CARNICOM INSTITUTE LEGACY PROJECT

A Release of Internal Original Research Documents

Authored
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5 SUBJECT

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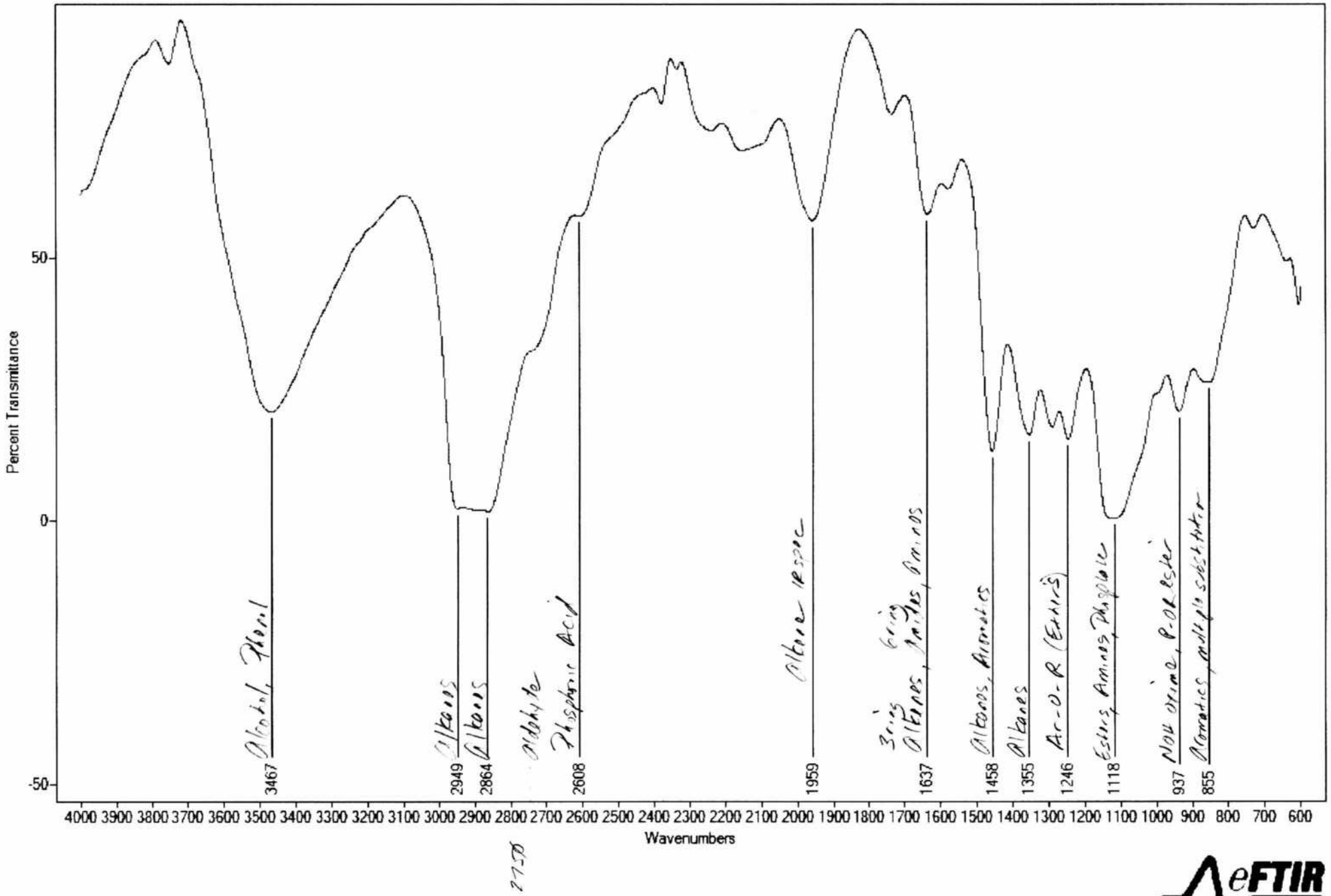
Chemistry Vol XI

August 2015

[redacted] Nov 11 2015 - 01.spc: Synthetic spectrum by averaging. See audit trail for details.

Page A



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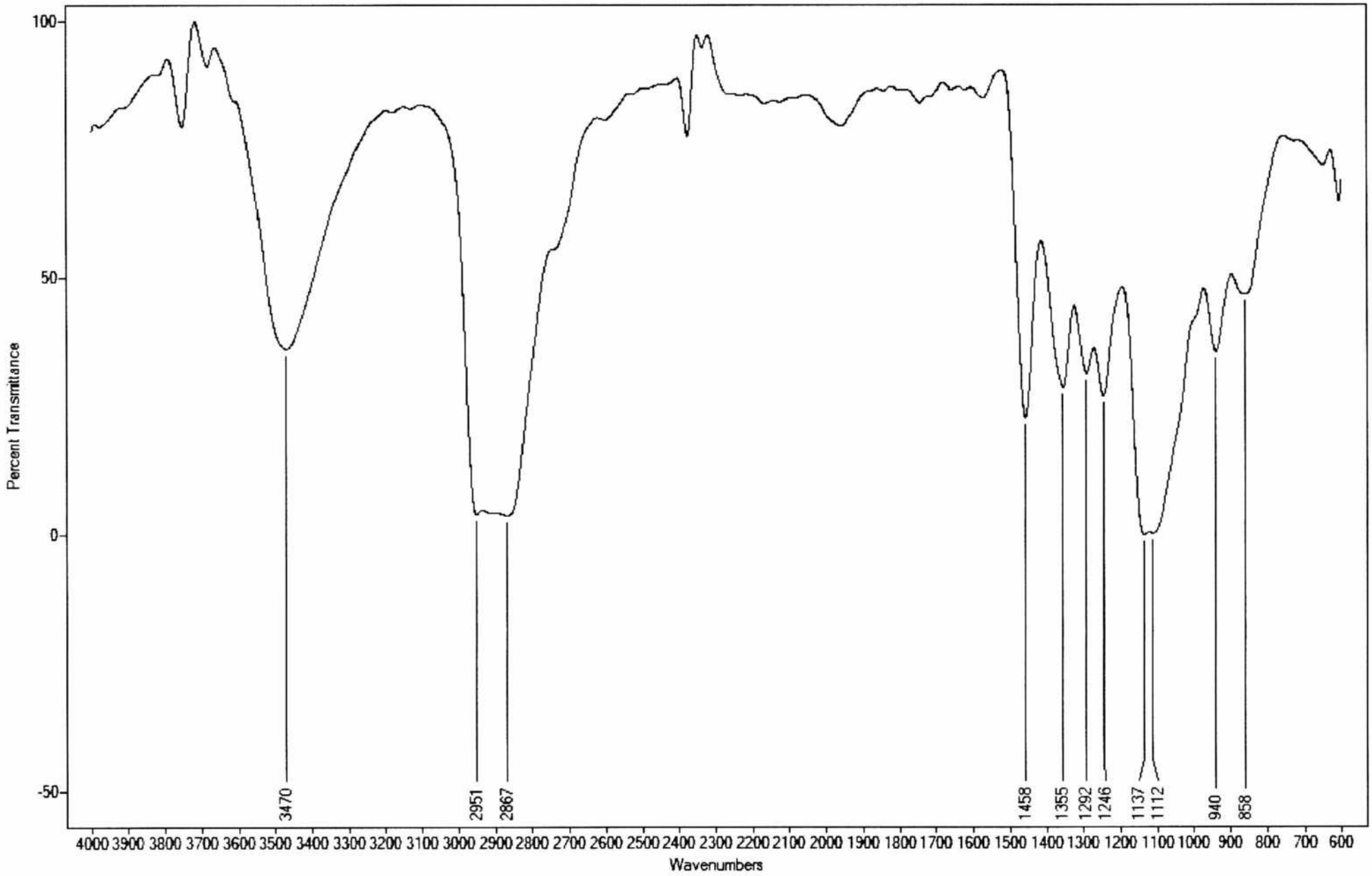
Page B



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August 29 2015

Last night we had our first successful
1g fluid run and separation.
Campto phenique in naptha @ 250° for 20 min.

Today we start variations on the theme.
First run today is @ 200° for 25 min.

Very interesting and marginal results.

The first peak formed (large peak) abnormally
and the second (largest peak) only made it
the top before running out of time.

So we learn here that temp is crucial
and that we do not have effective separation
here. We never would have known anything
@ 150°C or so it an guessing. We also lost
the first peak (small one).

The lesson is that temperature can make a
big difference. The detector can only be heated
to 275° so we really can't go any higher.

It can be seen that a case of double ya
may need to try more of an hour or more.

—

Now you are back up to 250°C.
But you did not get enough sample in.

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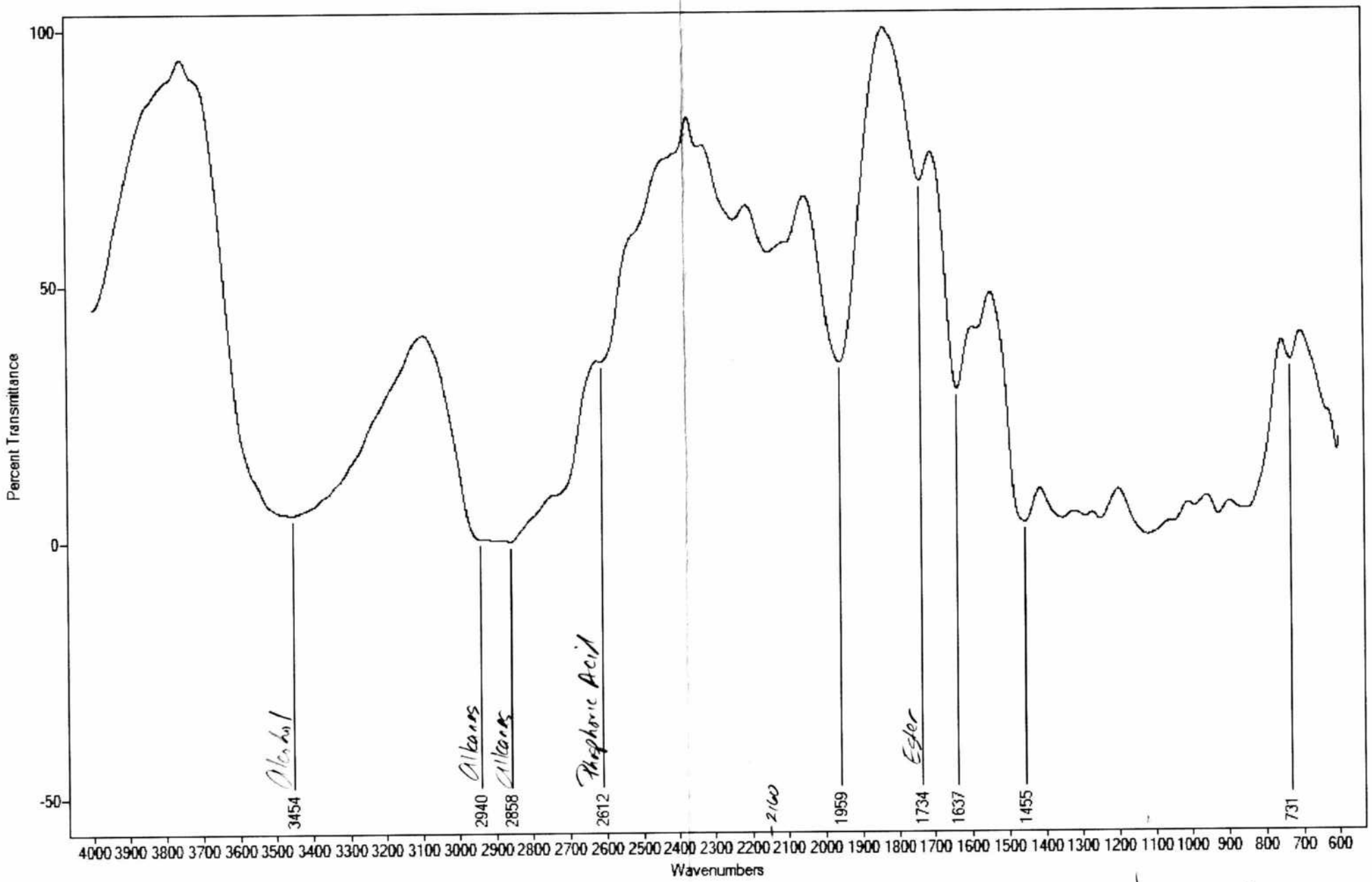
1.459 Index of Refracti-

Page C



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X=2959

1125

eFTIR

Page 2

Ethanol fast on non polar column

Camphor is a C10.
Boiling Point is about 210°C so there is why it was causing problems.

