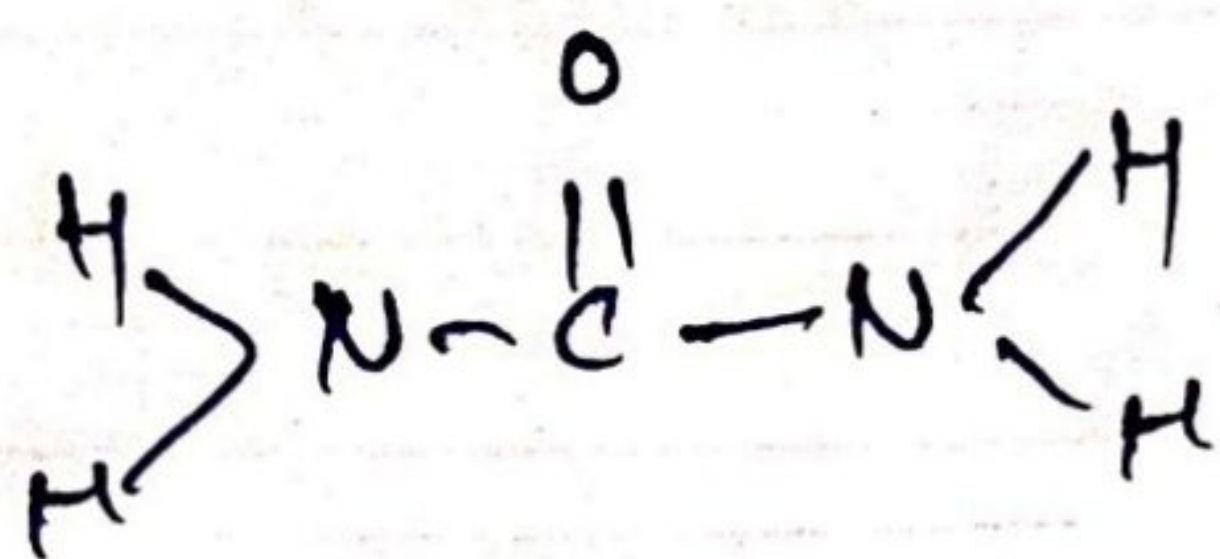
The solution of methylglyoxal in water is colorless. On adding a few drops of 10% sodium hydroxide solution, the color changes to yellow. This color change is due to the formation of a complex between the methylglyoxal and the hydroxide ions. The complex is a cyclic structure where the oxygen of the hydroxide group is coordinated to the carbonyl carbon of the methylglyoxal molecule. This complex is known as the methylglyoxal hydrate. The color change is reversible and can be used as a test for the presence of methylglyoxal in a solution.

Precaution → Beaker should be clean and dried.
 Beaker. Heat with the help of water
 reagent.

