

Systematic Qualitative Analysis

OF ORGANIC COMPOUND (UREA)

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Urea



Urea is white crystalline solid and is odourless, soluble in cold and hot water.

Preliminary Analysis

TEST	OBSERVATION	INFERENCE
<u>a. Physical State</u>	Crystalline Solid	Carboxylic acids, Carbohydrates, Amides, Nitro phenols and higher Hydrocarbons may be present.
<u>b. Colour</u>	White	Carboxylic acids, Simple amides, Carbohydrates may be present.
<u>c. Odour</u>	Odourless	Carbohydrates, Aromatic acids, Amides may be present.
<u>d. Flame (Ignition) Test</u> <u>Introduce ~5g of compound on metal spatula on low blue flame until it got burnt completely</u>	Non sooty flame with smell of ammonia	Compound is Aliphatic, most likely amide

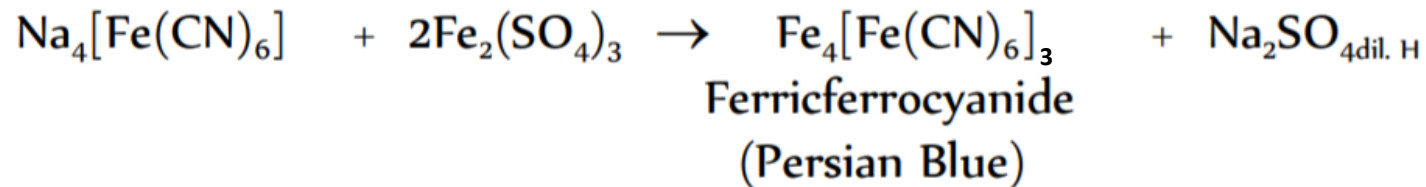
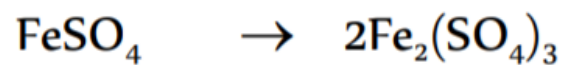
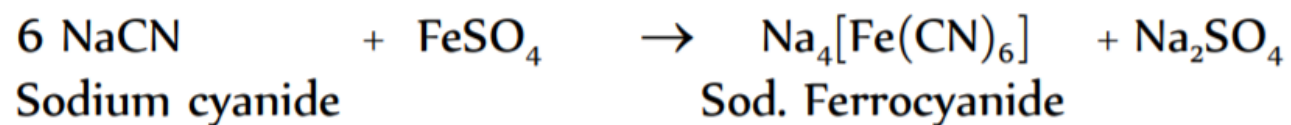
TEST	OBSERVATION	INFERENCE
<u>e. Solubility Test</u>		
1. Water (Cold and Hot)	Soluble	Lower Acid, Amide or Carbohydrate may be present
2. NaHCO_3 solution	Insoluble	Acidic group absent
3. Dilute NaOH solution	Insoluble	Acidic and Phenolic group absent
4. HCl solution	Soluble	Basic group present (may be amide)
5. Conc H_2SO_4 solution	Soluble	Basic group present (may be amide)
<u>f. Unsaturation Test</u>		
1. Baeyer's Test Compound (0.5 g) + 1mL water + 0.5 mL of dilute Na_2CO_3 solution + 1-2% of aq KMnO_4 Solution dropwise	No colour discharge	Compound is saturated
2. Bromine Solution Test Compound (0.5 g) + 1mL water + 2% of Bromine solution in water dropwise with shaking	No colour discharge	Compound is saturated

Extra Element Detection (N, S and X)