The higher the molecular weight, the higher the boiling point so that is what can cause you to reach your limits on detection.

They say mineral oil is in here - what is its boiling pt? Eucalyptus B.P.?

Mineral oils are C15 to C40.
The chart is quite impressive. It said light mineral oil so it is closer to C15 than C40.
But good info in general. Alkanes.

The boiling point table on p 37 Harger - Separation Science gives us an idea of or to what to expect. I would guess above C15 will start to give us problems.
But that is why the separate worked.

Peaks are @ 6.679 8.67

We are going to increase the Carrier flow to 5 vs 4!

It appears that a normal range of flow rate is between from 4-6 ml/min so 5 should be OK. It reduces retention rate but also resolution.

We got a very clean separation here. There is no impurity and low in quality and it increases your chance of collecting the separation.

A very clean baseline.

Solvent	IS .42	vs .54	-22%
1st peak	~~6.11~~ 5.10	vs 6.64	-23%
2nd	6.62	8.64	-23%

So this is all good.
Now to the free oil.

Now I see something different. I think what is happening is that we are separating Naphtha!

The small peak that is lost may have been the Camphor. We obviously need a control on Naphtha now.

.42
5.05
6.91 This varies enough to be questionable.

We will soon see what naptha is.

The baseline is running as clean as can be.

Sure enough, it is naptha that is producing the peaks. Naptha is clearly made of more than one compound. The levels allow it to be a very good standard of reference for us.

Naptha alone

0.42
3.44 (small)
5.02
6.97

Aug 30 2015

For the first time, we now have a realistic portrait of denatured alcohol. Conditions are 250°C isothermal for 30 min. We have peaks @:

0.42 m	0.851 mV
2.13 m	0.434 mV
6.70 m	6.16 mV

(very broad & tailing but it does close @ 20 m)

The still could be very useful for each peak imposed on the main tailing peak.

There is something else (broad & shallow) peak that occurs @ 2.13 m. Smoky is required but it does repeat nicely.

Now for denatured tea:

We have our first run of orange tea on methanol. We may have additional small peaks @

0.22 m	0.03 mV
0.62 m	0.11 mV

You all now how to divide to work up. Co-apply the small peak up front @ a lower temperature & higher current. Use D. Alcohol as control.

When you are all done, clean out the column @ 250°C.
Always clean when the key peak comes in.
Set run @
150°C for 10 min. High current!

If you do not reach the plunge 6.70 mV peak
after 10 min, then just continue the run
dry for another 10 min.

If this does not work, you bump up temp to 200°C
to find it. (10 min cycles again)

So you added a dry reset; this is smart enough.

We now have a D. Alcohol reference @ 100° for 15 min.
It looks very clean w/ a single peak @ 0.44 hr 1.504 mV
This is @ high current.

Now introduce Tea in D. Alcohol

What we need to do therefore, is zero in on a
specific low temp region w/ increased sensitivity
Compare it to a reference, and then bake out
the column when you are all done.

There is the peak seen earlier but it is too broad.
So now we will bump it to 200°C.

When you bumped it up to 200°C you got an
immediately sharp defined peak, so you are
on the right track.
Work towards 200°C 15 m High Current for the tea!

You have even a 2nd peak (barely) showing up now.

And now you have the third peak which appears to be the alcohol ~~a water~~ peak.

Therefore the idea is working perfectly. You zero in on a range.

* Basically instead of programming the sequence you can run the temp stage in discrete steps finding the activity of each interval as you go along. The ~~la~~ ~~st~~ effect of increasing the resolution of each step

100°C for 15 min failed
200°C ~~s~~ Stacked for 15 min succeeded.

This means you will also need a control but I believe you have identified two discrete compounds in the tea-ethanol mixture.

The appropriate sequence for both control & the tea is now 200°C for 20m @ high current

Reference D. Alcohol 200°C 15m High Current.
Perfect results

Peaks @	Ø.42 M	1.90 mV
	9.09 M	24.81 mV

You have run into some graphical problems.
I saw no deviation from the reference plot for D. alcohol
The large peak may be flooding it.
The software does not allow arbitrary vertical scaling
& this does cause some problems.

Ok, we do have something, but it is not what
I expected. But in retrospect, it does make
sense. It is very small, &

There is indeed something present in a
very small quantity and I was lucky
to detect it immediately following
the solvent peak.
As the temp is changing on the small
peak and I have no idea why.

It did not work. Do not ask me why yet
I tried to zero in on a small secondary peak
after the solvent w/ 100°C for 5 min. Very current
but I never did see it.

Is it possible that I needed to keep it @ 200°C
to see that small peak and that the lower
temperature lost it? I would have to try again
to see. I saw that peak twice @ 200°C
and lost @ 100°C.

Alcohols are harder by nature.

The small peak:

The condition say that this is a moderately polar ~~alcohol~~ component with a fairly ~~low~~ high !!! boiling point.

The component would be in the tea.
But notice also that coming out first on the column does not necessarily mean a lower boiling point, if the above bear out to be true. I would like to check that.

How about 200°C @ 5 min w/ high current?

Sure Enough! This worked!
It required the high temperature!
So your presumption says correct.
A moderately polar substance with a high boiling point. I wonder what this would be?

Something like ethylene glycol is in this category.

You can also see that it is a very small amount.
It appear to be about 1/5 of 0.9%
based on peak proportionate size to MSDS info. ≅ .18% or .02% of the solution or ≤ 1800 ppm

The "solvent" peak in D. alcohol appears that it should be Methyl Isobutyl Ketone — This part is somewhat unexpected.

Yes miscible in oil! soluble in water!

117-118°C What is the polarity of this? Should be polar
Boiling point? Should have higher boiling point
Molecular structure? Should be hydrocarbon

Now there is a case where lowering the flow rate & decreasing the temp slightly might help.

Keep 180°C 5 min w/ flow rate of 4

It actually came out broader and weaker so I say not better.

Next try
220°C 5 min w/ flow rate of 4.

Lower temperatures is NOT always more sensitive if it does not volatilize!

Very nice. ~~I have done it.~~ Nicely separated peak.
Actually not true. It is even smaller.
200° @ Flow of 5 w/ high current was the best.

Increase the temp a bit before accumulated materials out the column.

Aug 31 2015

It does look like we have identified one true component but it has been a struggle. We actually do have a D. alcohol reference chromo and that is useful, even though it is problematic also because of an alcohol.

Let's proceed to solvents. And in the background we will work on headspace ideas.

I suggest we start w/ acetone.

Acetone is showing the following:

Solvent.		0.42 m	1.99 mV
		1.33 m	.15 mV
		8.34 m	.30 mV
X= 11.23	10.93 &	11.54 m	70.5 Ø tailing peak

The headspace idea did work! There are, however, many interesting discoveries.

1. The acetone dissolves some of the balloon (could even just be the yellow dye)

2. You set nearly enough air volume to save; it will not blow up the balloon.

3. The chroma is incredibly pure w/ only the solvent peak. This tells us that the acetone has some water, alcohol, or other contaminants in it, since the chroma for liquid & gas are so different.

4. We may not be able to capture anything with a boiling point greater than acetone, which is almost everything. It is primarily going to evaporate the acetone & anything less, which is not very much.

5. The bigger the question, how would alcohol behave here?
   1. 100% Alcohol?
   2. 90% Isoprop?

   Use Xylene - it has highest boiling point.

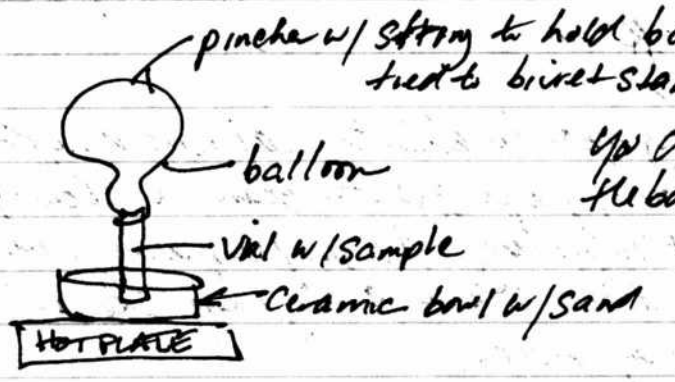
Then are other discoveries.
Headspace acetone ended up giving peaks @:

0.43m	996 mV
2.88m	.02 mV
10.30m	0.52 mV
19.39m	9.23 mV fading

do it longer it just the same general peaks are still there.

We want to think about headspace methods.

~~We~~ I am developing a method.



You do not need the balloon. Just cap it w/ rubber from balloon & clamp it w/ a rubber band.

You need to watch boiling points & temperature carefully as body will cause a lot of a disaster & is a serious fire hazard. Fortunately you are not dealing w/ a large amount of liquid but it still could cause a problem.

Headspace lessons today.

You learned some very important headspace methods, lessons & solvent applications today that are incredibly valuable.

Gas samples are by nature VOLATILE!
If you can get your sample into a gaseous form it will not harm the instrument since you know already that it is volatile.

Your peaks are incredibly strong as well and very sensitive. Remember that is how you picked up methane.

—

We have learned a lot here. In the headspace method the solvents do not even need to be miscible!! This is really powerful.

We had a XYLENE headspace reference. 250° 30M Low Current
Peaks are.

Heated to 250°C	0.41 m	1412 mV
	7.28	0.213 mV
	9.36	141.0 mV

—

Now we are trying Camphor phenique 8 drops added to 1 ml Xylene 250°C 30m.

You have also learned to window the software better.

You have your first bonafide chromo of separation today & printed on paper. Headspace method was used & it has numerous advantages to it. We know the sample is volatile w/ the headspace method.
Two specific peaks were identified.
You could try this again in D. Alcohol. Reference & Change.

Lab 50 for alcohol reference.
Is it possible your injected sample is too large? This causes tailing peaks

D Alcohol also came out very well
1/4 ml Headspace 250°C 30m Low Current
Peaks @:

0.41 m	524.8 mV
3.09 m	0.53 mV
11.07 m	35.86 mV
26.67 m	1.94 mV

Quite different from xylene

Our mix Low peaks @

0.40	388
3.08	.16
7.18	.08
8.79	.20
10.77	44.79
17.41	.10
22.19	5.48

So obviously we have a lot of activity here.

0.40	Xylene n alcohol
3.08	D. alcohol
7.18	Xylene
8.79	Possible Xylene
10.71	D alcohol
17.41	None - may be new
22.19	May be alcohol

Ultimately it seems as though we are picking up both solvents D alcohol & Xylene w/ a possibility of a new substance but not real likely.

Now lets look @ Xylene + Tea Tree Oil.
(Results look very similar to Camplo Phenique

	0.43 m	406	Xylene
	1.01 m	.425	
Tea Tree Oil	7.35 m	,10	Xylene
	11.88 m	13.17	
	23.08 m	5.20	

Camplo Phenique in Xylene was

	0.43	1152	Xylene
Campho Phenique	7.33	,40	Xylene
	9.58	129	Xylene
small spike	19.94	0.15	
	21.74	6.5	

So actualy they are quite different from one another.
Repeat these w/ smaller injections

There is a possibility that your needle was contaminated w/ the xylene reference. Something does not appear right on the chrom w/ methanol or 9 xylene is completely flat past initial peak @ 0.4. How can this be?

Xylene references are still needed w/ clean dry needle.

You also had a hole in the balloon. Maybe that made a difference?

Variables

1. Clean syringe
2. No holes in balloon.
3. Tight cap vs balloon?
4. Temperature/heat?

First one will be w/ clean syringe, clean solvent, xylene, and a tight cap. Pressure literally very quickly over @ 130°C. Cap is bulging already.

It popped too easily. We need the balloon. Reduce sample size input to 1/4 mL Heating to 200°C.

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Another Exciting Day!

[redacted]

2. Xylene reference for some questions
   1. temp change in cooling?
   2. Hole in balloon
   3. Rubber cap results
   4. Clean & Dry syringe?

3. Study Chromatography Course?
4. Study Chromatography Books?

5. Headed toward lipid analysis

Here is the report:
Xylene reference run. Sample heated to 260°C.
Baseline drift but only 2 peaks show

0.41	382 mV
27.51	1.417 mV

So the sample looks very clean.

