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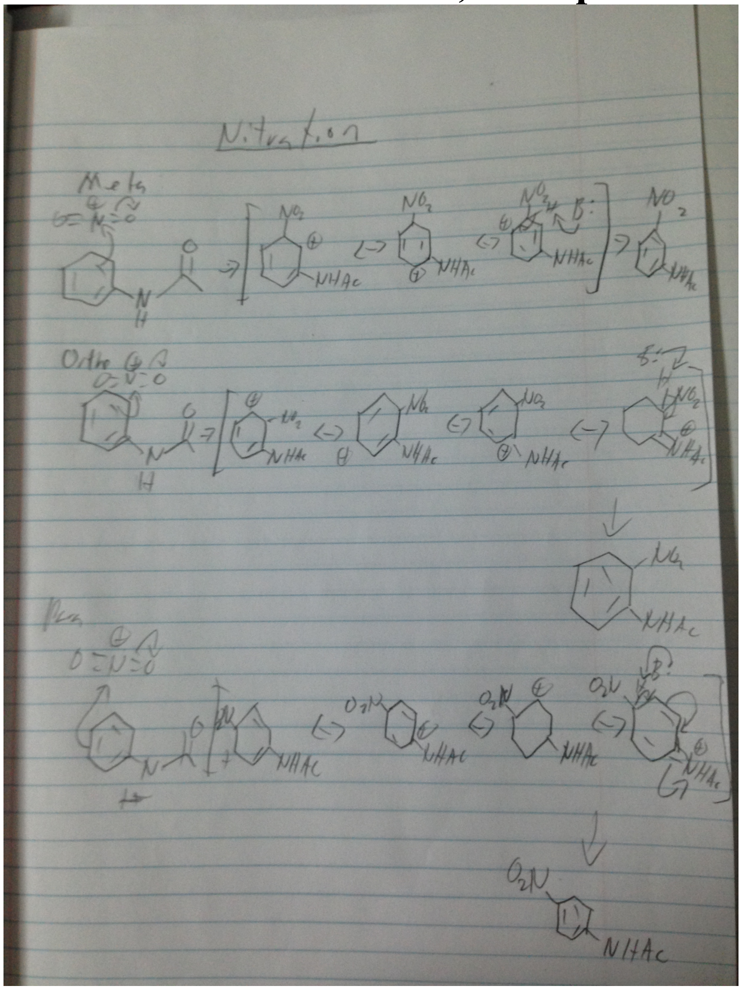
Section:A1

Experiment 6: Regioselective Nitration of Acetanilide

Introduction

Mechanism reaction or o, m or p



Procedure

See laboratory manual Introductory Organic Chemistry 2017 Pg 34 to 42

Observations

In step 6 when the solutions are mixed the solution turned into a brownish yellow colour, as time goes by it turned very dark brown.

In the isolation of crude product, a yellow precipitate has formed, the precipitate looks like salt. There was a small top layer of above the precipitate, clear solution.

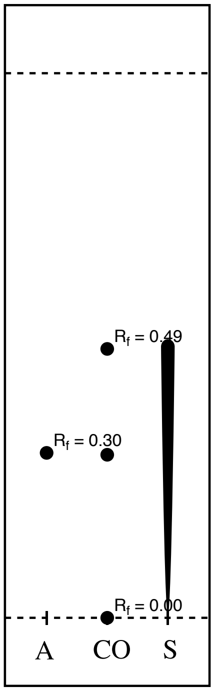
When the precipitate was collected by suction filtration the solid look like yellow chalked. While the filtrated solution was transparent orange yellow colour.

On step 16 when forming the crystals, the solution was cloudy with yellow precipitate.

When the crystals were filtrated, crystals look like yellow powder, the mother liquor had yellow precipitate.

TLC

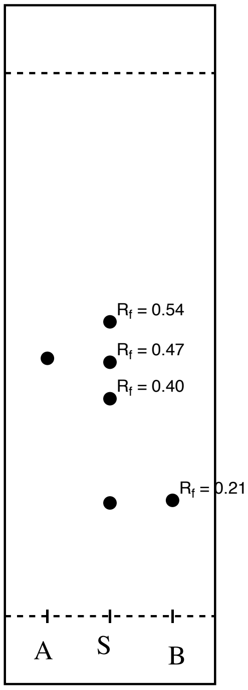
Diagram 1: **TLC Plate with Acetanilide EtOAc: Hexanes Solvent System**



