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PREPARATION OF TRIAZOLE WITH THE HELP OF M-NITRO PHENYL ACETIC ACID

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INTRODUCTION

Phosphorus pent chloride (4.5g) was added to a solution at m-nitro phenyl acetic acid (5g) in dry chloroform (40 mL) and then mixture was heated on a water bath with shaking for two hours. The excess at phosphorus oxychloride was removed by distillation under reduced pressure. The residual liquid on cooling in ice chest, gave solid which was separatcd by filtration and recrystallized from carbon tetra chloride to furnish the pure crystals at m-nitro - phenyl acetyl chloride (4.2g)

PREPARATION OF m-NITRO PHENYL ACETAMIDE

Place 5 ml of concentrated ammonia solution in a 20 mL flask and cool in ice 1.2 g of redistillised carboxylic acid chloride drop by drop from a separatory funnel whilst shaking the flask frequently. Filter off the precipitated acid amide wash with a little cold water and recrystalliscd from hot water The crystals were dried upon filter paper in the air. The yield of pure acid amide is 1.1 g m.p 76°C (Lit[131] – 78°C).

PREPARATION OF m-NITRO PHENYL ACET HYDRAZIDE

This preparation was done by reacting m-nitro phenyl acid chloride and hydrazine.

Following the method of Vogel, m-nitro phenyl acetate (3 g) in carbon tetra chloride (10 mL) was heated with hydrazine hydrate (5 mL); (90%) for three hours under reflux, when homogeneous mixture was obtained the volume at the solution was reduced to half, this solution was cooled, pale yellow solid was separated which was collected by filtration. Recrystallzation at the solid from methanol furnished m-nitro phenyl acetydrazide (2.1 g) as light yellow crystals m.p. 154-155°C (Lit[131] m.p. 155°C).

PREPARATION 2. 5-di-(m-NITRO BENZYL) 13, 4 TRIAZOLE

(By heating reaction between m-nitro phenyl acethydrazide and m-nitro phenyl acetamide).

This reaction was done under Various Conditions

(a) With Phosphorus pent oxide :

A mixture of m-nitro phenyl acet hydrazide (0,5 g) m-nitro phenyl acetamide (0.3 g) phosphorus pent oxide (0.02 g) in carbon tetrachloride (2 mL) was heated on water bath for four hours. The completion of reaction was indicated by TLC, Examination. The reaction mixture was left at room temperature overnight. The separated solid was triturated with benzene and ethanol and then recrystalised from ethanol to furnish light yellow crystal of pure 2, 5-di-(m-nitro benzyl)-l, 3, 4-triazole (0.40 g) m.p 167°C.

Analysis

Found	20.15%
Calculated for $C_{12}H_{13}N_5O_5$	20.77%

IR (Stretching frequencies)

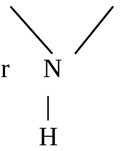
KBr 1590 cm^{-1} for C=N stretching

V_{max} 2905 cm^{-1} for C=H stretching

(Showing the pressure of CH_2 grasping)

.... for C–N bending

3250 cm^{-1} for



1620 cm^{-1} for showing disentitled benzene

1540 cm^{-1} for aromatic nitro group.

(b) With dry hydrogen chloride :

A mixture of m-nitro phenylacet hydrazide (0.5 g) m-nitro phenyl acetamide (0.4 g) in benzene (2 mL) was heated under condition of reflux on water bath for five hours and dry hydrogen chloride gas was passed in this reaction mixture slowly. The progress of the reaction was carried out time to time by TLC examination.

The completion of was indicated by TLC examination and then reaction mixture was set aside at room temperature overnight. The yellow solid separating from the reaction mixture was triturated was aqueous sodium bicarbonate, washed with cold water and recrystallised from ethanol to furnish the pure triazole (0.45 g) m.p. and mixed m.p. 167°C.

(c) Direct on heating :

A mixture of m-nitro phenylacet hydrazide (0.5 g), m-nitro phenyl acethydrazid (0.5 g), m-nitro phenyl acetamid (0.4 g) in benzene (2 ml) heated under reflux for six hours and placed at room temperature overnight. The yellow solid separating from the reaction mixture was obtained from filtration and recrystallised from ethanol to furnish the pure triazole (0.15 g) m.p. and mixed m.p. 167°C.

(d) In pyridine on heating :

A mixture of p-nitro phenyl acethydrazide (0.5 g), p-nitro phenyl acetamide (0.4 g) and pyridine (1 ml) was heated on a water bath for four hours. The completion of reaction was indicated by TLC. The reaction mixture was then left overnight and yellow solid was obtained. This was washed with benzene and ethanol to gave the pure triazole as yellow crystal (0.1 g) m.p. and mixed m.p. 211.1°C.

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