A hole in the balloon should not matter for a repeat. You have almost a flat run on xylene now. This is actually quite beneficial. We also need to increase to 40 m instead of 30

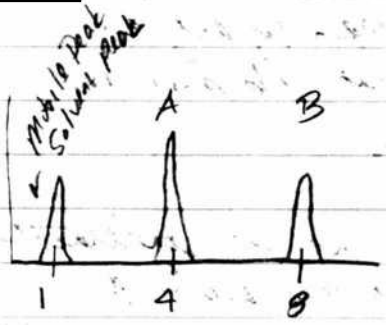
I think that you are proving the already that you had water condensation in the needle. This could have come from our exposure. Keep the needle clean.

It does seem that something boiled off the xylene @ the lower temperature. Maybe there is a water or alcohol component?

What if you took tea by itself, ground up the less?

Repeating test @ 250°C 40 m.
(Xylene) Sample heated to 200°C+

[redacted] Practice Problem



What is the fraction of time that A & B spend in the stationary phase.

$t_r = t_s + t_m$

t_s = stationary phase time; t_m = mobile phase time

1 min in mobile

A spent 3 min in stationary phase so answer is $3/4 = 75\%$

B spent 1 in mobile & 7 min in stationary $= 7/8 = 88\%$

(solvent Time)

So fraction of time in stationary phase = $\frac{\text{Retention time} - \text{Mobile Peak Time}}{\text{Retention Time}}$

$t_s = n\frac{\Delta t}{t_r}$ So for A: $\Delta t = 3$, $t_r = 4$, $n = 3/4 = 75\%$

This can be used.

for B $\Delta t = 7$, $t_r = 8$ so $7/8 = 88\%$

$n\,\Delta t = t_s \cdot t_r$ $\quad \Delta t = t_r - t_m$ so $t_r - t_m = t_s \cdot t_r$

$t_s = \frac{t_r - t_m}{t_r}$ OK

We are now using that Xylene is a very good solvent that is very pure w/ only a mobile peak & a small peak 26 min in. The ha many applications then.

We have a polar column so it attracts w/ polar substances such as water & alcohols & this is causing us some problems.

I want a non polar column so that pola materials will not interact as much with the column so they therefore come out fast.

Our repeat test is

(1)	0.40 m	429 mV
(2)	26.72 m	2.4 mV

We can also see that Peak 2 spent a lot of time in the column so it is more polar. The fraction of time that it spent in the stationary phase is

$$\frac{26.72 - 0.4}{26.72} = 98.5\%$$ which is too large.

I am now doing Fruity green orange tea heated to 250°C 40 m. Sample was also heated to 250°C.

Tea

We do have a peak already near 1.0 min. These indicate a polar substance of some kind.

You can slide down the balloon to get more than one run from a balloon.

Total screen on the heated tea!
I have 2 very nice peaks.

What is the first peak?

$$N = \frac{16 t_r^2}{w^2} \quad \text{or} \quad N = \frac{5.55 t_r^2}{w_{1/2}^2}$$

I have a fantastic separation on the tea here two a polar substance & a mid level polarity substance & water.

non polar ↑ ↓ polar

Peaks @	0.41 m	257 mV
	0.51 m	519 mV
	1.06 m	1.33 mV
	13.11 m	1.0 mV
	28.33	1.98 mV

So altogether we have 5 substances involved in the orange tea.

Peak area does not mean actual concentration. You will need to learn to calibrate.

Lets solve an example of theoretical plate

14.74 – 11.25

$w = 2b.24m - 2$

$t_r = 13.09$

$N = \frac{16(13.09)^2}{3.49} = 785$

apparently wrong

Is this good a poor?

Used an arbitrary peak from the tea leaves

Actually the formula appear to be

$$16\left(\frac{T_r}{W_b}\right)^2$$

$$= 16\left(\frac{13.09}{3.49}\right)^2 = 225$$ which is quite different

this means a very good theoretical column

Typical acceptance criterion is 2000. So we are 1/10 of that.

Typical theoretic plate is 100 to 1000 per meter. So I am in ~~the range~~.

OK, this is my answer

Tea

5. letter work on the first section only

3 peaks are tapering up to 5 min.
Lets use 150°C.

150° C 5 min High Current

We are doing marvelous work. The was a perfect set up for the polar non polar region.
We have peaks @

150°C		250°C
Ø.39 m	1180 mV	(.4)m
Ø.79 m	597 mV	(.51)m
~~1.49~~ 1.49 m	.03 mV	2 new peaks here
2.26 m	.09 mV	
3.56 m	.21 mV	1.06m

These two may be more quantifiable

So within the 1st 5 min, we have now identified 5 components!

This means that we have now identified 7 different components to the orange tea. Superb work.

Lets go again.
The 3.56 peak is now gone. So if you heat it long enough it can disappear.

Tea

How long you heat the sample affects the peak performance.

I did not heat it as long the time (about 10-15 min) @ full temperature (approx 230°C) but let to give a very good peaks up front.

The third peak is also much better. You also heat the balloon over to the side for a more sealed top.

Running 150°C 20 min High Current.

There are four peaks. - Very good work.

How would you go about collecting these?

Switch to Liquid Chromatography?
Since they are polar
you would think that a polar solvent
is pulling them out.

Run it dry @ 250 for 30 min @ Low Current to clean out.

0.41 m	829 mV
0.77 m	805 mV
3.48 m	0.90 mV
5.60 m	0.50 mV

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Then tells you that you have 2 non polar components in fairly significant & roughly equal quantity.

Notice you see 2 Colors w/ solvent
Denatured alcohol = red
Xylene, MEK etc = more yellowish

Those are very likely the two components.

You would therefore try to work with these two and non polar solvents w/ liquid Chromatography

You may also have them already w/ just two varying non polar solvents - the might be another way the problem has been solved except GC would have given you the information ahead of time rather than trial & error w/ solvents.

—

Get solvent samples [redacted]

Tomorrow we can go after the lipids?
Maybe tonight?

My pyrolysis is all black then there is nothing left to look at. You want to catch it while it is burning.

Very interesting. When you take the pyrolyzed product & mix it with water you get a yellow green solution. This mean that it is over a different component of some kind.

Two different solvents are giving you different materials. You see how GC might separate 3 right away with the naye eye

red - D. alcohol
yellow - MEK, xylene?
yellow polar - water.

Heat over to 250 and ~~the~~ & two more peaks came out. You are picking up to 2 polar peaks.

You might consider a program of

60 @ 150 250

10 min apiece? or 6 min apiece?

So what do you really want to know today?
How much have you already learned in a few hours!

1. NIR is fantastic! and you now have some of the capability.

2. Water, alcohols, CH groups, OH groups, NH Groups!
mixtures of all of the above!
Easy to establish controls!

[redacted]

4. The Environmental Filament does not absorb any NIR.
What is your interpretation of this?

* 5. What about headspace analysis of the Environmental Filament?

There is plenty for today -
Acetone & Water mix, and separate.
Try it!

Env. Filament Project
Headspace

We Environmental Filament has been volatilized via headspace into the GC tonight. Headspace temp was 200°C.

Program is 50°C 6 min
Ramp @ 20°/min to 150C
150°C 6 min
Ramp to 250°C
Hold 15 min @ 250

"Programmed"

We have important readings:

Non Polar — Ø.41m @ 2011mV

Slightly Polar Neutral — Another possible @ 4.41m @ .06mV

Polar Suspect Phenol — 35.58m @ 8.54 − 6.90 = 1.64mV

We have now run Env. Fil.
@ 250°C 30m Isothermal
Broad Peaks Develop after Initial Non-Polar Peak

Ø.41m @ 4467mV

8.83m @ 2.96mV

13.28m @ 3.91mV

Env Filament:
Nw @ 500° for 6 min

	Δt			
①	(.51-.34)=.23	Ø.41 m	1746.5 mV	40+200.0
②	(4.85-4.14)=.71	4.48m	.145 mV	.051

A

① % = $\frac{.051}{200.0+.051}$ * 100 = .025% = 253 ppm

② 99.975% = 999747 ppm
where ①+② = 1 MILLION PARTS.

We have a match.
2 Polar bonding substances

50°C

Ø.41m 250 ~~253~~ ppm.

Average of two runs

4.45m $\frac{999750 \text{ ppm}}{1 \text{ million parts}}$

With the 50° session.

[redacted]

Em Filament Project
Non Polar Compound Analysis.

Now lets run @ 150° 30 min

Δt			A
.77m − .32 = .45m	.40m	1920.0 mV	432.0
1.00 − .77 = .23m	Ø.85m	.85 mV	.098

(1) ~~(2)~~ $\frac{.098}{432 + .098} = .023\% = 227\,ppm \cong 225\,ppm$

(2) ~~(1)~~ $= 99.977\% \cong 999775\,ppm$

Avg of both runs = 227 + 253 ppm = 240 ppm of the ~~pst~~ non polar compounds.

I have done it. Polar compounds are a bit unsubstantiated but it appears we need a non polar column to work on that segment.

This is CO_2 & air. Not so it's CO_2

This is not necessary true. See Sep 4 Notes.

Env. Fl. Project:

Findys ae

1. IR spectra
2. NIR spectra - NIL
3. Catalase reaction ⇒ IR group ang.
4. Chromatography — Polar & Non Polar results.
5. Uniform across globe
6. Tentative structural formula

Sep 3 2015 Page 35

[redacted]

1. The culture progress is important.
   1. Glucose
   ~~2. Bromphenol~~
   2. Microscope
   3. Increase Concentration.

2. Combustion - does it not always produce water?
   NIR test?
   Test w/ Milk for example
   Tea should have produced this also
   Test for steam?

3. Polar groups by nature
   alcohols, amines, carboxylic acids

4. How could you test an amine in water?

There seems to be a lot of materials coming out of the column, especially after you baked the filter. - It is a Carbotrap filter.

Does this mean impurities in the gas? Possibly. It might be that truly contaminants in the helium are collecting in the filter and that it needs to be cleaned out very well also.

150 should be enough to handle all water. Even less should be possible.

Column is now clean again. That took a while. Knocky down temps 50°.

Settings: Oven Max 210° C
Detector SetPoint 220°C.

So do not take runs above 200 ~~except for baseline~~ even for baseline!

So, starting w/ water vapor. Why is there always a peak @ 0.4, even with water vapor?

Is it air? Notice you also have the secondary peak that came out to be 250 PPM. Sounds like air & CO_2???

So you may always have air, CO_2 & water!!!

We have learned, somewhat reluctantly, that your main peak @ the front is an air peak!

Or just maybe, it is a solvent peak - still sorta it out, but in all case it doesn't seem to be due w/ a gaseous sample.

To go further than that, you will have to work on it.

We have succeeded in pulling something from milk. What do you think it is? How would you find out?

One presence it is fairly polar so you could try a solvent.

New variation on headspace. Preheat w/ hotplate. Beg to higher temp w/ torch on vial.

First trial w/ Tea @ ~~60-120~~ 80°-180°C Ramp

Order
Peak Air = 1905.2 44.4%
Peak 2 = 2381.3 55.6%
Σ = 4286.5

No sheet a more of it than air in the tube. Good.

Sep 04 2015. Latent Tea Analysis

We are doing some interesting work now.
We have complemented the heating with the hot plate using a towel or surrounding card to elevate the temperature. It is working very well.

We are back to orange tea & we are picking up numerous peaks (4 so far).

We must start learning about response factors. Apparently TCD is complex.

Lipids in headspace?

W/ headspace it does seem like you want to drive off the moisture first.

Δt				A
(80 − 36) =	48.4% Peak#2	0.43 m	270.0 mV	59.4
(2.50 − 1.50) =	51.4%	1.76 m	126.3 mV	63.2
(10.61 − 9.89) =	530 ppm	10.25 m	.176+02 mV	.068
(12.64 − 11.94) =	820 ppm	12.26 m	.289 mV	.101
(14.39 − 13.81)	230 ppm	14.04 m	.107 mV	.028
(18.81 − 18.12) =	800 ppm	18.53 m	.074 mV	.025
				122.82

But Peak #1 is air so it can be disregarded.

Peak		
2	63.2	99.655%
3	.065	1025 ppm
4	.101	1600 ppm
5	.028	440 ppm
6	.025	400 ppm
	Σ = 63.419	

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9 Components.

You are getting better @ this

Now lets repeat..

	Δt		m	mV	A
Air		air			↑
		match	4.36		80°C
CO_2		CO_2	5.14		↓
		match	10.23		Ramp
		match	12.29		↑
		match	13.97		180°C
		new	15.22		
		match	18:11		↓
		new	>21.0		

Therefore, as before, we learn that orange tea is rather complex & it would take a lot of work to separate.

We see therefore that the best time to change to 15

80°C 8& min lets try this.
180° C 20 min

Once you figure out the layout, then you can switch to huge column for better measurements.