Solvent front
Solvent system:
EtOAc: Hexanes
5:5

A-Acetanilide
CO- Co-spot
S-Sample

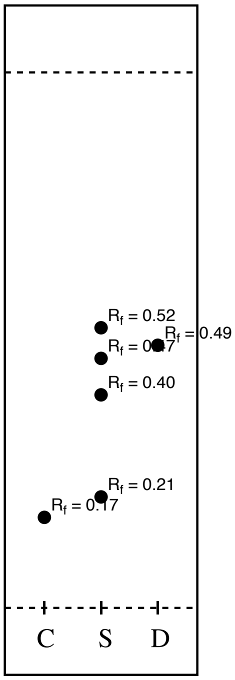
Diagram 2: **TLC Plate with Sample ,EtOAc: Hexanes Solvent System**



Solvent front
Solvent system:
EtOAc: Hexanes
5:5

A-Ortho
S-Sample
B-meta

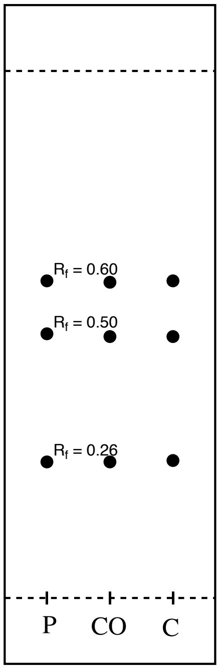
Diagram 3: **TLC Plate with Sample EtOAc: Hexanes Solvent System**



Solvent front
Solvent system:
EtOAc: Hexanes
5:5

C-Para
S-Sample
D-2,4-Dinitro

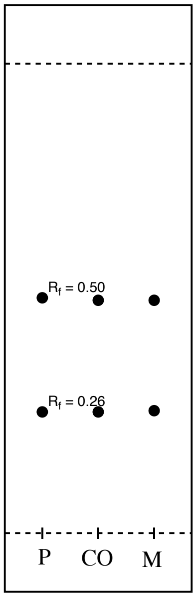
Diagram 4: TLC Plate with Sample , purified product and crude EtOAc: Hexanes Solvent System



Solvent front
Solvent system:
EtOAc: Hexanes
5:5

P-product
CO- Co spot
C-crude

Diagram 5: TLC Plate with Sample, purified product and mother liquor EtOAc: Hexanes Solvent System



Solvent front
Solvent system:
EtOAc: Hexanes
5:5

P-product
CO- Co spot
M- mother liquor

Table of results

Product	Amount(g)	Molecular weight(g/mol)	#Mol	%Yield mol
acetanilide	1.02	135.17	0.0099	132

Calculation

%yield

%yield=(# mol of product)/(# mol of starting material)x100

=(0.0099)/(0.0075)x 100%

=132%

Calculating %mole of the unknown using image J

Calculate percentage of absorbance

Absorbance of ortho=5204.903

Absorbance of para =2293.569

%Absorbance(m)=

5204.903/(5204.903+2293.569+10883.178)x100

= 28.32%

% Absorbance(p)=

2293.569/(5204.903+2293.569+10883.178)x100

=12.48

Calculate the mole percentage

Y= %absorbance(m)=69.41

$y = 7.9391E\text{-}03x^2 +1.6962E\text{-}01x +3.3788$ ortho para mixture equation

isolate x

%mole(m)=45.37

%mole(p)=54.62

Discussion

In step 6 TLC was taken the reaction was complete, the results was in diagram 1, because acetanilide was not in the sample. The Rf value of acetanilide did not match any Rf value indicating that acetanilide is not present anymore and the reaction is complete.

In step 10 we are determining if the crude product contained ortho, meta, para, and 2,4-dinitro by TLC, the results are in diagram 2 and 3.

Diagram 2 indicated that the crude contained ortho and meta because in the ortho lane(reference) was Rf of 0.47 matching one of the Rf in the sample indicating that it had ortho. The same for meta, it had 0.21 Rf value matching one of the spots of the sample indicating that it also contained meta. Diagram 3 similar results were obtained but the Rf of the reference was not as close as the ortho and meta TLC concluding that in diagram 3 para and 2,4-dinitro was not in the solution.

In diagram 4 and 5 it was comparing with purified product and crude(diagram 4) and purified product and mother liquor. For both results separation was not successful because each lane has the same number of spots and the same Rf value.

The percent yield that was obtained was 132%, the reason that it could be higher than 100% is that maybe the filtration of the crude was to short, need more time to dry.