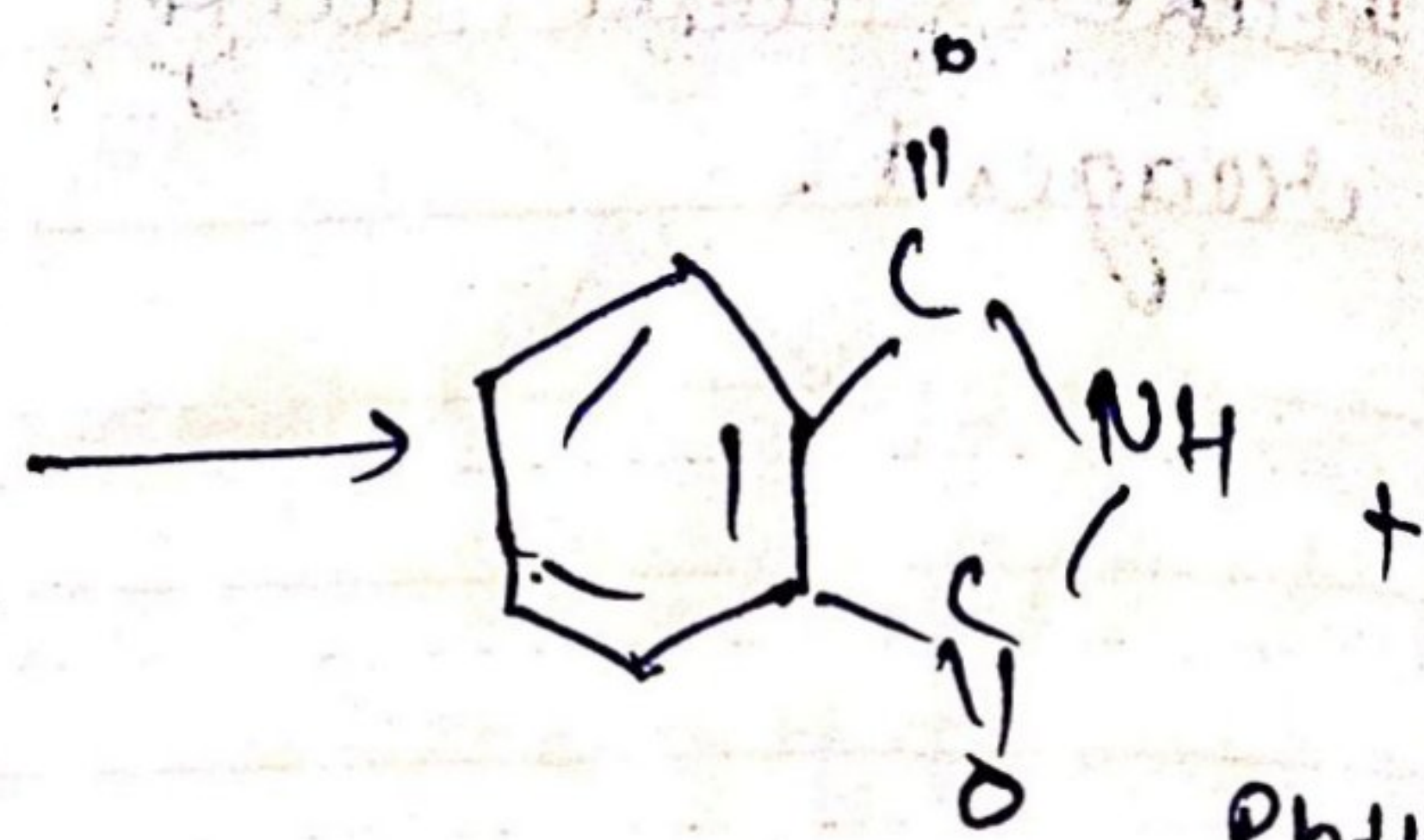




Phthalic anhydride



urea



Phthalimide

$\text{CO}_2 + \text{NH}_3$

Phthalimide

Experiment - 10

Object $\rightarrow$ Synthesis of phthalimide from phthalic anhydride

Theory $\rightarrow$ Phthalic anhydride when heated with ~~an~~ urea in the dry state (fusion), phthalimide is formed

Reagent $\rightarrow$ Phthalic anhydride 20 gms.
Urea 4 gms.

Procedure $\rightarrow$ Intimately mix 20 gms. of phthalic anhydride with 4 gms. of urea in a round bottom flask. Heat the flask to $130^{\circ} - 135^{\circ}\text{C}$ in an oil bath. When the contents have melted, effervescence commences which gradually increases in vigour. After 10-15 minutes, the mixture suddenly froths up to about three times its original volume and becomes almost solid. Allow the contents to cool, add about 20 cc. of water, stir and filter. Wash with water. Recrystallise 2 gm. of the product from ethyl alcohol or hot water.

Yield - 1 gms

M.P. $\rightarrow$ 138°C

Colour $\rightarrow$ white powder

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Precautions - Phthalic anhydride and urea should be mixed just before starting the preparation.

A large flask should be taken to heat the mixture as frothing take place.

Teacher's Signature.....