4-Nitroacetanilide

Reagents:

1. acetanilide 10g
2. nitric acid 4,4 ml
3. sulphuric acid 20 ml + 2,8 ml
4. glacial acetic acid 10 ml
5. ethanol or methanol 40 ml

Each activity/operation must be done under ventilating fan.

In a high, 200 ml beaker place 10 g of fine powdered acetanilide, add 10 ml of glacial acetic acid and while stirring vigoriously add sulphuric acid. The reaction mixture is heating up and the solution becomes colourless. Place the beaker in ice-salt bath stirring the reaction mixture mechanically. Over the beaker place the addition funnel containing cooled solution of 4,4 ml of nitric acid and 2,8 ml of concentrated sulphuric acid. When the temperature gains 0-2^{o}C start the gradual addition of acid mixture. The temperature should not rise above 5-10^{o}C. After the whole amount of nitrating mixture is added, the beaker should be left in room temperature for an hour. Than pour the reaction mixture into 100 g of grinded ice in 200 ml of water. Harsh nitroacetanilide crystallizes immediately. After 15 minutes, drain the product off using Buchner filter flask. Wash the precipitate with cooled water until all acid is removed (check the acid reaction of filtrate). Imprint the product and dry[1]. Recrystallize the light yellow precipitate from ethanol or methanol, filter it under reduced pressure and wash with small amount of well cooled alcohol. Leave nitroacetanilide to dry on a filter paper.

Yield of colourless, crystallic 4-nitroacetanilide is 60% (8g)[2]. Melting point is 214^{o}C.

[1] The most efficient way of washing the harsh precipitate is by removing it into beaker with cold water, with stirring the mixture. After that, filter the product once again.
[2] This compound is a reagent in synthesis of 4-nitroaniline.

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