Questions

1. **Explain the difference in polarity of the *ortho* and *para* isomers of nitroacetanilide with respect to the TLC, commenting on why one compound has moved further than the other.**
   The ortho isomers is less polar than para because ortho has a greater Rf than para, travelling a further distance, less attraction to the TLC plate greater Rf. Para isomer has a greater polarity because it has less Rf value meaning it is very attracted to the TLC resulting to less distance travelled.
2. **Why is the addition of a second nitro group in this reaction much slower than the first nitration of acetanilide?**
   The greater the electron density of the aromatic rings the easier and faster it will occur. By adding a second nitro it slows down because it removes the electron density by taking of elctrons causing it to be less electrophilic

3**. Why are *para* isomers favored over *ortho* isomers in electrophilic aromatic substitution?**

It has less steric interaction which causes addition on onto the ring causing more space. It also there is no dipole moment because the groups are in the opposite side of the ring while ortho has groups on the same side of the ring.

4. **What is the preferred position of nitration for the following?**

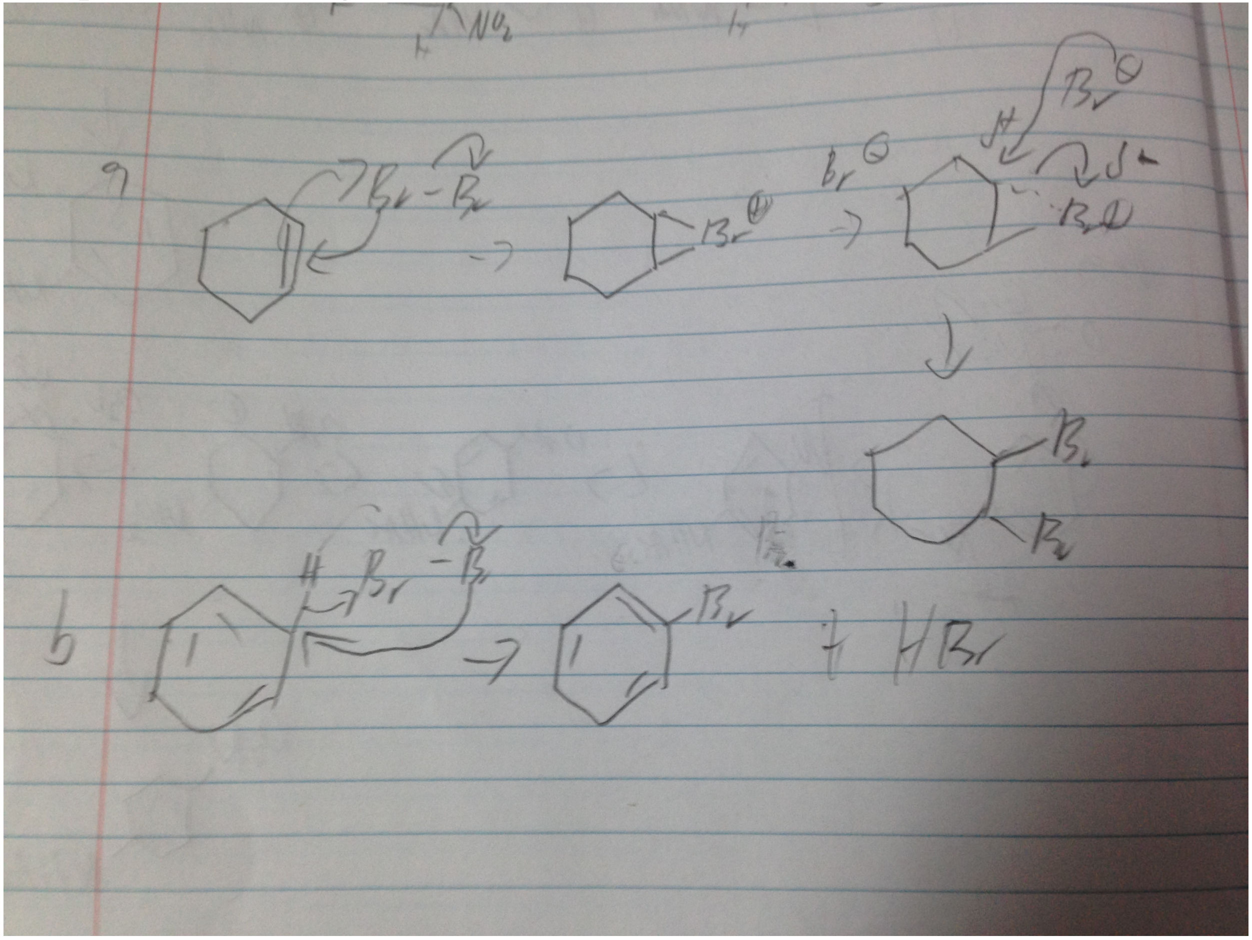
a. para isomer configuration

b. meta isomer configuration

c. ortho isomer configuration

d. the pi bonds of the structure

5. **Explain the following observation**



6. When 780 g of benzene (Molar mass = 78 g/mol) reacts with 750 mL of concentrated HNO_3 (16 M) and 750 mL of H_2SO_4 (18 M), 1000 g of nitrobenzene (molar mass = 123 g/mol) and 250 g of an unknown compound with a high boiling point are obtained.

a. **Identify the limiting reagent**.

benzene

b. **Calculate the percent yield of nitrobenzene**.

%yield=(# mol of product)/(# mol of starting material)x100
=(8.13mol)/(10mol)x 100%

=81.3%

c. **Give the name of the most probable side product with the high melting point.**
crystaline

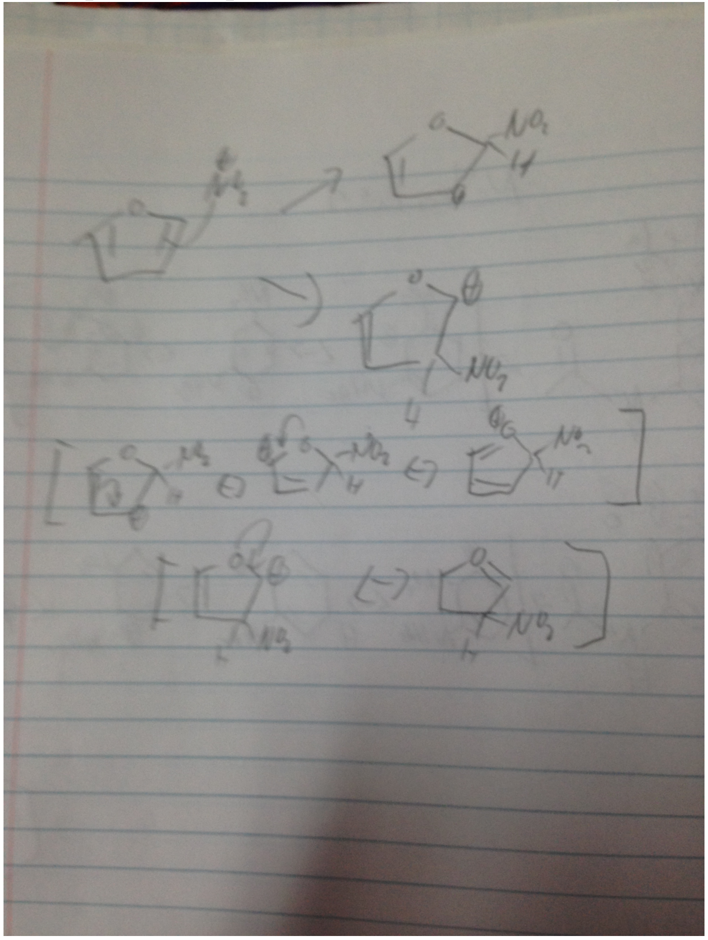
d. **Explain, with the aid of a mechanism, the role of H_2SO_4 in the reaction.**
it deprotonate the benzene ring

