

6. Experiment name: Preparation of Barbituric acid

Aim: To prepare barbituric acid from urea and diethyl malonate.

References: <https://labmonk.com/synthesis-of-barbituric-acid-from-urea-and-dimethyl-malonate>

Requirements:

Chemicals: Sodium metal, Ethanol, Diethyl malonate, Urea, Calcium chloride, Concentrated hydrochloric acid

Apparatus: Round bottom flask – 2000 ml, Reflux condenser, Beaker, Buchner funnel, Measuring cylinder, filter paper

Procedure:

Assemble a double surface reflux condenser with a 2 litre round bottomed flask, place 11.5g of clean sodium. Mix 250 ml of absolute ethanol in a portion and if the reaction is unduly vigorous, immerse the flask within ice. When all the sodium has completed reaction, add diethyl malonate 80 g (76 ml), followed by a solution of dry urea 30 g in 250 ml of hot (70 °C) absolute ethanol. Shake the mixture thoroughly, attach a calcium chloride guard tube to the top of the condenser, start reflux of the mixture for 7 h in an oil bath and heat to 110 °C. A white solid will be separated. Treat the reaction mixture with hot (50 °C) water 450 ml and then with concentrated hydrochloric acid, with constant stirring, until the solution will be acid (about 45 ml). Filter the resulting almost clear solution and leave it in the refrigerator overnight. Filter the solid at the pump, wash it with 25 ml of cold water, drain well and then dry at 100 °C for 4 hours. The yield of barbituric acid is 50 g. It melts with decomposition at 245 °C.

Principle:

The synthesis of barbituric acid is affected by condensation of diethyl malonate with urea in the presence of sodium ethoxide which may be prepared by reacting Na metal with ethanol and it undergo cyclization reaction with diethyl malonate.

Reaction:

