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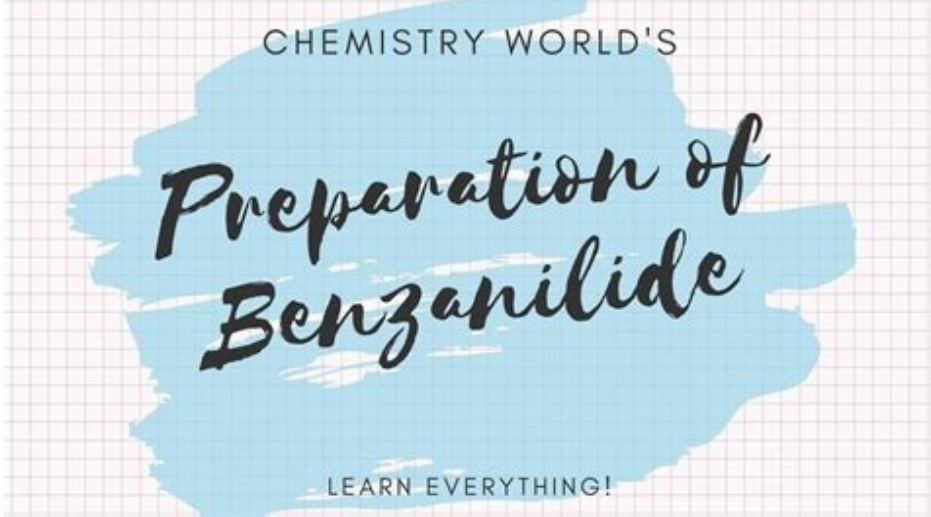
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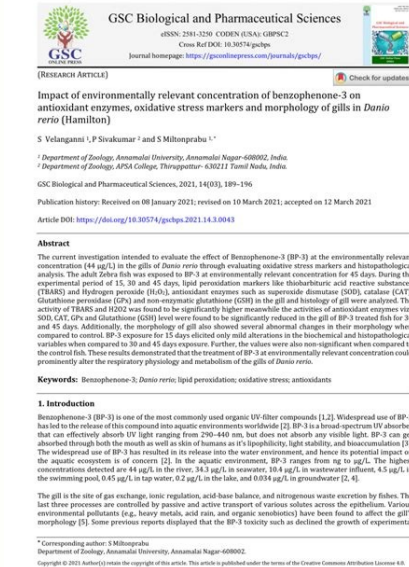
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## Preparation of benzanilide from benzophenone pdf

It rapidly rearranges to give an isomeric Oxime of higher melting point by acids-(beta or anti aldoxime). • Oximes of ketones undergo Beckmann rearrangement to substituted amides under the influence of variety of acidic reagents e.g HCOOH, PCl5, H2SO4, SOCl2, etc. • In this rearrangement, the migrating group is always anti-to -OH group and thus this reaction is stereo specific. • Here benzophenone oxime in presence of PCl5 rearranges to benzanilide. Step involved in Beckmann Rearrangement Reaction: Step-I: Preparation of benzophenone oxime Step-II: Preparation of benzanilide from benzophenone oxime Mechanism of Beckmann Rearrangement MECHANISM:- Step-I: Preparation of benzophenone oxime Step-II: Preparation of benzanilide: • Aldehydes and ketones react with hydroxyl amines in the presence of sodium hydroxide to give an Oxime of low melting point (alpha or syn aldoxime). • This is stable in alkali but rapidly rearranges to give an isomeric Oxime of higher melting point by acids-(beta or anti aldoxime). • Oximes of ketones undergo Beckmann rearrangement to substituted amides under the influence of variety of acidic reagents e.g HCOOH, PCl5, H2SO4, SOCl2, etc. • In this rearrangement, the migrating group is always anti-to -OH group and thus this reaction is stereo specific. • Here benzophenone oxime in presence of PCl5 rearranges to benzanilide. Copyright © 1921-2023 by Organic Syntheses, Inc.All Rights Reserved. No part of this Website or Database may be reproduced, stored in a retrieval system or transmitted in any form or by any means, electronic, mechanical, photocopying, recording, scanning or otherwise, except as permitted under Sections 107 or 108 of the 1976 United States Copyright Act. Synthesis of Benzanilide Benzanilide (CAS NO. : ) could be produced through the following synthetic route. In a 3-l. round-bottomed flask are placed 750 g. (8.1 moles) of aniline and 1 kg. (8.2 moles) of benzoic acid. When about two-thirds of the benzoic acid is in the flask the mixture is melted to make room for the rest. The flask is placed in a large oil bath and connected to a condenser for distillation.



