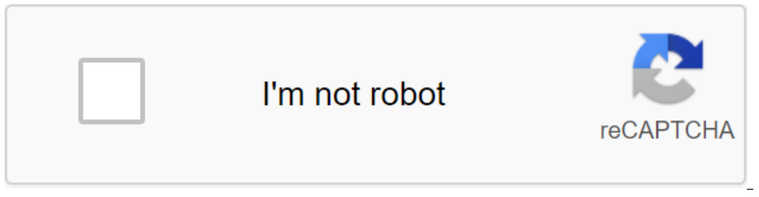

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Preparation of acetanilide from aniline

Preparing acetanilide synthesis and cleaning organic solid purpose In this laboratory we learn many important techniques used in the laboratory of organic chemistry. First, we will test a process called synthesis, where molecules are converted into different products. In this particular synthesis, the aromatic 1 amin (anilin) will be combined with carboxylic acid anhydride (a functional group we have not yet studied) to produce aromatic N-substitute amide (acetanylide) and carboxyl acid (acetic acid). Although synthesis is fundamental to organic chemistry, this laboratory is not primarily related to product formation. Organic chemists call the cleansing of their products after the actual reaction work for synthesis. In this lab we are primarily interested in developing methods to work when the product is solid. Our work will include the decolonization of the unclean product, followed by redistlation and insulation through vacuum filtration. Decolonization is a process by which a small number of deeply colored impurities (chromophors) can be removed from the product. Typically, the material dissolves into suitable solvents and the amount of activated charcoal is added to the chromophore adsorb. The activated charcoal is then filtered and discarded, leaving the decolored product. Recrystalization is the process by which raw (unclean) crystals are cleaned by dissolving in the slightest possible volume of hot solvent. The solvent must be carefully selected. The basic requirement is that crystals are much more soluble when the solvent is hot than when it is cold. The saturated solution is then cooled and the peeled product hardens. These crystals are then filtered and set aside to dry. Procedure In a 125-ml Erlenmeyer flask, measure 2.0 ml of anilin (cautiously! toxic; avoid contact with the skin) from the buret. Add 15 ml of water to the flask. In a small graded cylinder, get 2.5 ml of acetic anhydride. During the swirling flask, add the acetic anhydride to the anylin. Once the raw product forms remove a small sample (about 1 ml of wet solid) with a spatula and place on filter paper on the glass watch to dry. Stick raw acetanilide on this sample. This is the end of the synthesis; now the work begins. Add 50 ml of water and one boiling stone to the remaining flask of raw acetanilide. Heat on a hot plate until all solids are completely dissolved in water. Pour a few ml of solution into a small test tube with markings and set aside. Stick a label on this test tube partially cleaned acetanilide. Add about 1 teaspoon (note the coarse measurment) of activated carbon in a hot flask. Wait until everything is boiling to stop before Add carbon or flask can foam and boil. Bring the flask to a soft boil and whirl from time to time for 5 minutes. Waiting, Waiting, double thickness of fluted filter paper, as your instructor showed. Also arrange to have about 50 ml of hot water boiling water. It would be convenient for several students to share a common hot water flask. Set up a hot filtering by placing a stem funnel over a 250ml glass using an iron ring to support the funnel. Place the fluted filter paper in a funnel and rinse with boiling water to warm the filter paper and funnel. Add the hot solution to the funnel in a few small portions, swirling before each addition. Keep the solution hot by returning the flask to heat as you wait for the funnel to clean. Once the whole solution has been filtered, add 5-10 ml of boiling water to the empty flask, rinse the flask and pour through the funnel to dissolve any crystals that may have formed on the filter paper. Recycle filter paper containing carbon for your instructor' hire. Allow the solution to cool, placing it in a bath with ice water. Do not let the water from the ice bath get into the glass. Waiting for the crystals to form in the glass, filter partially peeled acetanilide using a Hirsch funnel and vacuum flask filter. Place these crystals on a marked watch glass to dry until the next lab. After cooling for 15-20 minutes, the main harvest of the peeled crystals can be insulated on a large Buchner funnel by vacuum filtration. Place the wet filter paper on a marked watch glass and let the crystals dry until the next laboratory period. Recycle waste liquid into a waste container. Watch the appearance of all three cultures of acetanilide. Notice the color, shape and any other details you may be watching. Determine the purity of the samples by carefully measuring the melting point of each in the duplicate. Comment on the relationship between appearance and the apparent purity of the samples. Your notepad findings should include determining the limit of the reagent in this synthesis, calculating the percentage yield (based only on the mass of the large final crop of crystals), the melting points tables obtained for all samples and any relevant comments. Literature Sources calculating limiting reagents and percentage yield Chance Discovery: Acetanilide and invention of Tylenol Aldrich Chemical Company Search Page ChemExper Chemical Catalog Acros Chemical NIST Chemistry Webbook (previously listed as Experiment 23) 1. Anilin is toxic and can be absorbed through the skin. Use in the smoke hood. 2. Concentrated salt acid can cause severe burns. 