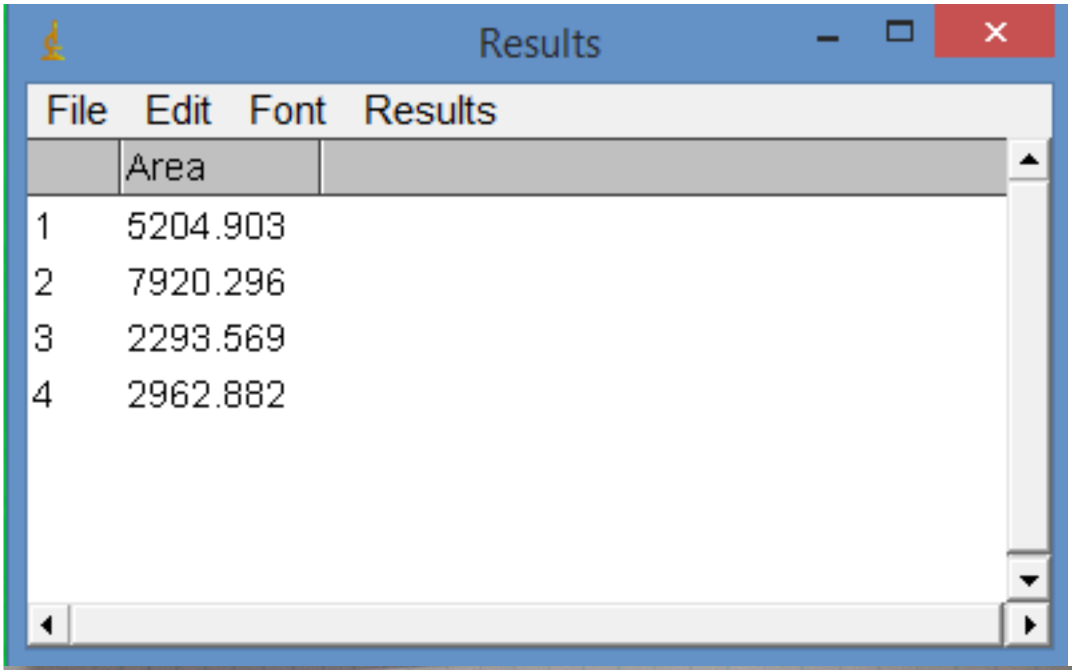
e. Supposing the crystalline compound (250 g) has the formula $C_6H_4N_2O_4$, calculate the yield of this side product.

%yield=(# mol of product)/(# mol of starting material)x100
=(1.48mol)/(10mol)x 100%
=14.8%

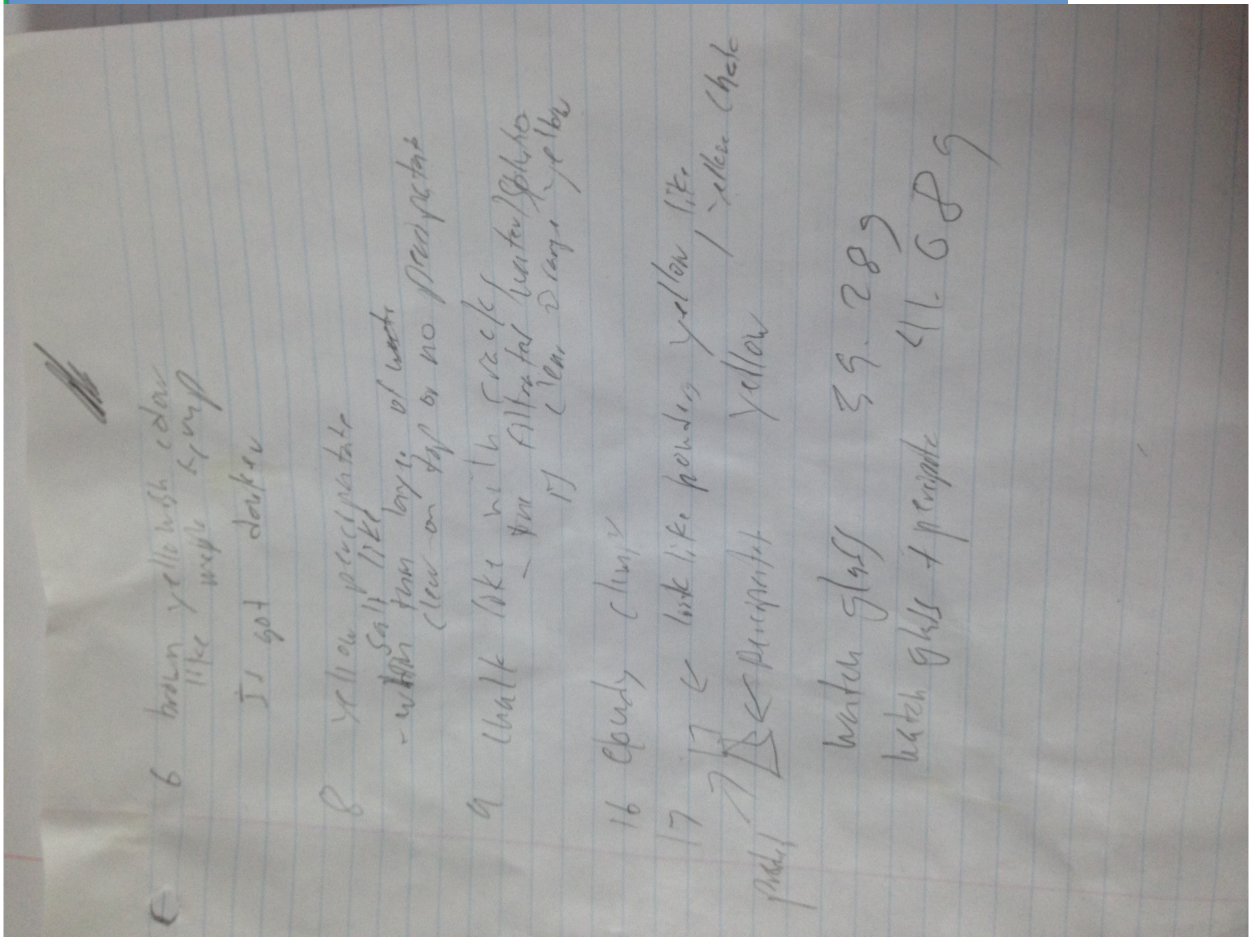
7.nitration of furan leads preferentially to 1- The nitrofuran rather than the 2-nitrofuran isomer. Explain by considering carefully the stability of the σ-complex leading to each isomer.