Superch work @ 80°C.

	Peak 1	Peak 2
80°C	.436	1.803
180°C	.430	.693

$z = f(x, y)$

This is an interesting problem.

Now we have our runs @ 100°C
We have peaks @

		m	mv
Air		0.430	1819.5
		0.693	716.0
=10.25ml		1.15	12.0
=12.26ml		1.48	16.2
Water Pressured	=721 ml	2.44	88.9

CO_2 not presumed to be detectable. Many changes to notice here. Water appears to have compressed in time close to 10 to 1.
Also notice sensitivity of med peaks (530 ppm & 820 ppm) is increased radically so.
High temps are a completely different ballgame.

In general, notice that you have most of the information @ the higher ~~temperature~~ and the highest current setting. But you did lose the CO_2. Notice you did not have it on one of the 80°C runs also.

Both runs do seem valuable in their respective ways and the 80-100 program probably is a good overall one to use.

Notice that you also picked up a deviation in the air peak. Maybe that is argon?

The pyrolyzer seems to work very well therefore.
Higher ~~temperatures~~ do burn off some product ~~therefore~~.

Now we know enough to go for lipids.

CDB Lipids

Looks like a lot of water vapor might be here.
Only one peak. Low Current.

0.403 m	647.4 mV
2.06	8.35
14.73	3.5 exceptionally broad peak

Repeat @ 80° w/ High Current for early segment
The balloon broke so only mediocre results.
Nevertheless lets do xylene next.

0.403
2.09 results repeated.

Now for xylene: Balloon immediately inflated

0.41
2.09 So it is the same.

CDB Raw:

Next: Low Current

Work on CDB:

	m	mV
	0.42	396.0
80°C Target →	1.85	93.2

There are back from 80°C Low Current.

Apparently a broad water peak after.

Lets shift to an alum. foil top.
Lets go 60°C for 10 min @ high current

	m	mV
	0.43	1559.0
60°C Target →	2.91	96.6

Now lets go to 180°C High Current.

At 180°C				
		0.41	1922.5	A 259.5
	(.60 - .33) = .27	0.70	407.1	
Air Unknown Suspect CO_2 Very broad	(1.61 - 1.30) = .31	1.49	0.213 150PPM	.039
		10.51	3.01] Polar	
		20 m+	4.56+] Suspected	

Yes, peak @ 1.49 is CO_2. So we have one very volatile, likely polar component & 2 likely more polar compounds, one of which is water?

~~Lets go for 30 min.~~ More important to see proteins.

Proteins: Survey Fused. 80-180°C High Current

80°C

80°C:

	Δt	m	mV	A
Air	.67-.34	0.41	2295.3	378.2
Unknown	2.35-1.78	2.02	100.2	28.56

28.56/(378.2+28.56) = 7.02%

180°C

Open Peak

~>21.0 7.8mV

Run @ 60°C for 10 min

60°C

Air		0.42	2079.5
Target →		3.16	25.67

Target of Proteins cannot be CO_2. CO_2 @ 80° is 5+ min so 2-3 min is impossible for CO_2.

$\Delta t \cong 30°C$ is a doubling. Rule of Thumb in Modern Chromatography.

$\frac{\Delta t}{30} = 2^n \cdot t$ eg $\frac{\Delta t = 60°C}{30} = 2 \quad 2^n \cdot t$

so $\frac{\Delta t}{30} = n$; $t^* = t_0 * 2^n$ Seems to work quite well.

$\frac{-20}{30} = -.667 = m$ $2^{-.667} = .63$ $.63(3.16) = 1.99$ vs 2.02

$\frac{20}{30} = .667$ $2^{.667} = 1.59$ $1.59(2.02) = 3.20$ vs 3.16

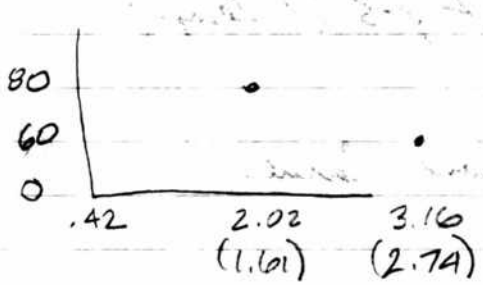
Env. Filament Survey 80-180°C High Current

80°C	m	mV	
	Ø.40	2099.Ø	
	2.04	11.30	= 5354 PPM

150°C	
	Ø.40
	Ø.86

The data came from a previous run on Sep 2. You claimed that that .86 corresponds to CO_2 but this cannot be the case if we have 5354 PPM & if these two sets of peaks are one and the same.

∞ Broken Curve



$y = a + b\ln(x)$

$Temp = 97.9 - 37.6 \ln(t_r')$

or we can just plug to adjusted ~~retention~~ time.

80°C 2.02 − 0.41 = 1.61
60°C 3.16 − 0.42 = 2.74

$y = a + b\ln(x)$

$t_r' = 18.82 - 3.93 \ln(temp)$

t_r' = adj retention time or

So if temp = 150°C Something is not working here.
$t_r' = -.87$ so not feasible

The may not be too bad. Add 0.41 C and to get actual t_r. If it is less than 2 les (t_r') or 0.41 (t_r') then it probably will not a very smart temperature.

100° C $t_r' = 0.72$ m or $t_r = 0.72 + .415 = 1.14$ m
looks reasonable.

45° C $t_r' = 3.86$ or $t_r = 4.28$ min
looks reasonable.

~~135~~ When does $t_r' = 0$? $18.82 = 3.93 \ln(temp)$
$\ln(temp) = 4.79$

temp = 120.1° So you should never use a temp ≥ 120°.

Go have to do a min of 2 pts (2 temps) to get this curve.

Lets look @ Env. Filament Curve to compare

(4) 80°C .40m Temp = 107.2 − 55.06 ln(tr')
(X) 2.04m

150°C .40 tr' = 9.86 − 1.88 ln (Temp)
.86m

so the get a list essentially so that we can now form ratio of tr'

but solve for tr' = Ø for env. filament first

$\left(t_{r_\emptyset}'\right)' = \emptyset_C$ $\ln\left(\frac{\text{temp}}{\text{tr}}\right) = \frac{9.86}{1.88} \Rightarrow \text{tr} = 189.6°$ Temp

so the env. filament should not exceed 189.6°
the plotter should not exceed 120°.

We can form a ratio:

$\frac{tr'_{\text{env. filament}}}{tr'_{\text{proton}}} = \frac{9.86 - 1.88 \ln(\text{Temp})}{18.82 - 3.93 \ln(\text{Temp})}$

What does this look like?

The is a very interesting equation.
The ratio $C = 1$. $C \sim 80°$.
but no where else. What does that mean?
Is this just a coincidence that we get the same t_r @ 80°C

What if we were to have measured the ene. ful. @ 60°C instead of 150°C ?
Would we have a flabbb ratio curve then?

So if you have any time @ any temperature you can determine what it will be at any other temperature.

$$\frac{\Delta t°}{30} = n \qquad 2^{\wedge}(t_r) = t_r^{*}$$

eg $\frac{150-80}{30} = 2.33$ $\qquad 2^{2.33} = 5.04$

this did not matter.

Sep 06 2015 - Sunday.

The last ending day in the lab. Time to start your planning on what to take ahead.

1. We have worked very hard w/ GC the season.
   1. We have shifted toward pyrolysis & only
   Gas samples.
   You have left w some confusion on CO_2. You have gradually learned about what the air peak is.

2. IR of xylene & heptane for comparison could be helpful.

3. You have made a major discovery of NIR.[1] on the Thermo Scientific machine. Incredibly simple to use. MM calibration could be done.

4. Major discoveries on Saccom IR this week, headspace & pyrolysis in general. The opening many avenues for investigation.

5. Yr have the GC prep idea coming up - there's a Jarcenter prospect also.

6. Don't forget to put away your crystals![1]

Page 50

Lets start today w/ the Xylem - Lipid question, that is rather critical.

Good Work. We love it.

Raw for Env. Filament & Catalysis Product.

Sep 01 2015

1. Last couple of hours in the lab.

2. We have made good progress w/ improved spectra. The background was not properly taken care of.

3. Phenol Comparison? Menthol standard? Ethanol standard?

4. I now have 4 solvents - pure:
   1. Benzene
   2. Methanol
   3. Aniline
   4. Isopropanol
   5. ~~Ethylene~~ Propylene glycol ~~194°C~~ 188°C perfect

   These will be very helpful references. 184°C

We are working w/ phenol & GC. Some very interesting work. We have 5 peaks altogether that are showing up. One @ 80°C and 4 @ ~ 25 min

You should try a 180°C dwell @ 60 min. The peaks @ 200 are not separating cleanly. You might even need to go to 150°C.

Herman Lair is very simple to pyrolyze and gives very interesting results. Lots of activity

Herman Lair must be raised to 200°C. to pick up the second level of volatiles.

The 2 minute peak is CO_2.

Sep 11 2015
Outside of Rainier, Washington.

Crippen's book on identification of organic compounds is a gold mine.

Because we had two peaks on the column that remained in good form but eluted late (in the study of the phenol solution) We now understand that the substances are

1. more polar than non polar.
2. they have a higher boiling point.

These are telling you a great deal. Ca~~n~~ polar substance with a high boiling point were make alone w/ phenol - dilute it!

3. You could also tell when you heated it that it had a lower boiling component, ~~an~~ namely it was water) and you could have determined this w/ micro distillation.

4. You have seen how water interacts w/ the column and that generally it is undesirable.

Page 54

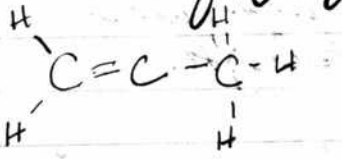
We can now formulate structures on the ligand.
But the most useful right now is the Env. Filament.
Start w/ the average. You need a file with
the background properly removed. You did this. Sep 06 file.

3251, 3279. Phenol expected?
Not necessarily. We have nothing @ 700-800.
What does seem to be a glove is alcohol & amine.

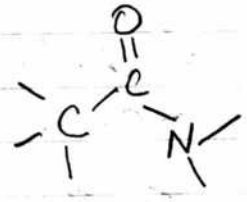
OH
NH

Nothing here shows an aromatic @ this point.

What we do see next is alkane & alkene.
The lack of pyrolyzes indicate a high molecular weight.

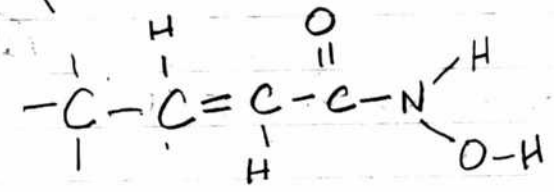


w/ OH & NH.



1642 has

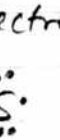
but we also have alkene.
This leads to



but O has 2 bonds
so you do not have
OH yet

1516 brought in a Nitro group
I do not see a basis for the aromatic nitro yet.
1224 brought in the amine & the nitro which we have.
Nothing else is required except what we have above.

—S—
Sulfur has
6 valence
electrons



We have a fairly strong indication of S=O
The strength of the material indicates disulfide bonds.
How many bonds does sulfur make?

Sun Sept 13 2015 Olympia WA

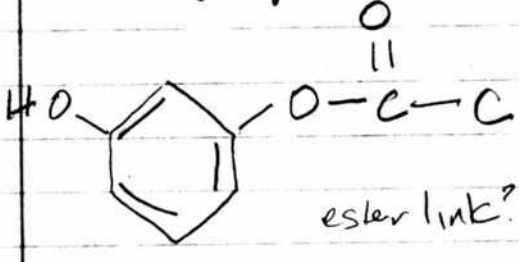
CDB Lipids IR Analysis:

We positively have a phenol
McMurry Indian shows

3400 Alcohol peak, secondary peak
3050 Are alkenes
Weaker than alkane @ 2920.
~1600] alkene
~1500 & 1460]
690 & 760 Mono substituted aromatics

We likely have a perfect meter along with an alkane chain of undiscriminate length.

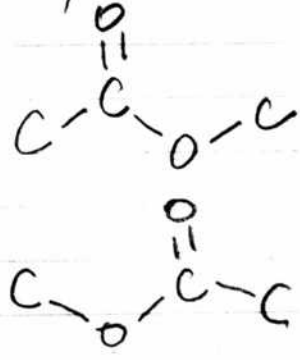
We are therefore looking for a ketone or ether group.



Ester?

ester link?