TEST	OBSERVATION	INFERENCE
<p>a. <u>Test for Nitrogen (FeSO₄ test)</u></p> <p>2mL of sodium extract + 1mL freshly prepared FeSO₄ solution (if a dark green ppt is not formed, add 2-3 drops of NaOH solution to make it alkaline). Boil gently for a minute. Add 3-4 mL dil H₂SO₄ solution to hot mixture.</p>	Prussian blue colour	Nitrogen confirmed
<p>b. <u>Test for Sulphur</u></p> <p>1. Lead Acetate Test</p> <p>2mL of sodium extract + 2mL of dil Acetic acid + 4-5 drops of Lead Acetate solution.</p>	No black precipitate observed	Sulphur absent
<p>2. Sodium Nitroprusside Test</p> <p>1mL of sodium extract + 2-3 drops of freshly prepared Sodium Nitroprusside solution.</p>	No violet colouration observed	Sulphur absent

TEST	OBSERVATION	INFERENCE
<u>c. Test for Halogens</u> <i>2 mL of sodium extract + conc nitric acid. Boil and Cool. Add Silver nitrate solution</i>	No precipitate observed	Halogen absent

REACTION (FeSO₄ test for Nitrogen)



Functional Group Detection

Functional Groups Without Extra Elements

TEST	OBSERVATION	INFERENCE
a. <u>Carboxylic Acid</u> 1. Litmus Test <i>Added blue litmus paper in aqueous solution of compound</i>	No colour change	Carboxylic acid may be absent
2. NaHCO_3 Test <i>Added small amount of compound in 2 mL cold saturated NaHCO_3 solution.</i>	No effervescence observed	Carboxylic acid absent
b. <u>Alcohol</u> 1. CAN Test <i>0.5 mL solution of compound in water + 2-3 drops of Ceric Ammonium Nitrate solution.</i>	No red colouration observed	Alcohol absent

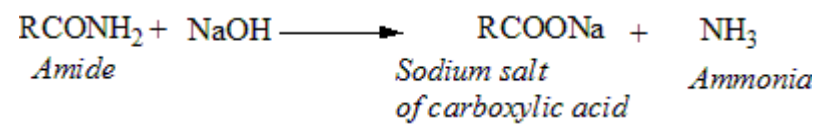
TEST	OBSERVATION	INFERENCE
<p>c. <u>Phenol</u></p> <p>1. <i>nue</i> FeCl₃ Test Added 1-2 drops of neutral FeCl₃ solution in very dilute solution of compound</p>	No blue/ green/ violet/ brown colour observed	Phenol absent
<p>d. <u>Carbonyl Group (2, 4 DNP Test)</u></p> <p>Added 10-12 mL of 2, 4 DNP reagent in 2-3 drops of solution of compound in water. Shaken vigorously and heated in water bath for 5-10 minutes. Allow to stand it for 5-10 minutes.</p>	No precipitate observed	Carbonyl group absent
<p>e. <u>Ester</u></p> <p>1. Phenolphthalein Test To 1 mL of solution of compound in water, added 2 drops of phenolphthalein and dil NaOH solution dropwise till pink colour persist.</p>	No discharge of pink colour	Ester absent
<p>f. <u>Carbohydrate</u></p> <p>1. Molisch's Test 0.05 g of compound + 2 mL water + 3 mL of freshly prepared Molisch's reagent (10% ethanoic α-naphthol). Shaken well. Poured 2-3 mL of conc sulphuric acid along sides of test tube.</p>	No violet ring observed	Carbohydrate absent

Functional Groups With Extra Element (Nitrogen)