The 1 position is preferred because it can undergo more resonance structure than the 2-position





	Area
1	5204.903
2	7920.296
3	2293.569
4	2962.882



E

6 brown yellowish color
like maple syrup

It got darker

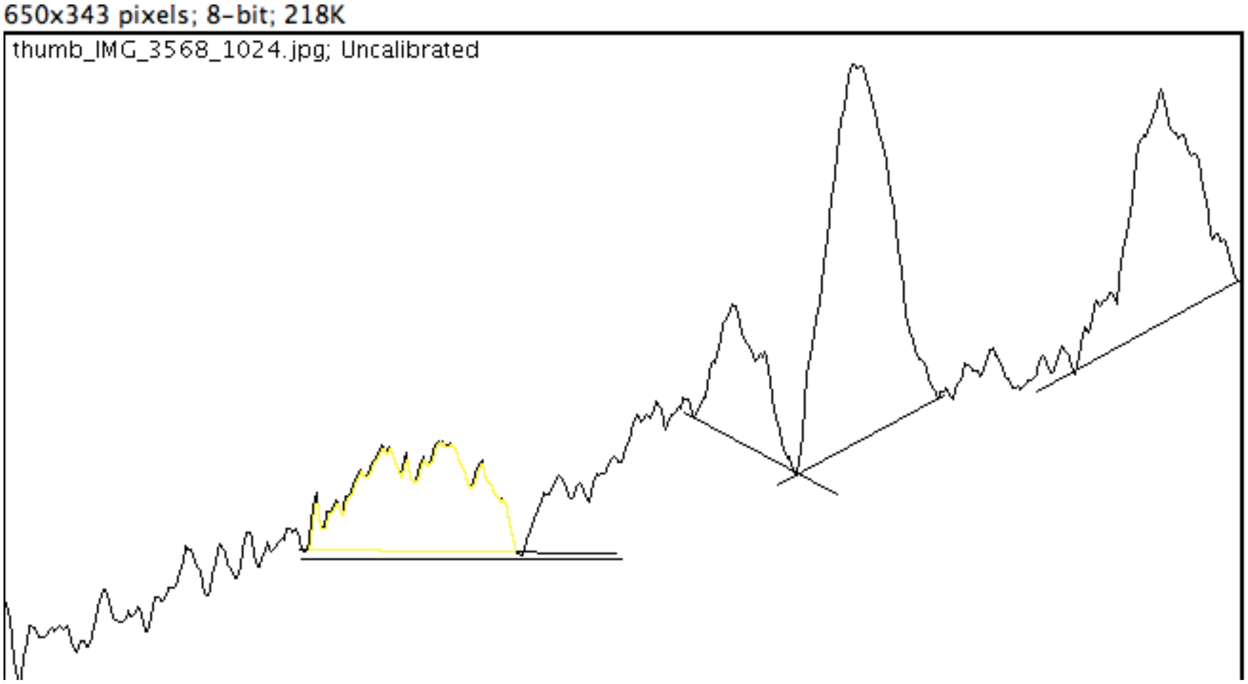
8 yellow precipitate
salt like
- when thin layer of water
clear on top or no precipitate

9 chalk like with cracks
- the filtrate water/solution
is clear orange yellow

16 cloudy clumpy

17 ← look like powder yellow like
/ yellow chalk
product ← precipitate yellow

watch glass 39.28 g
watch glass + precipitate 41.68 g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