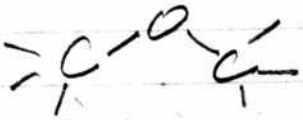
Now, an ester is:



or

Now, the ester does not show a carbonyl @ 1740. There is clearly pulsimetric alkyl (CH) peaks below 3000. ok here. We should also see a C-O band @ 1200 and we do not. This indicates no ester linkage in the COB Lipids. There is impurities

Now, an ether linkage is:



Ether has

Ethers Ar-O-R is 1220 to 1260
Ethers R-O-R is 1070 to 1150
There is a problem here.

SDBS Match: #4570 Vitamin U Chloride
#2786 Dimethyl pyridine oxide

CBS 18711 ~~Aromatic~~ Aminobenzyl Alcohol
#15 3009 D-Tryptophan
phenyl
29190
11072
28071
7703

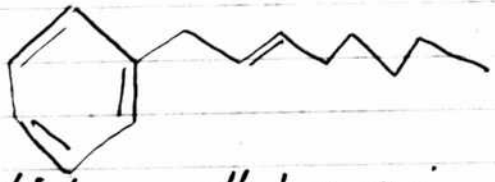
+-10
86
phenyl 29374

10 3412, 3022, 2961, ~~2922~~ 2924
76 1614 1495 762 692
phenyl

Sep 14 2015 Olympia WA

Continuing the assessment of the lipids.

There does not have to be an ester or an ether linkage necessary. Look @ the phenolipids.



B16b1, an alkylresorcinol (a phenolic lipid)
It is not either a ester or an ether linkage!

The r-group crucial.

Phenolic lipids are derived from mono & dihydroxyphenols

What I would like to do next is check known phenolic lipids for ester & ether linkage
No check this! It simply shows
OH
COOH
double OH
double OH & Me (Metal?) (cardiol)

Ester

Ether

1850 - 1830	NO	1285 - 1110	NO
1780 - 1750	NO		
1765 - 1720	NO		
1290 - 1180	NO		

Disulfide bonds are R-S-S-R
Sulfur forms 2 to 6 bonds

The primary additional peaks are @

2084 R-N=C=S

1376 Alkane RCH_2CH_3
N-O Aliphatic (No 1540 secondary)
S=O sulfate ester

1033 S=O This is best fit

Sep 15 2015 Olympic N.P.

You want to identify the peaks.

Let's work on this 2084. Also let's sort out the hydrocarbon

Koji will get one in the neighborhood.

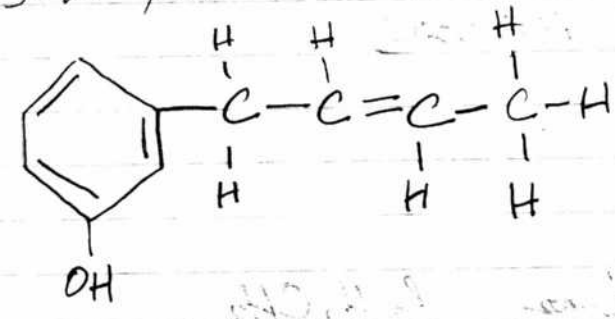
2961 is Alkane CH_3 2960

2924 is Alkane CH_2 2925

Arenes also, however, w/ Aryam.

3022 is Alkene $=CH-$ 3020

So we know that we have



as a minimum.

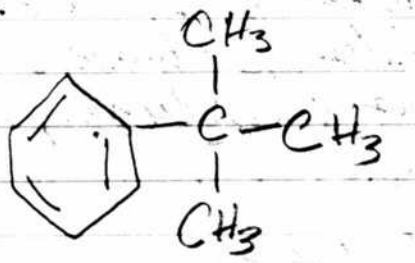
Now as far as Koji is concerned, 2084 is quite clear.

Table 7 $C=N+-H$ These seem pretty clear. w/ a range of 1800-2200

But there is some competition

Table 4

Auram gives some details on p206 on monocyclic benzene.

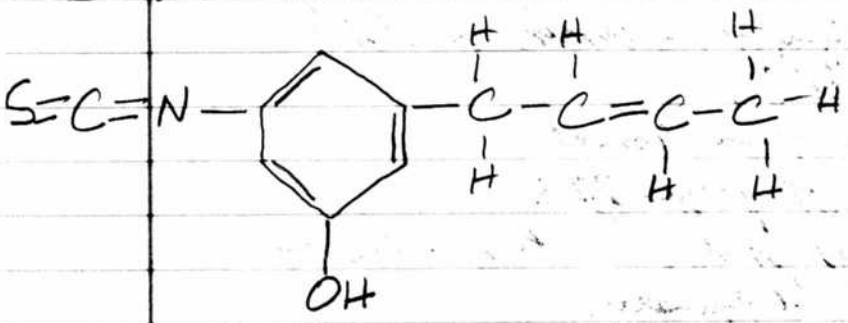
Indicates  @ 762 & 692

Keep it basic. Back to 2004
Koji is Table 4 and Table 7

Our strongest candidate w/ koji is is an isothiocyanate aromatic

$-N=C=S$ which is ~~negative to~~ identical to IR poly $R-N=C=S$

So this leads to:

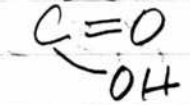


Remember the color.

Koji supplements @ 1250 & 930 cm. ~~not confirmed.~~

Notice koji's comment that it is a broad split or w/ shoulder. The molder perfectly. Topic is Isothiocyanate

930 is a possibility.

Auram matches this also. This is 3 sources.
In Auram, this is under the section of Carbonic acid derivatives. Carbonic acid is 

Aram p 468 is making very interesting comments about urea, & the spectra of amides, isocyanic acid and derivatives of Carbonic, not Cat carboxylic acid.

Now lets look to 3412.
Also Koji Tables

Now, with 2084 we also have Koji, the possibility of an "unsaturated amine".

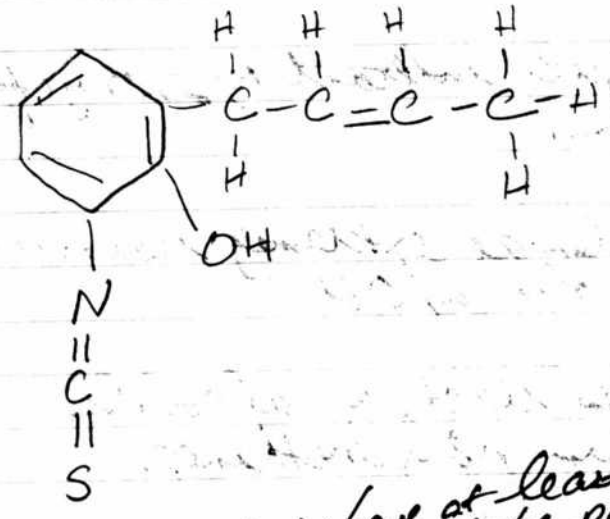
$$C=\overset{+}{N}(-H)- \qquad 1800-2200$$

Called the "Immonium Band".
This is not Category of an amine salt"
Unsaturated.
It is a derivative of an amine.
Aram letter of page 223.
It does just not seem to match. as well as the isothiocyanate.

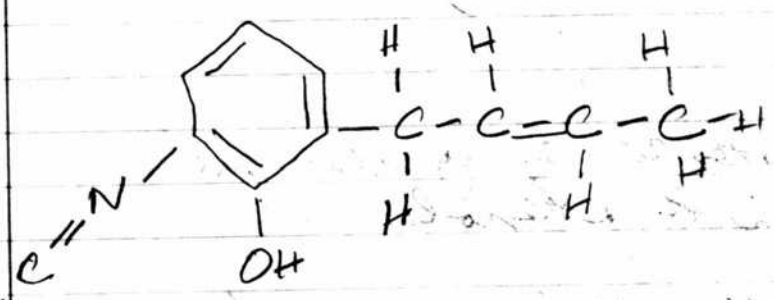
Phenols
Dimers at @ 3500
Polymer @ @ ~~3440~~ 3320
We are @ 3412.
This indicates a low wt polymer phenol.

Now if we look @ Koji, we seem to be @ a 1,2,3 substitution pattern.

This therefore indicates



or should have at least 2 to be poly.



trans a
Cis affects
melting point
Check!
Also check
Col—

trans alkenes are less polar, more symmetrical have lower boiling points and higher melting points.

CIS	TRANS
1. Less Symmetrical	1. More Symmetrical
2. More Polar	2. Less Polar
3. Lower melting point (freezing)	3. Higher melting point (freezing)
4. Higher boiling point.	4. Lower Boiling Point

Olive Oil is a highly poly unsaturated fat
Linseed oil
Hemp oil

Aromatic Isothiocyanates can be used as blood substitutes

Phenolic lipids extracted w/ dichloroethane methanol.

Isocyanates are extremely reactive & also w/ OH

Isocyanates react w/ alcohols to form polyurethanes.

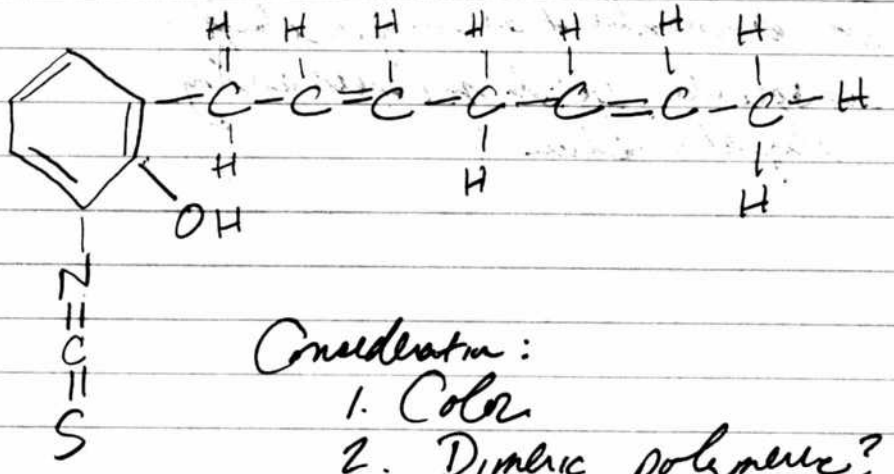
and w/ amides to form ureas.

R-

Isothiocyanates are very very reactive with phenols

It says the reaction is very exothermic & even dangerous

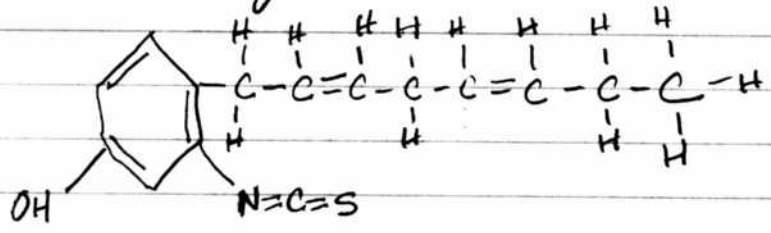
Lipid Assessment.



Consideration:
1. Color
2. Dimeric, polymeric?
3. NCS in aliphatic portion?
4. OH repeats?
5. SDBS interpretation.

Let look @ 1033 & 1376.
1033 can also fit alcohols and phenols (1000-1200)
We do have 1033 and 1103

A substitution pattern from Aroma p212 that fits quite well (1035 vs 1033, 1101 vs 1103, 1200 vs 1200) is that of 1,2; 1,4 or 1,2,4. We pick the latter 1,2,4 right now to fit everything in.

