Chapter:
06

Ch.Id:-ASU/GRF/EB/AEHFPOC/2022/Ch-06
DOI: https://doi.org/10.52458/9789391842697.2022.eb.grf.asu.ch-06

AIM: TO PERFORM SYSTEMATIC QUALITATIVE ANALYSIS OF AMINES

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Requirements

Chemicals

1. Glacial acetic acid
2. Bromine water
3. Sulfonyl chloride
4. Amine
5. Sodium nitrite
6. H_2SO_4
7. NaOH
8. Methanolic KOH
9. Aniline
10. Benzoyl chloride

Glass wares

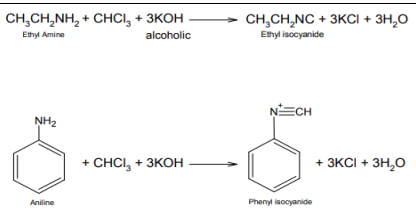
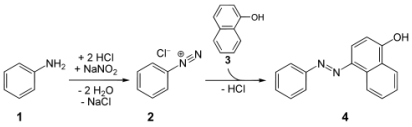
1. Test-tube
2. Brush
3. Holder
4. Glass rod
5. Beaker

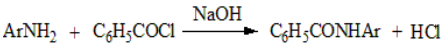
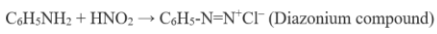
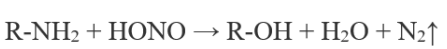
Theory: Amines are nitrogen-containing organic molecules that are considered derivatives of ammonia. According to the amount of alkyl groups connected to the nitrogen atom, they are categorised as primary, secondary, or tertiary. Similarly, to ammonia, amines are electron donors that function as both bases and nucleophiles. Alkyl amines are far more powerful bases than aryl amines. Due to the

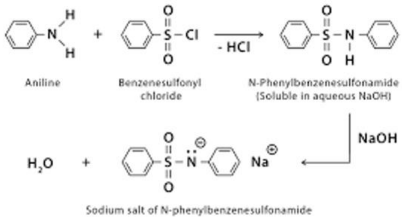
substantial overlap of the lone pair with the pi-electrons of the benzene ring, the basicity of aryl amines is decreased. Alcohols create hydrogen bonds more strongly than amines. Oxygen is more electronegative than nitrogen. Tertiary amines are incapable of forming hydrogen bonds. Amines have boiling values between those of alcohols and alkanes. Tertiary amines have a lower boiling point than 10 or 20 compounds of comparable molecular weight. Each amine is capable of forming hydrogen bonds with water. This hydrogen bonding makes amines with up to six carbon atoms water-soluble. As the length of the hydrocarbon part of the molecule grows, water solubility decreases. Classifying amines according to the amount of carbon atoms immediately linked to the nitrogen atom: A primary amine possesses one (RNH_2 = 1°), a secondary amine possesses two (R_2NH = 2°), and a tertiary amine possesses three (R_3N = 3°).

Functional Group Test for Primary Amines

S. No.	Identification Test	Observation	Inference
1	**Carbylamines test (Isocyanides test):** This test is used to distinguish among 1 °, 2 °, and 3° amines. Both aliphatic and aromatic amines contribute to its formation. To prepare a sample, combine alcoholic KOH with 2 drops of $CHCl_3$ and slowly heat. An offensive odour (phenyl cyanide) is indicative of primary amine.	Offensive smell	Primary amine is confirmed

			
2	**Azo dye test:** First, dissolve a tiny sample in 2 mL of conc. HCl. After that, dilution with water and chilling on ice should follow. The cold solution of nitrite in water should have sodium nitrite already dissolved in it. Add the sodium nitrite solution to the cold solution of nitrite in water drop by drop. The cold alkaline phenol (β-naphthol) solution should be supplemented with this solution on a drop-by-drop basis. 	Red colour dye	Primary amine is confirmed
3	**Acetylation:** In a clean and dry test tube mix 1 ml of glacial acetic acid and 0.5 ml of acetic anhydride to 0.1 g of substances, boil slowly for 3-5 minutes, and transfer to a beaker containing 20 ml of water.	White crystalline ppt	Primary amine is confirmed

4	**Benzoylation:** In a clean and dry test tube mix 1ml of NaOH solution and 1ml of benzoyl chloride to 1ml of a given sample and shake well.  	White ppt	Primary amine is confirmed
5	**Nitrous Acid Test:** 1 °, 2 °, and 3° amines may all be distinguished with the use of this test. When an aromatic primary amine is exposed to nitrous acid, the resulting reaction produces a diazonium salt, which, when subjected to higher temperatures, breaks down into its component elements.  1° **aliphatic amines:** 1° aliphatic amines react with nitrous acid to produce nitrogen gas, observable as bubbles.  	Aromatic amine diazonium formed & aliphatic amine N_2 bubbles	Primary amine is confirmed
6	**Hinsberg reaction:** The name of the Hinesburg reagent is benzene sulfonyl chloride. On reaction with benzene sulfonyl chloride and NaOH, primary aliphatic amines produce *N*-alkyl sulphonamide, which includes an acidic hydrogen	Sulphonamide ppt	Primary amine is confirmed

	and hence dissolves in NaOH solution to create the soluble sodium salt. The resulting solution, upon acidification, produces an insoluble precipitate of free sulphonamide. A primary amine will create a sulphonamide salt that is soluble. This salt's acidification precipitates the main amine's sulphonamide.  Aniline Benzenesulfonyl chloride N-Phenylbenzenesulfonamide (Soluble in aqueous NaOH) Sodium salt of N-phenylbenzenesulfonamide		

Result: The results of the systemic qualitative tests performed and primary amine was found and reported.