The temperature of the oil is raised quickly to 180-190°, at which point distillation starts. The bath is held at this temperature until practically no more aniline and water distil (about two hours), and then the temperature is slowly raised to 225° and maintained at this temperature until no further distillation takes place (one to two hours). The oil bath is now removed and the contents of the flask are allowed to cool below 180° and 550 g. (5.9 moles) of aniline is added. The distillations at 190° and 225° are repeated (about six hours).



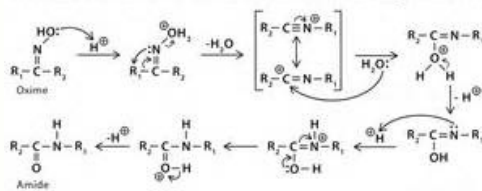
The hot mixture is poured into two 20-cm. evaporating dishes (Hood) and is allowed to cool. The crude product weighs 1600-2000 g., depending on the amount of aniline retained. The purplish-gray solid is ground in a large mortar and is poured with vigorous stirring into a 12-l. (3-gallon) crock containing 6 l. of approximately normal hydrochloric acid (5.1 of water and 500 cc. of concentrated hydrochloric acid). The stirring is continued for one hour after all the benzanilide has been added, and then the solid is filtered on a 20-cm. Büchner funnel. The process of stirring with acid and filtering is repeated twice to remove the excess of aniline. The solid is stirred for two hours with 6 l. of water and is filtered.



It is stirred for one hour with 6 l. of normal sodium hydroxide solution to remove the excess of benzoic acid and is then filtered. The stirring with alkali and filtering is repeated. The solid is next stirred for two hours with 7 l. of water and is filtered, sucked dry, and air-dried overnight on paper. After drying on paper the purplish solid is dried to constant weight in three 20-cm. evaporating dishes at 90-100° (about two days) and is then repowdered. The product is light purplish-gray and weighs 1270-1325 g. (80-84 per cent of the theoretical amount). It melts at 157-160°. The product is pure enough for use in the preparation of p-dimethylaminobenzophenone (p. 217) and for most synthetic purposes. When 100 g. of benzanilide is dissolved in 750 cc.

hot alcohol and the solution is boiled with about 10. g. of decolorizing carbon (Norite), and, cooled at 10° overnight, 80–86 g. of an almost colorless product melting at 160–161° separates. A second crystallization from alcohol using decolorizing carbon gives a white product with approximately the same loss in the mother liquors as in the first crystallization. Prev.No record Next.No record [Back] [Close] [Print] [Add to favorite] Health and Chemical more » 4466-18-6 1,3-Tris(4-hydroxy-*p*-*a*-dimethylbenzyl)benzene 15362-40-1 (2-(6-Chlorophenyl)-2-indolinone 104-76-2 Ethylhexan-1-ol 1682-37-1 3-Benzodioxole-5-diazonium, tetrafluoroborate(1-) 6836-19-7 7-Methoxy-1,3-propanediol 147-82-0 2,4,6-Tribromoisobenzene 70266-48-2 2-Buten-1-one, 1-(2,4,4-trimethyl-1-cyclohexen-1-yl)- 97281-24-8 Fatty acids, C8-10, mixed esters with neopentyl glycol and trimethylolpropane 19554-74-5 Ethonium 9010-75-7 POLY(CHLOROTRIFLUOROETHYLENE-CO-VINYLIDE NE FLUORIDE) 26 MOLE% VINYLIDE NE FLUORI 78-26-2 1,3-Propanediol-2-methyl-2-propyl- 63641-63-4 Tolueneamine, propylene oxide polymer 3-benzenediamine, ar-methyl- polymer with methylloxirane 1,3-benzenediamine, ar-methyl- polymer with methylloxirane 1,3-benzenediamine, ar-methyl- polymer with methylloxirane Tolueneamine, propylene oxide polymer Principle:Isertion of benzoyl moiety instead of an active hydrogen atom present in hydroxyl (OH) primary amino (NH2) or secondary amino group (NH) is usually termed as benzoylation reaction.1 Telegram:SearchFacebookFollowWebsiteClick hereThis particular reaction essentially bears a close resemblance to the phenomenon of acetylation except that in this specific instance the reagent is (benzoyl chloride) which reacts in the presence pyridine or 10% NaOH and not benzoic anhydride.The amines are more soluble in acid chloride than in NaOH, the reaction occurs preferably between benzoyl chloride and amine. In the preparation of benzaldehyde, NaOH neutralizes the liberated HCl and also catalyze the reaction.2Aim: To prepare benzaldehyde from aniline.Reaction:Mechanism:Benzyloxylation of compounds those are containing active hydrogen such as phenol, aniline, alcohol etc. form benzoyl chloride in the presence of aqueous NaOH (Schotten Baumann reaction).Use:it is used as fungicide and acaricide (pesticide that kill ticks and mites).Chemicals: Aniline - 2 ml, 10% NaOH -30 ml, Benzoyl chloride - 3 ml, Cold water, Hot alcoholApparatus: Conical flask - 250 ml, Buchner funnel, Measuring cylinder, Filter paper,Place 2 ml (2.08 g) of aniline 30 ml of 10% NaOH solution in 250 ml conical flask, then add 3 ml (3.4 g) of benzoyl chloride slowly with vigorous shaking.