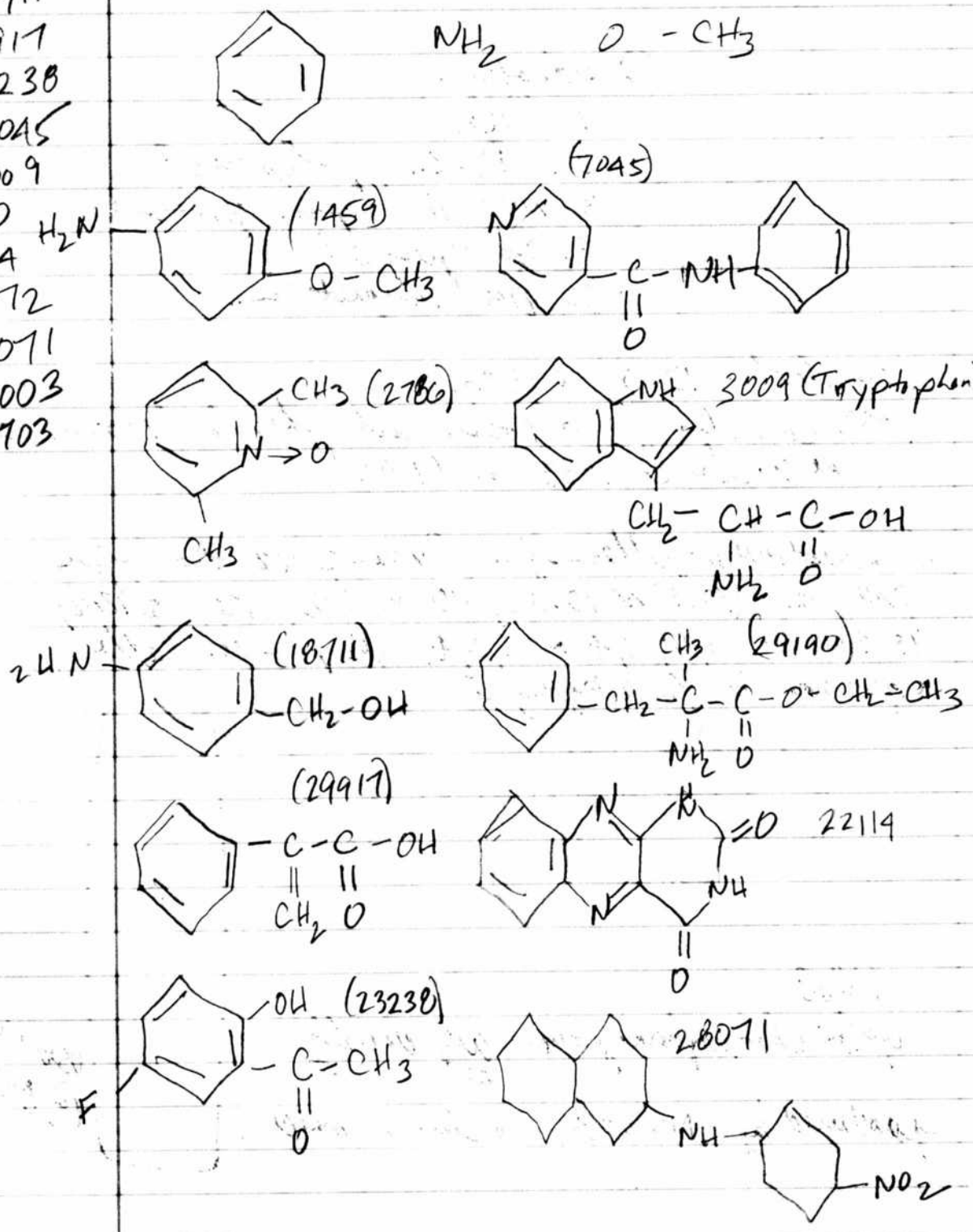


3412	1033
3022	1103
2961	1376
2924	
2084	85-15
1614	
1495	
1451	used these @ 85-15
762	
692	

Color?
Yellow chromophore groups are usually produced by N=C=S. We have a match.

But remember that benzene itself is insoluble. You do not need a long aliphatic chain.

1459
2786
18711
29917
23238
7045
3009
29190
22114
11072
28071
18003
7703



Lipid Proposal Sep 15 2015
Olympic National Park

What we find, therefore, are the following groups.

Aromatic Ring	10/10	100%
CH_3	4/10	40%
$-O-$	2/10	20%
CH_2	4/10	40%
OH	3/10	30%
NH	4/10	40%
NH_2	2/10	20%
N	3/10	30%
C=O	6/10	60%
C=C	10/10	100%
Aliphatic C=C	1/10	10%
N within Benzene Ring	4/10	40%

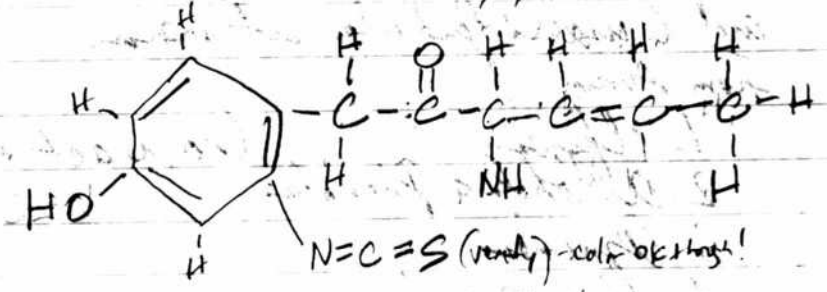
Ranked:

%
100%
100%
60%
40%
40%
40%
40%
30%
30%
20%
10%

Aromatic
C=C
C=O
CH_3
CH_2
NH
OH
N
C-O, NH_2
Aliphatic C=C

N=C=S

This leads to, along w C-N=C=S and Colin's 1,2,4 Substitution



~~The only real change here is a shortening of the aliphatic chain~~ and the addition of the carbonyl groups and the amine groups.

Verify when the C=O comes from. Maybe SDBS is sufficient. Used 65-15 from 2 pages ago.

MW: 13·(12) + 2(16) + 2(14) + 13 = 229

Could you be off this much? Maybe.

A Lipid Model is in play.
You will check 1. Cola
2. albus
3. isothiocyanate connection
4. repeat duality of isothiocyanate.

Let go on to the env. Islands.
Low point @ 3257
koji: polymerase OH TS(3)
It is the only candidate.
On JUN 08 run, we low the same result.
here it is 3279.

$\bar{X} = (3257 + 3279)/2 = 3265$
koji is clear on this w/out competition.

Now let's look for alternate source to verify and to understand what it means.
From Avram
1. alkyne (no reactive it does not appear like)
2. alcohol & Phenols
3. Amines
4. Quinonoxime
5. Amides.

So Avram has many more choices than koji.
Avram states polyhydroxylic phenol @
3320.
Koji. got 3200 - 3400

What is a polymeric phenol? (polyphenol)
What is a polymeric alcohol? also known as polyhydroxy phenols

There are polymeric alcohols & there are polymeric phenols
Which is it? ↑
water soluble.

There are at least some soluble polymeric phenols

Polyphenols possess a significant binding affinity for proteins.

dyes. They are tough & rigid
plastics
resins

Polymeric alcohols seem to be defined by water solubility.
Polymeric phenols? they are used for high strength high temperature applications. Evelyn says a polymeric phenol, bakelite.

We have 2 independent graph sets. We will use those data points that overlay.

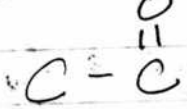
3265 is our first point.

We do have alkane + alkene that show up but we will come back to these.

$(1642 + 1645)/2 = 1644$ very strong peak.

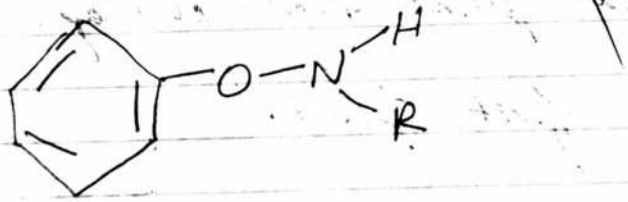
IRSpec 1645 is alkene.
1650 is amide C=O

since the alkene is medium, and the peak here is so strong, we must assume that more is going on here.
An amide is:



Sidenote: Check the aromatic isothiocyanate structure for the location of the nitrogen.
Yes, it can happen NCS external to the benzene ring. Yes it can definitely happen.

It had also been given us
RCONHR bring



Page 70

Koji: green on alkene T2 (3-E) @ 1600-1670

also RNH_2, $ArNH_2$ T1 (3) 1560-1640
(Not the best)

also C=N T1

also guadinium salts T1 (13) 1600-1680

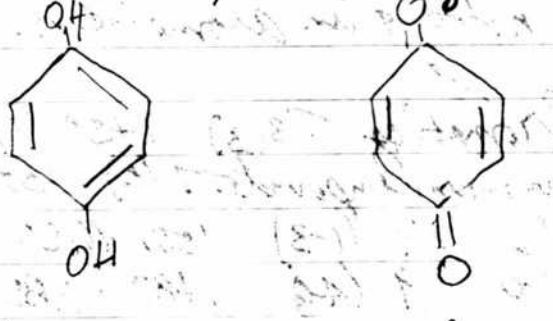
also Carbonyls T8 1600-1950

also N=O T9 (4-6) 1480-1680

Guanidinium salts from the unsaturated amines (also not labeled aromatic amine).

Aurum mela an important comment in the section on carbonic acid derivatives on p 465. She says the spectra of urea and its substituted derivatives show the characteristic patterns of primary secondary & tertiary amides.
It looks like it's hard to tell them apart.

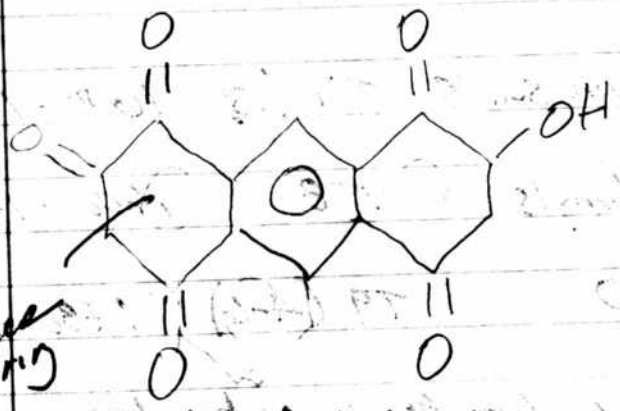
Polycyclic ~~quinone~~ quinones p366 Aurum seem right on target. 1635-1655



hydroquinone quinone

Koji: p 43 also has "extended quinone" @ 1645 vs our 1644
Can't get any closer.

Polycyclic quinones occur in some bacteria, fungi & parts of higher plants.



notice no resonance in this ring

This is to idea

" PQU's

PAH's polynuclear aromatic hydrocarbons

It certainly looks like we have the hot bottom.

* polymeric phenols
* polycyclic quinones

Now lets go to 1516.
This is strongly a Nitro Group
IR Spec gives 1520 for a nitro.
IR Pal says a nitro & an aromatic nitro.

Koji gives us

Aromatics	T3(3)	1450-1600
Ammonium Compounds	T7(6)	1500-1600
NO_2	T9(1-3)	1500-1650
N-O	T9(4)	1480-1680

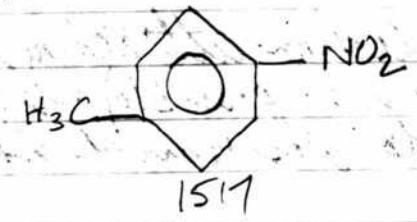
The nitro clearly dominate the packages.
1516

So Koji, before we go to Avram, clearly sees
in a nitroso group C-N-O
as an Aromatic Nitroso Circa 1500.
Nothing else really comes close. Aliphatic
nitroso is @ 1550.

We therefore already have a hint for Avram.
Two sources IR Pal & Koji are
both seeing an aromatic nitro group.
specifically a nitroso w/ Koji

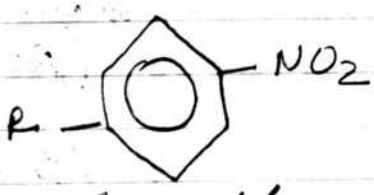
Avram is already showing us that the nitro groups
are likely attached to an alkene.

What we see now is that we seem to be dealing w/
something of the nature

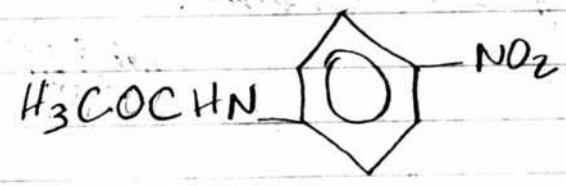


1517

We could close it to



but our best fit is actually more like

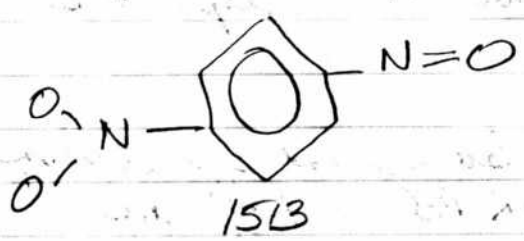


This would be more realistic

R = OH
OCH_3
CH_3
$NHCOCH_3$

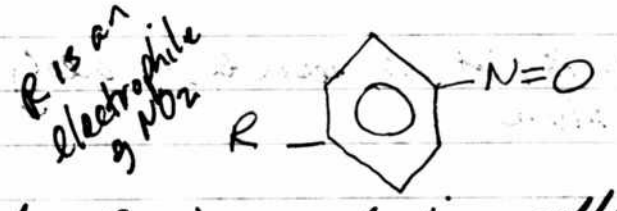
Now, we look @ Avram a little closer.

