Journal of Visualized Experiments

Syntheses, crystallization and spectroscopic characterization of 3,5-lutidine N-oxide dehydrate

Ref. JoVE57233R1

Dear Editor Bing Wu,

Thank you for your comments on our manuscript. We have considered fully your concerns and we hope to have appropriately answered all them; this is a fully revised manuscript which we are resubmitting to Journal of Visualized Experiments.

We have modified the manuscript accordingly, and detailed corrections are listed below point by point.

1. There are still some protocol steps which are not in imperative tense. Please rewrite those steps

In page 3

line 92

1.2 Cool the flask to 24 °C, after the reaction time, with ice to make it manageable without exposure to the acetic acid gases, and plugged it to a high vacuum distillation unit for 90 to 120 min to remove excess acetic acid. Caution, do not use hot material, wait until the glassware reaches a manageable temperature, this will also avoid that the vapors up stream in top of distillation unit.

Line 98

* 1. Add distilled water (10 mL) twice to ensure the removal of any trace of acetic acid and to concentrate the mixture as far as possible.

Line 106

2.2 Place carefully, the solution in a separation funnel of 250 mL and wash it 5 times with 250 mL of CHCl3 to improve the yield. Recover and dry over solid Na2SO4 for 30 min maximum the organic phase, which containing the product. If necessary re-extract the aqueous phase with the desired amount of CHCl3.

Line 113

2.3 Remove the solvent under reduced pressure with a high vacuum distillation unit, until the formation of a very hygroscopic clear beige crystalline powder (70%).

Line 121

… pour the filtrated out liquid into a glass Petri dish, leaving it to slow evaporation at 4 °C in a laboratory fridge.

Line 124

3.2 Ensure that, after two days, are obtained clear colorless crystals. Then measure the melting point which has to be in the range of 310–311 K.

Line 129

4.1 Remove by decantation from the flask’s walls the crystals that are formed, of prismatic shape and colorless, for further X-ray analysis. Keep them into diethyl ether to avoid crystals hydration in case of not immediately use.