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Department of Chemistry
Indian Institute of Technology Guwahati
Guwahati-781039, Assam
CH 110 Laboratory report

Experiment No. 2

Reaction of 4-aminotoluene and o-vanillin

(Preparation of Azomethine)

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Marks Distribution

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Signature of Student अर्पित जैन

Date: 28/08/2018

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Signature of Invigilator/TA

Signature of Coordinator

Objective

To prepare Azomethine by the reaction of 4-Aminotoluene and O-Vanillin. And read Melting Point of the Compound and calculate the Percentage yield.

Theory & Mechanism

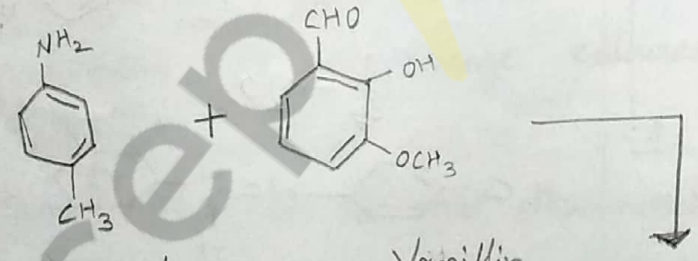
Imines are typically prepared by the Condensation of primary amines and aldehydes and less commonly ketones.

In term of Mechanism, Such reactions prepare via the nucleaphillic addition giving a hemiaminal $+C(OH)(NHR)-$ intermidiate followed by a elimination of water to yield the imine. The equilibrium in this reaction usually favors the Carbonyl compound & amine, So that azotropic distillation or uses of a dehydrating agent, Such as molecular V or magnisium sieve

Sulfate, is required to push the reaction in favor of imine formation. In recent years, Sellral reagent such as $[B(OCH_2CF_3)_3]$ or $[Ti(OEt)_4]$ have been shown to catalysis imine formation.

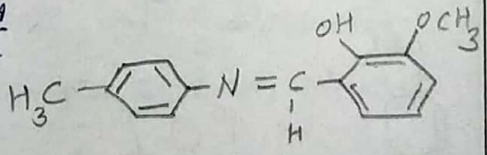
This reaction actually shows that few covalent bond forming bi-molecular organic transformation that proceid rapidly and to a high degree of completion between the solid reactant. actually accur in the solid state.

The typical reaction is as follows.



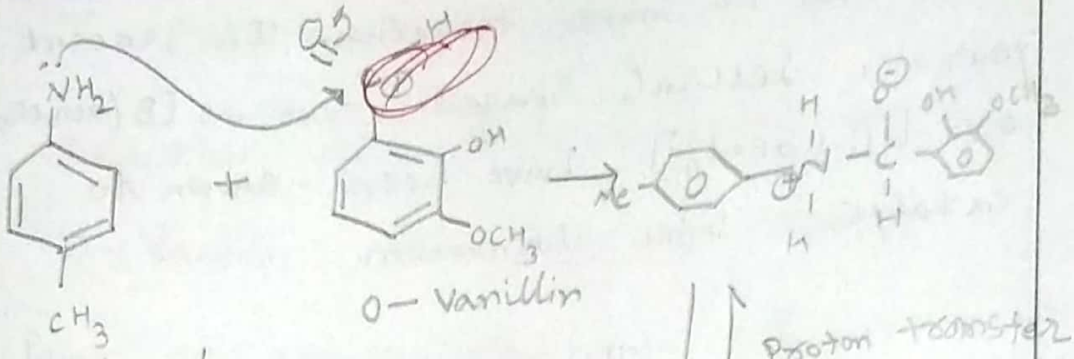
4-Aminotoluene o-Vainillin

R_f Value = $\frac{\text{distance moved by compound}}{\text{distance moved by solvent}}$



Azomethine

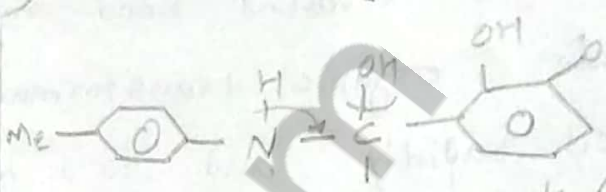
Mechanizm :-



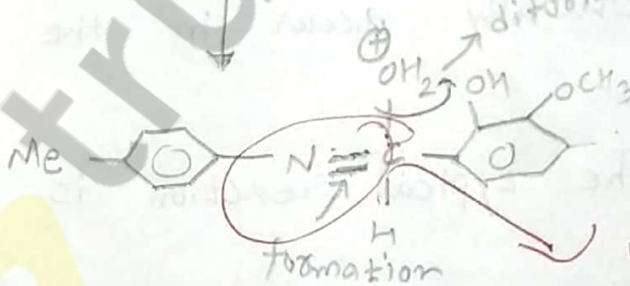
4-Amino-toluene
(P- Amino, toluene)