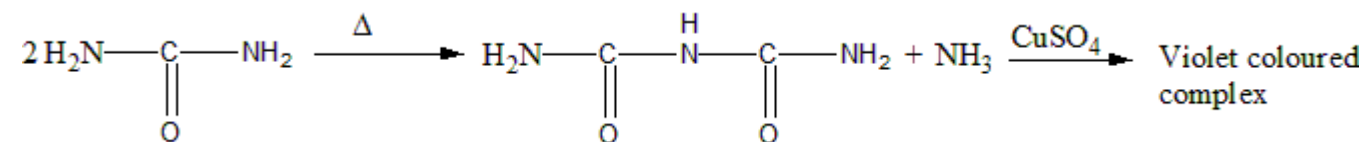
TEST	OBSERVATION	INFERENCE
a. <u>Amide Group</u>		
1. Hydrolysis Test <i>5 mL 10% NaOH solution was added to 0.5g of compound and boiled. (Test evolution of ammonia by placing a red litmus paper OR a glass rod dipped in HCl on mouth of test tube OR by passing gas through Nessler's reagent (Potassium tetraiodomercurate(II))).</i>	Ammonia evolved. (Turns red litmus paper to blue, or white fumes of NH_4Cl appears when came in contact with HCl, or turns Nessler's reagent brown)	Amide group confirmed
2. Hydroxamic Test (Unsubstituted amide) <i>Added $\text{NH}_2\text{OH} \cdot \text{HCl}$ solution and ethanol in 0.5g of compound. Boiled for 2-3 minutes and cool. Added 4-5 mL of freshly prepared FeCl_3 solution.</i>	Colour changes from red to violet	Unsubstituted amide group confirmed
3. Biuret Test (Urea and mono substituted Urea) <i>Heated 0.5g of compound gently until melted substance just solidifies. Ammonia will be evolved leaving white solid (Biuret) behind. Dissolved Biuret in warm water and 1mL 10% NaOH solution. Added few drops of very dil CuSO_4 solution.</i>	Violet/Purple colour observed	Urea (or mono substituted urea) confirmed