3. Acetic anhydride is lacchristic. To 60 ml of water in the Erlenmeyer flask 100 ml add 2 ml of concentrated salt acid with mixing. The next step should be taken in the smoke hood. Add 2 ml anilin (density 1.02 g cm-3) and Mixture. If the solution is painted, add a small amount of charcoal decontamination, swirl the flask by about one one and filter carbon using corruged filter paper (see app). In a separate container dissolve 3 g of sodium acetate in 10 ml of water. Heat an ailinia chloride solution to 50 degrees Celsius in a water bath and add 3 ml of acetic anhydride (density 1.08 g cm-3). Stir for the dissolution effect and add aqueous sodium acetate quickly. Swirl the flask a couple of times and set it in an ice bath for 20 minutes. The filter, with suction, amide crystals are formed and washed with a little ice water. Continue to suction into the B chner funnel for a few minutes. Dry the material between the filtered paper and send a sample for evaluation. Determine the yield and melting point of your product. Examine the spectra of I.R. anilin (start material) and acetanilide (product) and fix on the sheet the position of the main bands that differ between them (see annex 3). Link this data to the reaction that occurred. Conduct a nitric acid test on 1 aliphatic amin and on 1, 2 and 3 aromatic amines provided. Keep the results in table shape. Return to Chemistry, UWI-Mona, Created and maintained by Professor Robert Lancashire, Department of Chemistry, University of the West Indies, Mona Campus, Kingston 7, Jamaica. Established in March 1997. Links were verified and/or last changed on March 27, 2012. URL acetanilide is a white organic solid compound used mainly in organic synthesis. N-phenyacetamide, acetanylide and acetanyl are other names of this compound. It has been used in the past to treat fever and headaches and has been known as Antifebrin by its brand. Purpose: Prepare an organic compound of acetanilide from aniline, glacial acetic acid/acetic anhydride and zinc dust. Theory: Acetanilide is cooked from aniline when it reacts with acetic anhydride/glacial acetic acid in the presence of zinc dust. A mixture of anilin, glacial acetic acid, acetic anhydride and zinc dust reflux in the state of angidus, and then poured the mixture into the ice water to get acetic anhydride sediment. The raw sediment of aceidride is redistallized to obtain pure crystals of acetanilid. The chemical reaction is below. The zinc is used to prevent anilin oxidation during a chemical reaction. Acetanilide is medicinally important and is used as febriphus. Acetanylide feces also be prepared by acetylizing aniline with acetic anhydride in the presence of concentrated aldic acid. Dissolve the anilin in the salt acid and add the acetic anhydride to mix well. Pour the mixture onto sodium acetate in water. Acetanilide is produced, which can be and redistallized ethyl alcohol. Other names - N-phenylacetamide, N-phenylethanamid, Acetanyl Materials Materials Anilin Glacial Acetic Acid Acetic Angirid zinc dust distilled water Round lower flask Beaker Pipette Reflux condenser Funnel Stirrer Bunsensurner Filter Paper Electronic Balance Device Setting: Procedure: Wash the entire apparatus with distilled water before the experiment. Take a round lower flask, which add 10 ml of anilin and 20 ml of vinegar anhydride and glacial mixture of acetic acid and add zinc dust. Fix the reflux capacitor with a round lower flask. Heat the mixture gently for 15-20 minutes in an oil bath. Pour the hot mixture into a glass containing ice water with constant stirring. Stir the mixture vigorously to allow hydrolysis to excess vinegar anhydride. Once all the acetanilide is deposited collect and filtered into the funnel the buchner. The resulting sediment represents if a raw sample of acetanilide is called. In order to get clean crystal crystals crystallization must be carried out. Crystallization: Transfer the raw sample to a glass containing 20 ml of water and heat gently. If the solution is painted, add a small amount of activated carbon. Filter a hot solution with a funnel. Cool the mixture for 30 minutes, so that the white shiny crystals of acetanilide separates. Filter the crystals, wash them with water and dry them in folds of filter paper. Observations: Color crystals Colorless Crystals Form Crystals Plate Shape Floating Point 114oC Results and Discussion: The yield of acetanilide is _____gm. Precautions: Do not inhale acetic anhydride fumes. Always experiment in a smoking cell or near a window. Use a water capacitor to reflucs the reaction mixture. Dry the crystals of acetanilide before you find the weight and its melting point Continue to visit BYJU'S to learn more about the chemistry of the class 12 CBSE practical. Acetic anhydride and acetylchlorid are two acetylating agents. The zinc is added to prevent anilin oxidation during the reaction. Reduces color impurities present in the solution. A mixture of concentrated acids, such as nitric acid and sulphuric acid, is called a filathy mixture. The name IUPAC for acetanylide N-phenylacetamide acetanylide is used in the synthesis of penicillin and in other pharmaceuticals. It is also used as an antipyretic agent meaning a lower temperature agent. Agent. preparation of acetanilide from aniline and acetyl chloride. preparation of acetanilide from aniline and acetic anhydride. preparation of acetanilide from aniline mechanism. preparation of acetanilide from aniline reaction. preparation of acetanilide from aniline and its isolation. preparation of acetanilide from aniline using zinc dust. preparation of acetanilide from aniline practical. preparation of acetanilide from aniline discussion

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