We can indeed have an aromatic nitro so which is very close with a para-substitution of NO_2 so



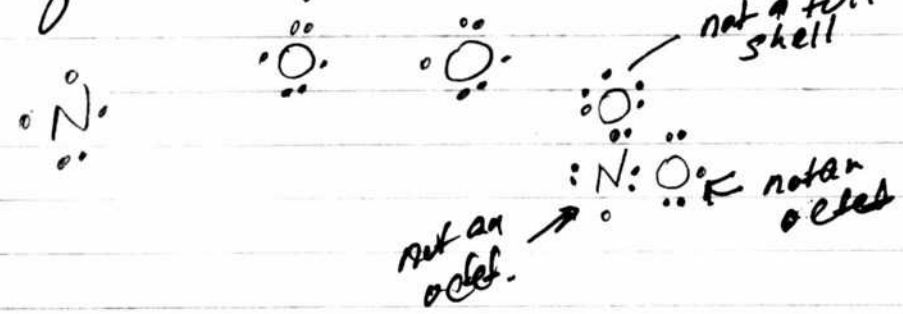
and in this case, there's no symmetric asymmetric pair of frequencies to be worky with.

We could also say

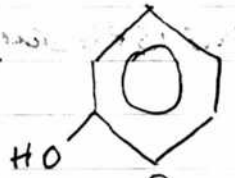


where R is an electron attracting substituent. (para-substitution is probable) eg NO_2 (ortho & meta positions do not affect the frequency).

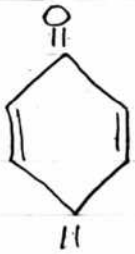
Referral to p 313 Avram.



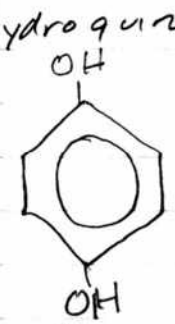
The a all v. important.
You have the following components



n≥2 polymeric ~~polycyclic~~ phenols.

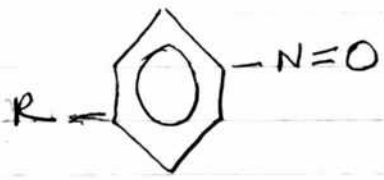


n≥2 ~~poly cyclic~~ polycyclic quinones



hydroquinone

(fully conjugated cyclic dime structure)



Aromatic Nitroso (double bond)
(Probable substitution—para)
Electrophilic

Lets move on. There a still a lot of activity.
(1234)
IR Pal. We apparently can have a nitroso again
But IR Pal also shows us an aromatic ether Ar-O-R
Now IR spec gives us an aromatic ether also
@ the top of the list (1220-1260)

not true R is an alkyl group
Now, it seem to me that could simply be a phenol and it would qualify.
R=H

↑ look @ next page!

This would be perfectly in keeping

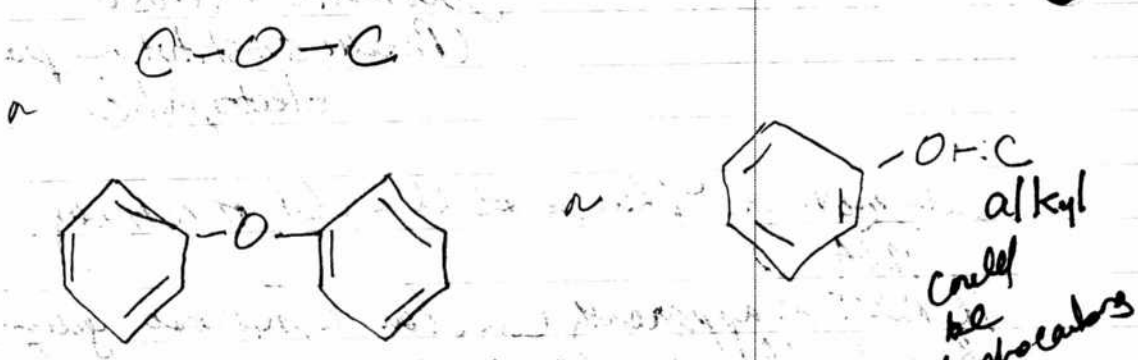
Our next strongest is @ 1034

IR Pal gave an S=O.
IR spec gave nothing strong.
Koji gave us an aromatic ether (this is the same)

Sulfur bonds are supposedly intense.
This is not intense.

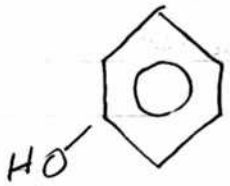
POC is also an intense absorption
this is not intense.

An aromatic ether makes sense.
An ether is:

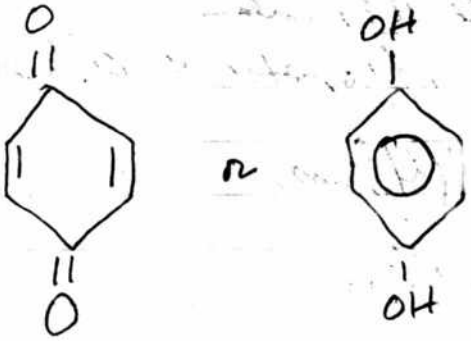


We now have two aromatic ethers
which indicates a separate bioassay type.

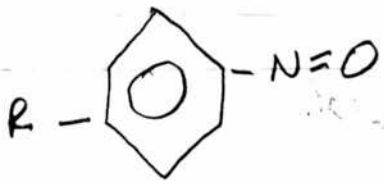
Env. Filament Project
Thesis Sand. You now have.



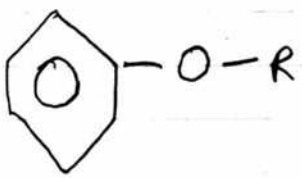
$n \geq 2$ Polymeric phenols, polyphenols



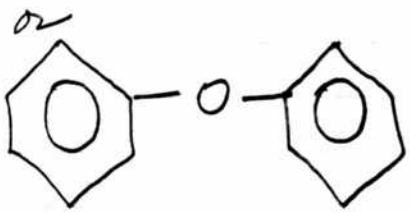
or $n \geq 2$ polycyclic quinone



Aromatic Nitroso
R = probable electrophilic para substitution.



Aromatic Ether
when R is an alkyl group



and CH_2, ~~CH_3~~ ?

Alkanes

Now lets move on to 694.

We see an aromatic a an alkene w/ IR spec, which is exactly what we expect.

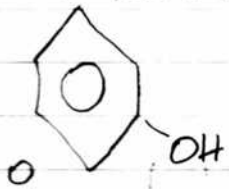
Now lets pick up the alkane
We have 3061 Aromatic + alkene - good

2919 Alkane (methylene) CH_2

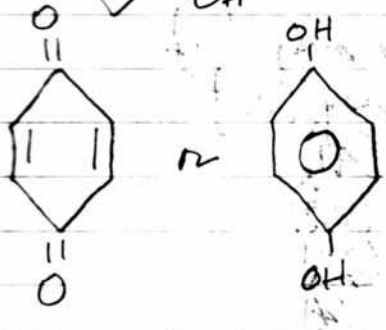
2854 ~~2581~~ Alkane (methylene)

We clearly have a CH_2 group but not a CH_3 group.

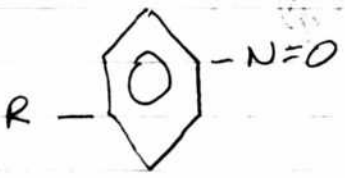
Ok, we have it pegged.



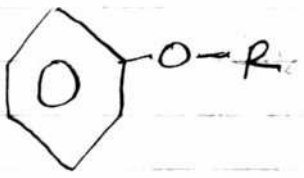
$n \geq 2$ Polyphenols, polymeric phenols



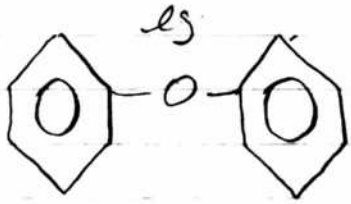
$n \geq 2$ Polycyclic quinones

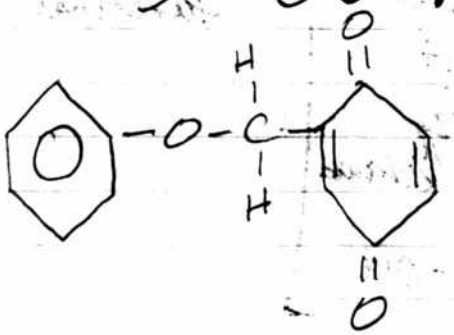


Aromatic Nitroso
R = electrophile w/ para-substitution (probable)



Aromatic Ether
R = any alkyl group





CH_2 alkane

Now we go to the Problem.
But before we do so, let's do a SDBS search on the filament

~~3251~~ 3265
1644
1516
1234
1034
3061
2919
2857

15-65 too many (21)
15-60 too many (170)
10-80 (41)
10-75 (29)
10-70 (22)
10-65 (14)
10-60 (9)

CH_3
-O-
Ar
C=O
NH
CH_2
O
||
C-OH

SDBS Search Functional Groups

Now on to Protein Complex

3405 Amine 3300-3500 dead center

Alcohols also from 3200-3650 strong, broad
Alcohols 3500-3600 free
2500-3200 polymeric OH

2960 CH_3
2946 Alkane
2922 CH_2

IRSpec
2098 Isocyanide C-N-R Table 4 Koji
Alkyne
Remains Uncertain 2100-2260 Not Broad

$C\equiv N^+-H$ T1(12) 1800-2200 Look @ this
Unsaturated Amine "(Immonium Band)

Koji - Aromatic Isocyanate $-N=C-S$
is centered on 2085. vs 2098 measured.

The center of the "Immonium" band is 2000 (1800-2200)
Check on intensity call.
Notice the parallel of the lipid - does the make sense?

Next is 1738.
Carboxylic Acid @ 1735.
C=O unsaturated or aromatic.
Esters also @ 1735.

You need to look @ correlative peaks.

1738
In IR Pal, no matter what you are looking @ it is always an only an ester.
The range is 1730-1740 which I feel fits well. It is indeed a sharp peak.

It is listed as either
RCOOR'
or
RCOOR' 6 ring.

Now, if it is RCOOR' then we also should have a strong absorption @ 1320-1000

With the 6 ring version, there are no associated vibration absorptions.

From Koji, it looks to me like we have a straightforward ester.

$$R-\overset{O}{\overset{\|}{C}}-O-R'$$

Because in addition to IR Pal & Koji, Koji says we expect two additional absorptions @ ~~1350~~ 1300 & 1050
We have them both. 1050 is big (must be combined w/ something else) and we have 1300 weak.
I am voting for a straightforward ester.

For an ester, R & R' must be carbon based.
For an acid, it is COOH by itself.
Actually, an carboxylic acid is RCOOH
and it ends in =O & -OH
So an ester really is different. It does not end.
It is a linking functional group.

The topic here is Carbonyl Compounds,
not Carboxylic acids.
How do we know this?

Notice in the Fast & Easy spectrum that
Carboxylic acids are centered around 3000.
The carbonyl group is definitely around 1700
but the alcohol portion is around 3000, not
3400.
Now with alcohols, that is a different story.
Alcohols are indeed centered around 3350
but they do not have a carbonyl.

This says to me that we must definitely can
have an OH group with a carbonyl and
so then mean an acid. We likely, however
also have an amine. The suggests an
acidic proton. Let's go back to Avram
& what she really says about alcohols.

This is a very unsteady case. Continue ...

What we see, where we see that the hydroxyl group, i.e. the alcohols, is practically absent for:

3200-3400 alcohols
(actually the hydroxyl groups)

The say we expect to see an alcohol centered at around 3300, plus or minus (remember it is very broad).

So, our 3405 is already really pushing that limit. It could indeed be an alcohol, but it is likely combined with an amine.

Free alcohols, which apparently do not really exist, are okay near the 3600 level. If it is broad it can extend all the way from 3250 to 3550. Look @ that!

and we are well within that range.

So

Avram says	3200-3400
Toolbox says	3250-3550.

Note this

There is a huge difference. There is a lot of slop w/ alcohols.

What does Koji say?

Koji covers all the way between 3200 – 3600
But with very important qualifications.
If it is broad it is 3200 – 3400
If it is sharp it is 3500 – 3600

So you see, it "depends".

You see that our shape is broad. But it also has a peak. Not only that, it has a small shoulder @ 3500.
This means an R–NH_2 group
or a pyrrole, indole, etc. (whatever that is).