REACTIONS

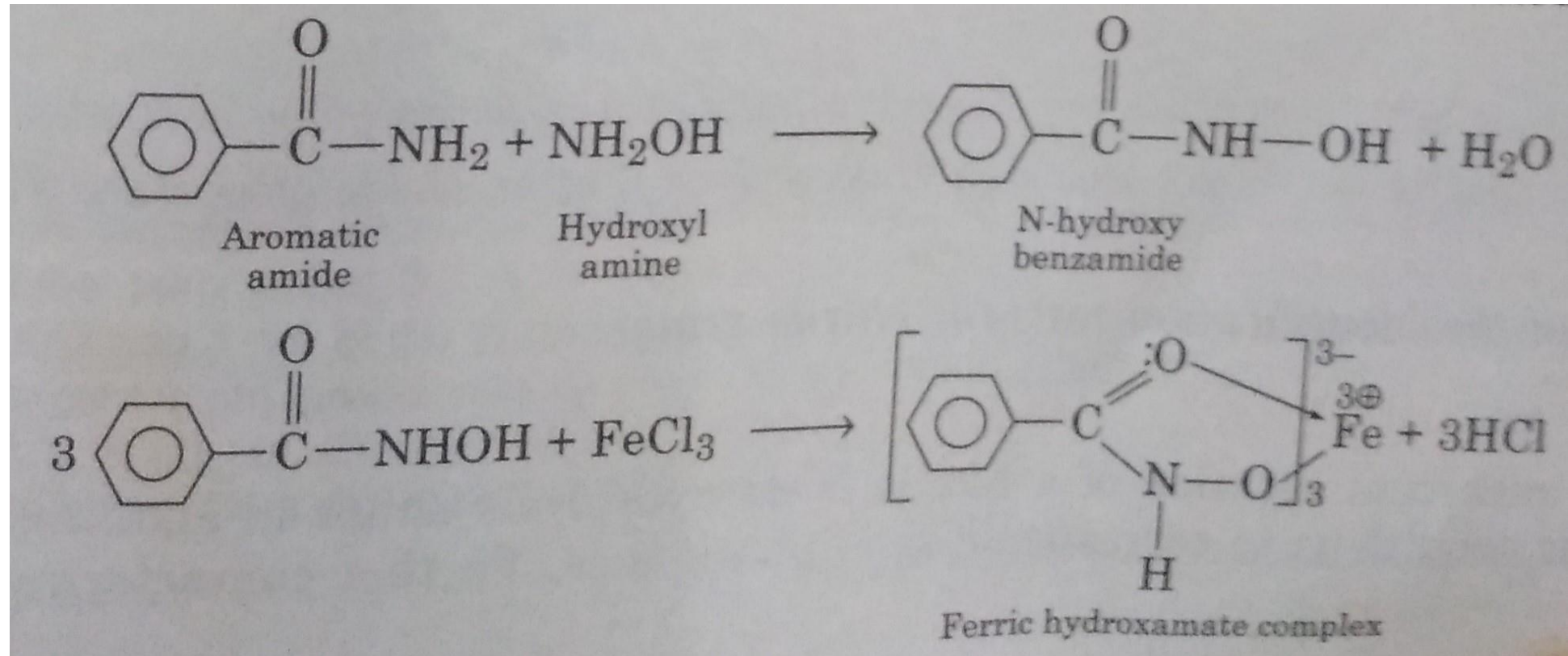
Hydrolysis Test



Biuret Test



Hydroxamic Test



Melting Point and Probable Compounds

Melting Point

Melting point can be measured either by kjeldahl's method or by electrically heated apparatus. Melting range of compound is determined when it starts melting and when it melts completely. Most probable melting point range for Urea is $\approx 130-131^\circ\text{C}$

Compound	Melting Point
Urea	132°C
Isobutyramide	129°C
Succinamide	126°C
Isovaleramide	136°C

Note : Out of above compounds, only Urea gives positive Biuret test. So most probable compound is Urea.

Preparation of Derivatives

Urea is a monoacid base. It reacts with acids to produce corresponding salts.

Urea Oxalate

Urea reacts with Oxalic acid (simplest dicarboxylic acid) to produce crystalline salt, Urea Oxalate which has a sharp melting point of 171 °C.

