Experiment: 07

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

REFERENCES

1. Vogel's Textbook of Practical Organic Chemistry by Brian S. Furniss, Antony J. Hannaford, Peter W. G. Smith & Austin R. Tatchell; Fifth Edition; Page No. 1176.

Requirements:

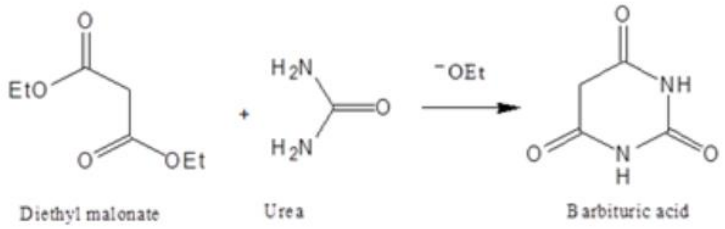
Apparatus: 2000 ml round–bottom flask, Reflux condenser, Beaker, Buchner funnel, Measuring cylinder, Filter paper, etc.

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

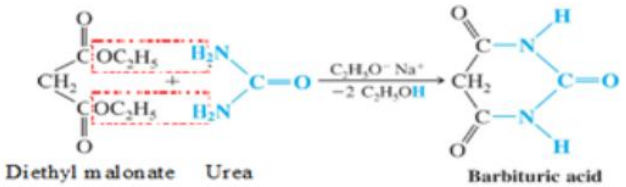
Theory:

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. It undergoes a cyclisation reaction with diethyl malonate.

Chemical Reaction:



Mechanism:



Use:

It is not active pharmacologically, but its derivatives are used as sleeping pills and sedatives.

Procedure:

1. Set up a double surface reflux condenser with a 2-liter round bottomed flask and place 11.5 g (0.5 mol) of clean sodium in it.

2. Mix 250 ml of absolute ethanol in the flask. If the reaction becomes too vigorous, place the flask in an ice bath.
3. Once the sodium has fully reacted, add 80 g (76 ml, 0.5 mol) of diethyl malonate, followed by a solution of 30 g (0.5 mol) of dry urea in 250 ml of hot (70 °C) absolute ethanol. Shake the mixture thoroughly.
4. Attach a calcium chloride guard tube to the top of the condenser, then start refluxing the mixture for 7 hours in an oil bath heated to 110 °C. A white solid will form.
5. Treat the reaction mixture with 450 ml of hot (50 °C) water, followed by concentrated hydrochloric acid while stirring constantly until the solution becomes acidic (about 45 ml).
6. Filter the resulting almost clear solution and leave it in the refrigerator overnight.
7. Filter the solid using a pump, wash it with 25 ml of cold water, drain well, and dry it at 100 °C for 4 hours.
8. The yield of barbituric acid should be 50 g (78%). It melts with decomposition at 245 °C.

Calculation:

Here, the limiting reagent is diethyl malonate; hence, yield should be calculated from the amount taken.

The molecular formula of diethyl malonate = $C_7H_{12}O_4$

The molecular formula of barbituric acid = $C_4H_4N_2O_3$

The molecular weight of diethyl malonate = 160 g/mole

The molecular weight of barbituric acid = 128 g/mole

Theoretical yield:

160 g diethyl malonate forms 128 g barbituric acid

Therefore, 80 g diethyl malonate will form …….? (X) g barbituric acid

X = (128 ×80)/160 = 64 g

Theoretical yield = 64 g

Practical yield = ————- g

% Yield = (Practical Yield)/(Theoretical Yield) × 100

Result:

Barbituric acid was synthesized and the percentage yield was found to be……..%