O- Vanillin

Proton transfer



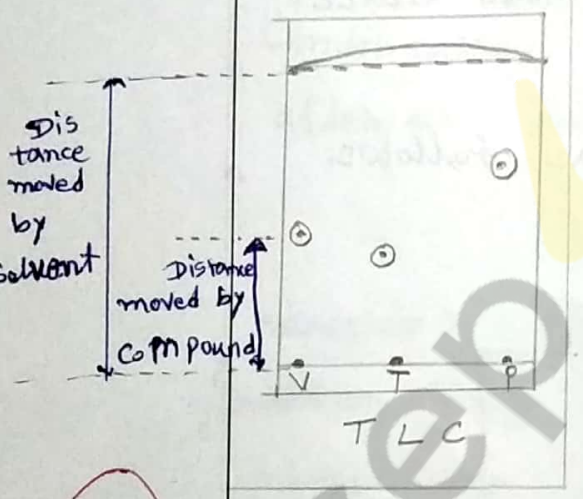
Unstable Add^{n} product



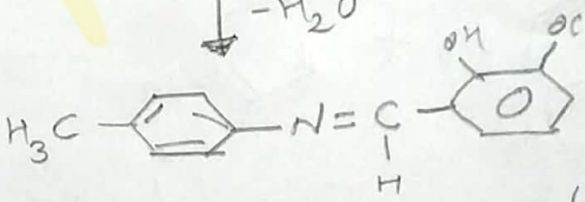
dihydration

5 valency

formation

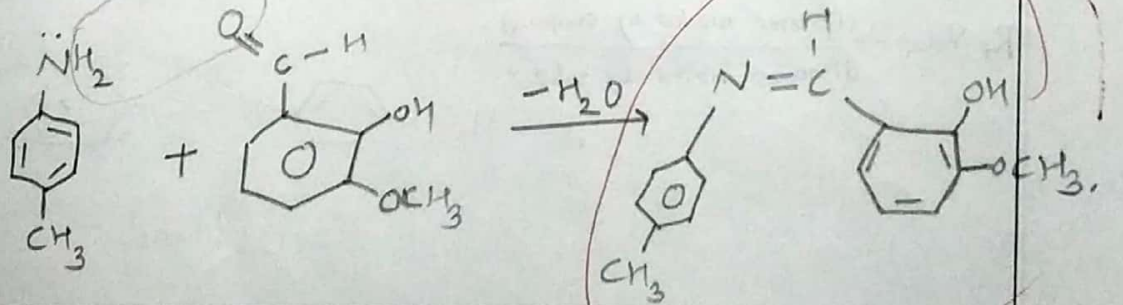


$-H_2O$



Azomethine (orange colour)

Shortly we can say that,



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Observation

Weight of 4-Aminotoluene used = 0.300g

Weight of o-Vanillin used = 0.430g

Weight of Azomethine formed = 0.633g

Melting point Observed = 81°C

TLC Observation :-

Distance moved by 10% ethylacetate in Hexane solution = 2.95cm

Distance moved by Azomethine = 1.6 cm

Distance moved by 4-Aminotoluene = 1.05 cm

Distance moved by o-Vanillin = 1.15 cm

After mixing 4-Aminotoluene and o-Vanillin we get an orange liquid phase immidiately.

After mixing we get an orange coloured homogenius liquid (Azomethine), which crystalies to an orange coloured solid rapidly.

During TLC we see that Azomethine travels maximum distance being least polar and 4-Aminotoluene travels most distance being most polar. In 10% ethylacetate in hexane solution Azomethine is a orange coloured, plesent smelling compound.

Solvent System !!

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Calculation

Molar Mass of 4-Aminotoluene = 107 g/mol

Moles used of 4-Aminotoluene = $\frac{0.300}{107}$ = 2.80 m.mol

Molar Mass of O-Vanillin = 152 g/mole

Moles used of O-Vanillin = $\frac{0.430}{152}$ = 2.82 m.mol

Molar Mass of Azomethine = 241 g/mol

Theoritical moles of Azomethine = 2.80 m.mole

Theoritical yield (mass) = $2.80 \times 10^{-3} \times 241$ = 0.675g.

Experimental mass = 0.633 g.

Yield % (mass) = $\frac{0.633}{0.675} \times 100$ = 93.77 %

Experimental moles = 2.62 m.mole.

Yield % (moles) = $\frac{2.62 \times 10^{-3}}{2.80 \times 10^{-3}} \times 100$ = 93.77 %

Rf of 4-Aminotoluene = $\frac{1.05}{2.95}$ = 0.355

Rf of O-Vanillin = $\frac{1.15}{2.95}$ = 0.389

Rf of Azomethine = $\frac{1.6}{2.95}$ = 0.542

Result

Mass of Azomethine obtained = 0.633g.

The yield of Azomethine is = 93.77%

Rf for 4-Aminotoluene = 0.355

Rf for O-Vanillin = 0.389

Rf for azomethin = 0.542

Melting point of Azomethine = 81°C

Precautions

1] Melting point should be noted carefully.

2] While performing TLC, the paper should not be disturbed.

3] And while you are using the UV machine hand should be kept away.

4] Two powder when mixed, should be mixed with glass rod only.

5] While using the UV machine, don't insert whole hand as UV rays are harmful for skin.

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Applications

1] Azomethine and Azo compounds are used in synthesis of Nitrogen containing Compounds.

2] Azomethine is widely used as in Synthesis of martinellic acid

3] It is also widely used in the synthesis of benzadiazepinones.

4] It's also used in total synthesis of Spirotryprostatin A.

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