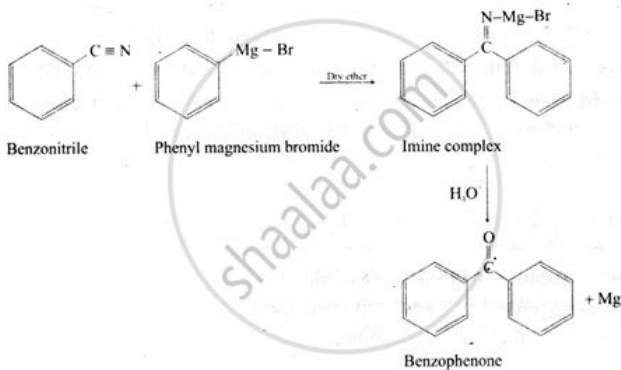
### Mechanism of Beckmann Rearrangement



Cork the flask and shake for further 15-20 min or till the odour of benzoyl chloride can no longer be detected. Dilute the reaction mixture with cold water, filter the crude benzanilide with suction on a Buchner funnel, wash with cold water and crystallize from hot alcohol. Dry the product and calculate the percentage yield.

Calculation: Here limiting reagent is aniline; hence yield should be calculated from its amount taken. Molecular formula of aniline =  $C_6H_7N$  Molecular formula of benzanilide =  $C_{13}H_{11}NO$  Molecular weight of aniline = 93 g/mole Molecular weight of benzanilide = 197 g/mole Theoretical yield: 93 g aniline forms 197 g benzanilide Therefore, 2.08 g (2 ml) aniline will form .....%

(X) g benzanilide  $X = 197 \times 2.08 / 93 = 4.4$  g Theoretical yield = 4.4 g Practical yield = ..... g % Yield =  $(\text{Practical Yield} / \text{Theoretical Yield}) \times 100$  Benzanilide was synthesized and the percentage yield was found to be .....%.



(Benzonitrile is colourless crystals, m.p. 163°; yield 3.2 g) Would you like to attempt Labmonk Quiz? Click here Check out Jobs & Exam Notices. Labmonk Notice Board Labmonk Scholarships. Click here Labmonk Blog. Click here Do you need notes? Click hereWatch Career related videos on Youtube. Watch now !!Laboratory Manual of Organic Chemistry by Raj K. Bansal, Page No.111.Practical Organic Chemistry by Frederick George Mann and Bernard Charles Saunders Published by Longan Inc., Fourth Edition; Page No. 245.College Practical Chemistry by V K Ahluwalia, Sunita Dhingra; Published by Universities Press (India) Private Limited 2005; Page No- 235. Also Read: