Synthesis of Aspirin (Lab #11) Los Angeles Trade-Tech College - CHEMISTRY 102 LABORATORY

In today's lab you will synthesize aspirin from simpler starting materials. After the reaction that creates aspirin, you will purify the aspirin using a technique called *recrystallization*. You will work on a small scale today to avoid creating excessive waste and unpleasant odors.

Fig. 1: Preparation of Aspirin

Procedure:

Part 1: Making Aspirin:

- Prepare a 150 mL beaker about 2/3 full of water. Place it on a hot plate and heat to 80°C.
- Start with a dry 25 ml. Erlenmeyer flask. Place about 600 mg of Salicylic acid in the flask. To this
 flask, add 1.40 ml. of acetic anhydride. Caution! Perform this operation in the fume bood! Acetic
 anhydride is foul-smelling and toxic!
- 3. Add about five drops of 85% phosphoric acid to the mixture. Caution! Phosphoric acid is corrosive! If you get some on you, wash it off immediately with cold water! Clamp the flask and immerse it in the hot water bath for 10 minutes. Swirl the flask occasionally to get the solids to dissolve. The water needs to be hot enough to dissolve the solids, but not boiling hot. Hot tap water is usually enough.
- 4. Remove the flask from the water bath. While the mixture is still hot, add 10-20 drops of water to destroy the excess acetic anhydride. Let stand for five (5) minutes. Then add 5 mL of additional water and let the flask cool to room temperature on its own. Crystals should form after the solution cools.
- If the aspirin hasn't crystallized yet, cool the flask in an ice bath until it does. Collect the solid that forms using vacuum filtration. Wash the solid with 1-2 mL of cold water.

Part 2: Purifying Aspirin:

- Transfer the crude product to a 10 mL (or 25 mL) beaker. <u>Add about 2 mL of ethyl alcohol</u> using a
 plastic pipet. <u>Warm the solution until the aspirin dissolves</u>, then add roughly the same volume of
 water (volume of dissolved crystal + ethyl alcohol) to the mixture. Cover the solution and let it cool.
- After crystals form again, collect them using vacuum filtration. Let the crystals dry for a few minutes before weighing them. You can also press the crystals down on a paper towel to dry them.

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Fundamentals of Analytical Toxicology Robert J. Flanagan, Eva Cuypers, Hans H. Maurer, Robin Whelpton, 2020-05-21 Fundamentals of Analytical Toxicology is an integrated introduction to the analysis of drugs poisons and other foreign compounds in biological and related specimens Assuming only basic knowledge of analytical chemistry this invaluable guide helps trainee analytical toxicologists understand the principles and practical skills involved in detecting identifying and measuring a broad range of compounds in various biological samples Clear easy to read chapters provide detailed information on topics including sample collection and preparation spectrophotometric and luminescence techniques liquid and gas liquid chromatography and mass spectrometry including hyphenated techniques This new edition contains thoroughly revised content that reflects contemporary practices and advances in analytical methods Expanding the scope of the 1995 World Health Organization WHO basic analytical toxicology manual the text includes coverage of separation science essential pharmacokinetics xenobiotic absorption distribution and metabolism clinical toxicological and substance misuse testing therapeutic drug monitoring trace elements and toxic metals analysis and importantly the clinical interpretation of analytical results Written by a prominent team of experienced practitioners this volume Focuses on analytical statistical and pharmacokinetic principles Describes basic methodology including colour tests and immunoassay and enzyme based assays Outlines laboratory operations such as method validation quality assessment staff training and laboratory accreditation Follows IUPAC nomenclature for chemical names and recommended International Non proprietary Name rINN for drugs and pesticides Includes discussion of designer drugs novel pharmaceutical substances NPS Fundamentals of Analytical Toxicology Clinical and Forensic 2nd Edition is an indispensable resource for advanced students and trainee analytical toxicologists across disciplines such as clinical science analytical chemistry forensic science pathology applied biology food safety and pharmaceutical and pesticide development The Tubby Traveler from Topeka Brian S. Edwards, 2012-04-27 The Tubby Traveler from Topeka The Handbook of Research Synthesis Harris Cooper, Larry V. Hedges, 1993-11-23 The Handbook is a comprehensive treatment of literature synthesis and provides practical advice for anyone deep in the throes of just teetering on the brink of or attempting to decipher a meta analysis Given the expanding application and importance of literature synthesis understanding both its strengths and weaknesses is essential for its practitioners and consumers This volume is a good beginning for those who wish to gain that understanding Chance Meta analysis as the statistical analysis of a large collection of results from individual studies is called has now achieved a status of respectability in medicine This respectability when combined with the slight hint of mystique that sometimes surrounds meta analysis ensures that results of studies that use it are treated with the respect they deserve The Handbook of Research Synthesis is one of the most important publications in this subject both as a definitive reference book and a practical manual British Medical Journal The Handbook of Research Synthesis is the definitive reference and how to manual for behavioral and medical scientists applying

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