

C 241 (Experiment. No. _____)

NAME: _____

DATE: _____

ROLL NO: _____

SEMESTER _____

SIGNATURE: _____

SYNTHESIS OF 3,5-DIMETHYLPYRAZOLE

AIM

To Synthesize 3,5-dimethylpyrazole from acetylacetone.

APPARATUS

Round bottom flask, condenser, guard tube, septum, syringe (or dropping funnel), measuring cylinder (25 mL, 10 mL), thermometer, retort stand, melting point apparatus and tubes, rotavapor apparatus, ice-bath, buchner funnel etc.

CHEMICALS REQUIRED

Acetyl acetone, Hydrazine hydrate, ethyl alcohol, n-hexane.

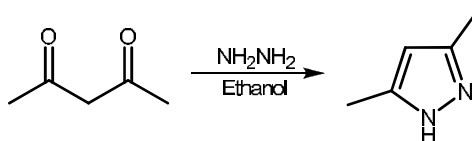
PROCEDURE

1. Take 3 mL (~60 mmol) of hydrazine hydrate in a 250 mL flask. To this add 25 mL of ethanol with constant stirring.
2. Now place the flask in an ice-old water or ice-bath and wait for 10 min.
3. Add slowly 5 mL (~50 mmol) of acetylacetone drop wise to the above solution at low temperature with constant stirring.

NOTE: The addition of acetylacetone requires about ~20 min. to complete.

4. Allow the reaction mixture to come to room temperature and then reflux for an hour (Oil-bath temperature ~110°C). Then remove the solvent to dryness on rotavapor and add few mL of n-hexane to dissolve the solid in lukewarm condition. Place the flask in refrigerator to get crystalline solid. Collect the solid product by filtration. You may use cold hexane to wash the product.
5. Perform all required calculations and submit the product.

CHEMICAL REACTION



OBSERVATIONS AND CALCULATIONS

1. Calculate the yield and % yield.
2. Determine the melting point of the product.
3. Record the NMR for the sample obtained and comment on the results.

RESULTS

Yield: _____

%Yield: _____

M.P.: _____