Nature Of Invention: Chemical molecule and synthesis route

Applicant: ChemiEvolve Industries

Inventors: Nitin Gautam, Raunak Jalan

Chemical Formula: $(C_6H_4CO_2H)_2$

Chemical Name: Diphenic Acid

Chemical synthesis routes:

LAB-SCALE SYNTHESIS OF DIPHENIC ACID:

Method 1: Diazotization of Anthranilic Acid

Raw Materials:

- **A: For the Diazotization of Anthranilic Acid :**
  - Anthranilic acid (50 grams)
  - Water (150 cc)
  - Concentrated hydrochloric acid (92 cc, with a specific gravity of 1.19)
  - Sodium nitrite (26.3 grams)
  - Water (350 cc)
- **B: For the Preparation of the Reducing Agent :**
  - Hydrated cupric sulphate (126 grams)
  - Water (500 cc)
  - Concentrated ammonium hydroxide (210 cc, with a specific gravity of 0.90)
  - Hydroxylammonium sulphate (42 grams)
  - Sodium hydroxide solution (6 N, 85 cc)
  - Water (120 cc)
- **C: For the Synthesis of Diphenic Acid :**
  - Diphenic acid (from the diazonium solution prepared in Part A)
  - Concentrated hydrochloric acid (250 cc)
  - Sodium bicarbonate (40 grams)
  - Norite (0.1 grams)
  - Cold water (50 cc)
  - 6 N hydrochloric acid (excess)
  - Water (200 cc)

Reaction Steps (Product Yield):

A: Diazotization of Anthranilic Acid:

- 50 g of anthranilic acid were ground with 150 cc of water and 92 cc of concentrated hydrochloric acid in a mortar.
- The suspension was transferred to a 1-litre round-bottomed flask surrounded by an ice bath.
- A solution of 26.3 g of sodium nitrite in 350 cc of water was added dropwise over 30 minutes while maintaining the temperature below 5°C.
- The diazonium solution was filtered as it was not entirely clear.

B: Preparation of the Reducing Agent:

- 126 g of cupric sulphate pentahydrate were dissolved in 500 cc of water in a 2-litre beaker.
- 210 cc of concentrated ammonium hydroxide were added, and the solution was cooled to 10°C.
- A solution of 42 g of hydroxylammonium sulphate in 120 cc of water was prepared, cooled to 10°C, and 85 cc of 6 N sodium hydroxide solution were added.
- The hydroxylamine solution was added to the cupric sulphate solution while stirring by hand until reduction occurred.

C: Synthesis of Diphenic Acid:

- The reducing solution was surrounded by an ice bath to maintain the temperature at about 10°C.
- 80-90 cc of diazonium solution were added dropwise at a rate of about 10 cc per minute while stirring.
- Stirring continued for an additional five minutes.

Separation Steps (Final Purity):

- The solution was heated to boiling, and 250 cc of concentrated hydrochloric acid were carefully added.
- The precipitated diphenic acid was collected by filtration after overnight standing, washed with cold water, and dried.
- The crude product was suspended in 200 cc of water, solid sodium bicarbonate was added, and the mixture was filtered. It was then boiled with Norite.
- The mixture was filtered, the filtrate was acidified with excess 6 N hydrochloric acid, and the precipitated diphenic acid was collected by filtration, washed with cold water, and dried.

Reaction Conditions:

- For diazotization: Temperature below 5°C.
- For reduction: Temperature around 10°C.
- For synthesis of diphenic acid: Boiling followed by acidification.

Reaction Statistics:

Melting point of diphenic acid: 222–227°C (crude), 225–228°C (purified). Yield: 88–91% of theoretical amount (crude), 72–84% of theoretical amount (purified).

ALTERNATIVE ROUTE TO SYNTHESIS OF DIPHENIC ACID:

Method 2: Oxidation of Phenanthrene:

Raw Materials:

- Phenanthrene (Commercial grade)
- Glacial acetic acid
- 30% Hydrogen peroxide (H2O2)
- Benzene (solvent)

Reaction Steps (Product Yield):

- In a four-necked flask equipped with a stirrer, thermometer, fractional column, and dropping funnel, phenanthrene, glacial acetic acid, and benzene were introduced in appropriate proportions.
- The mixture was heated to boiling.
- 30% hydrogen peroxide was gradually added dropwise over 3-12 hours.
- Water was continuously distilled off in the form of an azeotropic mixture with benzene during the reaction.
- The conversion of phenanthrene to diphenic acid was monitored.

Separation Steps (Final Purity):

- After the reaction, acetic acid and benzene were distilled off under reduced pressure.
- The residue was treated with 10% sodium hydroxide.
- The undissolved materials, mainly unreacted phenanthrene, were separated by filtration.
- The filtrate was acidified with concentrated hydrochloric acid.
- Diphenic acid precipitated in crystalline form.
- Diphenic acid was obtained by filtration.
- Neutralisation was repeated by adding base and acidification was repeated by adding acid several times to obtain purer diphenic acid.
- Repeated experiments were conducted under optimum conditions to achieve higher yields and purity.

Reaction Statistics:

Yields of diphenic acid were 60~67%while purity was 96~98%. The melting point of the product was obtained to be 232~233°C.

References:

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List the contributions of each author:

- **NITIN GAUTAM** carried out the literature search and found the **reaction steps**, and **product yield.**
- **RAUNAK JALAN** and **NITIN GAUTAM** found necessary **separation steps** to achieve desired **product purity.**

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