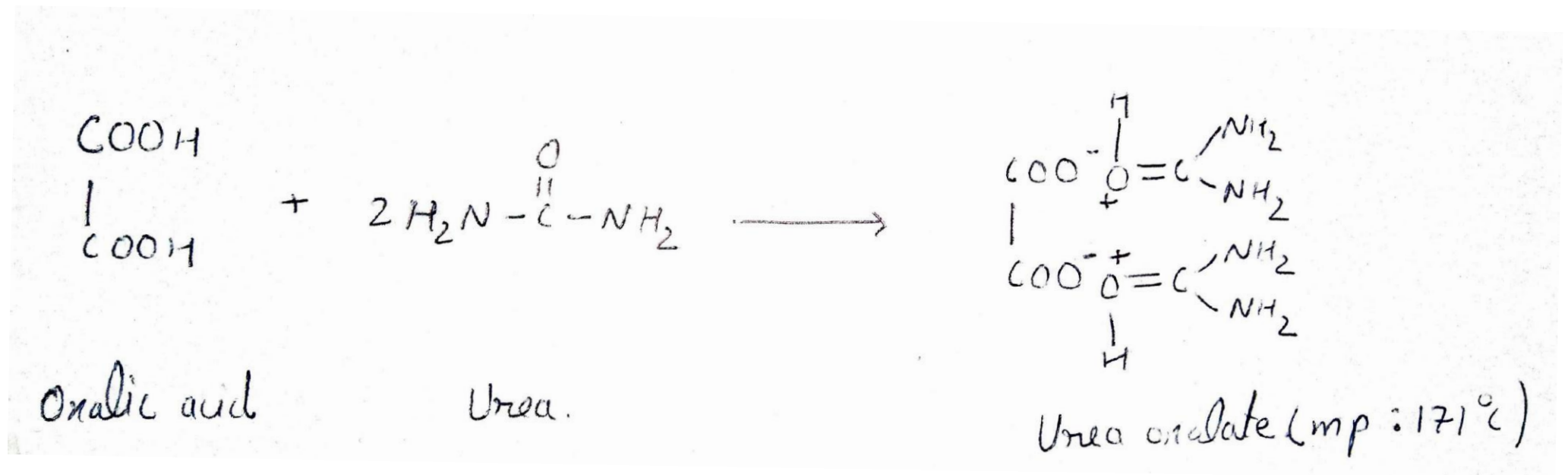
Preparation

3 mL of conc aqueous solution of Oxalic acid was added to 3 mL of aqueous solution of Urea (Cool the mixture in ice bath while stirring since it is exothermic). Crystals of Urea Oxalate separates out of reaction mixture. Recrystallized with dilute ethanol.

Melting Point

Melting point of recrystallized derivate is most probably ≈ 170 -172 °C. It is in close agreement with that of Urea Oxalate (171 °C).

Reaction



Urea Nitrate

Urea reacts with conc Nitric acid to produce explosive salt Urea Nitrate which has a melting point of 163°C.

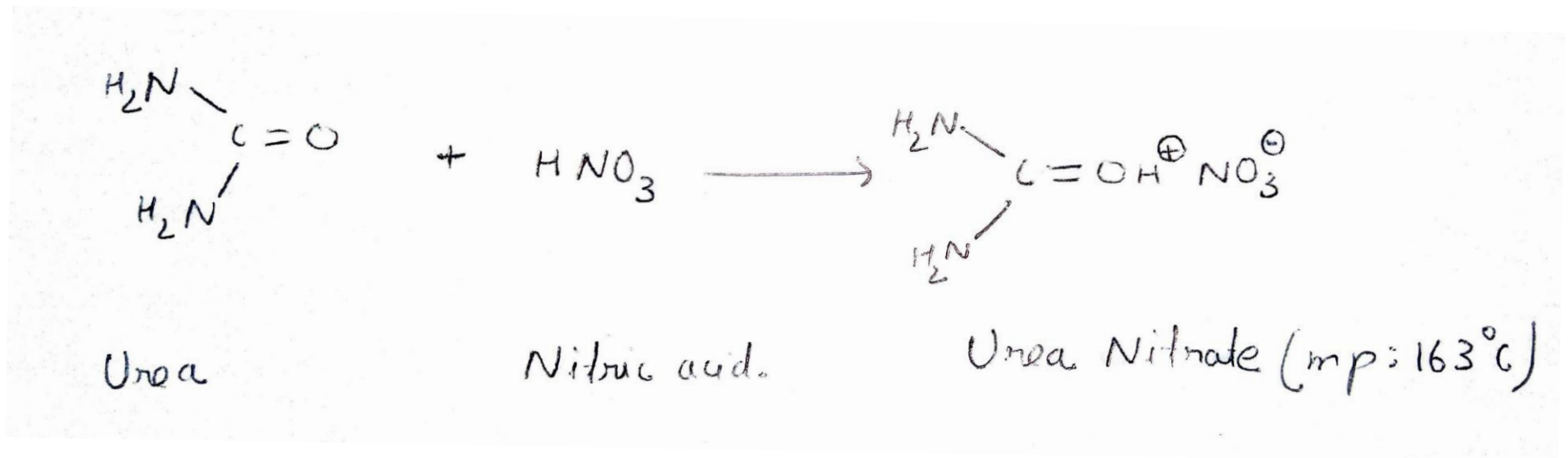
Preparation

3 mL of conc Nitric acid solution was added to 5 mL of conc aqueous solution of Urea (Cool the mixture in ice bath while stirring since it is exothermic). Allowed the reaction mixture to stand for 5 minutes. Filtered the reaction mixture to obtain Urea Nitrate. Recrystallized with dilute ethanol.

Melting Point

Melting point of recrystallized derivate is most probably $\approx 162-164$ °C.
It closely corresponds to Urea Nitrate (163°C).

Reaction



Result

THE GIVEN ORGANIC COMPOUND IS IDENTIFIED AS UREA.