The sharp peak @ 3405 could also mean an Ar–NH_2 as well.
So we know we have an amine but we also have an acid.

So what you are now saying is that ya have an

OH group
Amine Group, either NH_2 or NH
Alkanes
Apparently R–N=C=S

and now we look at the Carbonyl group.
IR Pal is positive by an Ester group.

Our difficulty is that we are caught up in the debate as to whether we have a carboxylic acid or a alcohol & an ester.
How do we know?

Well, remember the carboxylic acid in fat & soy was centered around 3000 so that is a pretty big difference from 3400.

What we see now is that koji combines the carboxylic acids within the carbonyl groups, while ozone splits them up.

* This was exactly my question.

It shows that the problem can be handled in more than one way.
But notice koji. With COOH he gives 2500–3000 as very characteristic. Then exactly what I was noticing that we are not matching.

Also he is giving the carbonyl groups @ 1760, 1710 & 1720.
This also does not match us so there is another way of knowing that we

ARE NOT DEALING W/ A CARBOXYLIC ACID

We are however, dealing with an

Amine
an Alcohol (OH) ie hydroxyl group
and almost certainly an ester.

So next we separate away the ester and the amino acids are fully understood.

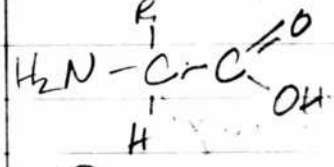
Guess what? The two acidic amino acids are set up for alcohols forming ester existing!

Question: Why are the two aspartic acids acidic? What make them acidic? They wish to accept H^+, or protons I can see that.

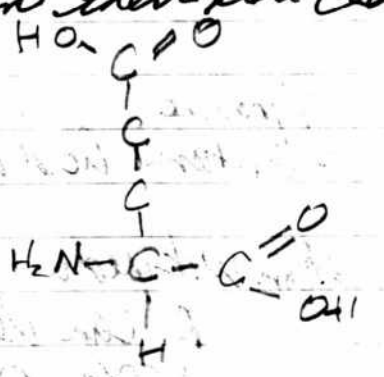
Definition. An acid is an electron acceptor or a proton donor. Ok, I am confused, but these definitions are correct.

They are acid because they "have an extra Carboxylic acid function in their side chains"
McMurray India p 103

Lets look @ Glutamic Acid:



Generic
Amino Acid

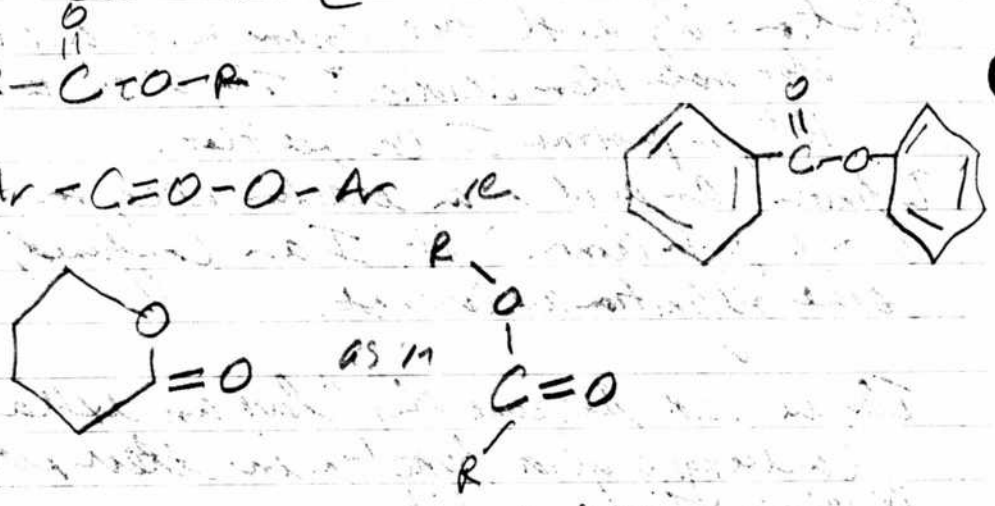


So it adds another COOH group & thus make it acidic.

Now we have an understanding why glutamic acid & aspartic acid are acidic – they have a COOH group as R in the amino acid structure.

Now we can go back to 1738.
We have determined that we have a
OH group
NH2 or NH amine groups, possibly aromatic
and likely an ester.

From Koji, we have several types of esters that can occur @ 1735 (vs) or 1738):



Tyrosine
& Glutamic Acid are strong candidates.

From Wikipedia:
R can denote any alkyl or aryl group or hydrogen.

Esters are derived from a Carboxylic acid & an alcohol.

So it seems to be the most that we can say now is that we very likely have an ester. So current claim is

Alcohol
Amine
Ester
Alkane
R-N=C=S

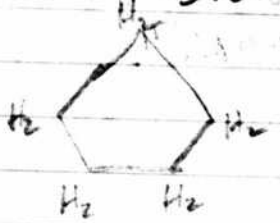
OH
NH or NH_2
O
R-C-O-R
R-N=C=S
CH_2, CH_3

Next is 1457:
IR Spec gives alkane at 1470 (strong)
benzene ring @ 1450-1600 weak
* the does not look favorable.
IR Pal gives RCH_2CH_3

Actually 1470 & 1380 are both alkane. This looks good & it is helpful for backbone.

It appears that we have cyclopentane.
A strong match here w/ Avram p 145 & 147

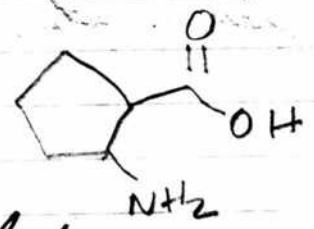
Vs	Avram
1451	1455
1315	1376



Cyclopentane.
Highly flammable
Boiling Point 49°C !!
Water Insoluble

cycloleucine?

Cyclo leucine is a non metabolizable synthetic amino acid; formed by cyclization of leucine.



Then the bad news.

But, before that, let's look @ other amino acids that have this structure.

Proline seem to be one
Tryptophan has others, however.

Proline is important in forming links between protein chains.

We do seem to have a need w/ proline in the sequences.

Proline is cyclic.
Technically, apparently it is not an amino acid, it is an "imino acid".

It is apparently non polar.

It is involving in linking linking & the Conformation of proteins.

Glutamic acid can apparently lonely form from proline.

Above from "The Biology Project" Univ of Arizona

We have Confirmation of 1457 from one source (Cold in Bluefin) and of 1375 from another.
(Int'l Jour. of Scientific & Research Publications)

1455 is also confirmed from the Jour. cited above
1376 also.

(1) We also have Confirmation of our 1630 (1640) in the such noted of Table 3.
"to an extended left-handed poly-pro helix with a non bonded H group to the Carbonyl.

(2) We also have 1456 confirmed (neg. side) bending (δ) (γ) absolutely
γ (oddball stuff)

with bending (δ) CH_2
It can also be the γ (Stanley) CN

(3) and 1375 (δ) CH
The 1300 also seems to be in there.

The main C=O absorption is unusual compared to other amino acids.

There is an entire table devoted to Proline on p 154 of this paper by Barth

"The Infrared Absorption of Amino Acid Side Chains"

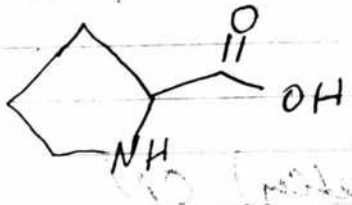
Journal: Andreas Barth
Progress in Biophysics & Molecular Biology 74 (2000) pp 141-173

"Amino acids side chains play fundamental roles in stabilizing protein structure and in catalyzing enzymatic reactions"

Proline is aliphatic & hydrophobic

Proline plays an important role in molecular recognition, particularly in intracellular signaling.

It can bind/~~stick~~ to a surface containing aromatic residues.



Page 92

Now we have I have a match of all of these in proline:

OH
Amine
Ester
Alkanes
$R-N=C=S$

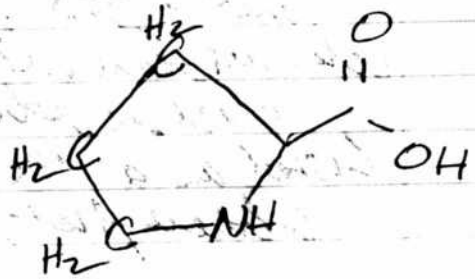
Alcohol

$NH-NH_2$

$R-\overset{O}{\overset{\|}{C}}-O-R$

$CH_2(CH_3)$ not within proline.

and



And Proline is

Proline a a major amino acid found in Cartilage, repair of muscle, connective tissue & skin damage; Joints & tendons Proline is a Component of Collagen.

Proline is oxidized to form Glutamic Acid. Proline is involved in tissue repair & wound healing.

(Side note: there is one phenolic lipid in the lipidmaps.org database) it is called "phenolic phthiocerol")

Back to IR analysis of protein.

We have something story that has happened @ 1038.

IR Spec has an amine. 1020-1230 medium, overlapped.
Also we have alcohol @ 1040-160 which is strong & broad.
Listed as a "C-O per primary" which we have not seen.

IR Pal gave Amine, C=O, P, Carboxylic & Ester

So what is a primary alcohol?
A primary alcohol is (1°)

2° has 2 R's
3° has 3 R's

Koji gives alcohols
P-O-C is dead center narrow range
1030-1050 T12 (3)
Thioketones 1040-1200 edge of band
Sulfoxides also have 1173

Even the 1038 looks like 2 components
so the candidates seem to be

Alcohols	T5b	1200 - 1000
Ethers	T6	1075 - 1020
Aliphatic Amines	T7 (5)	1230 - 1030
Thiolesters	T11 (6, 8, 11)	1200 - 1040
P-O-C	T12 (3)	1050 - 1030

Of these P-O-C is the tightest range.
~~but P-O-C should show odd. absorption~~
There is indeed a possibility.

The alcohol seems like a real possibility
1050 ± 15 What does p-OH mean?
p means a primary alcohol.
s means secondary, t means tertiary.
This fits again.

There is no conflict between overlapping P-O-C, primary alcohol and an ether.

An ether is C-O-C so we assume R-C-O-C-R
We have no evidence of this yet.

Aram has many many choices on 1038
so there is hard to sort out. The alcohol is
the only safe bet so far.

It is a broad strong peak, however.
Koji shows examples of an alcohol that matches

a primary alcohol is the simplest interpretation of 1038.

There is all we keeping that.

Our next is @ 724.

724 strongly hint of a monosubstituted) Aromatic (760-750) but you would need some corroborate on this. Where would you get it?

An arene require

NO	3100-3000	several bands
NO	1630-1590	
NO	1520-1480	
YES	900-650	

so an arene does not fairing well.

My next strongest candidates are:

Alkene 650-1000
Alkyl Halide 600-800 strong.

Sep 19 2015

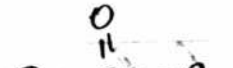
Now let's take a look @ Catalyzed filament.

1. The first thing that you notice is what looks like a strong shift towards the hydrocarbons
2. There still seems to be a lot of activity in the amine region.
3. 1642 This was assigned, via Avram to polycyclic quinones. p 366
4. What we see now is a shift from 1642 to 1628.

IR spec says 1628 as alkene 1620-1680 strong

Avram says carbonyls are 1650-2000.

Unsaturated ketones seem to be in our region. 1620-1640. What is a ketone?



is a ketone.

Since polycyclic quinone

quinone.

The formation of a ketone would make a lot of sense. It would be a separate of the ring structure.

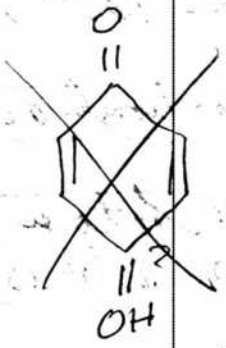
	Δ
Ortho	1
Meta	2
Para	3

Koji has

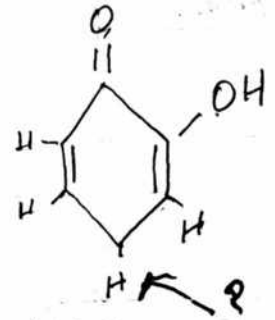
Alkenes	1600-1670	T2 (3-2)
RNH_2, $ArNH_2$	1560-1640	T1 (3)
C=N	1640-1690	T1
Guanidium Salts	1680-1600	T1 (13)
Carbonyl Compounds	1600-1950	T8
NO_2	1500-1650	T9
N=O	1430-1490	T9

NO

So many many choices again.
Need Corroborative analysis of ketones
It does look like
Koji is saying

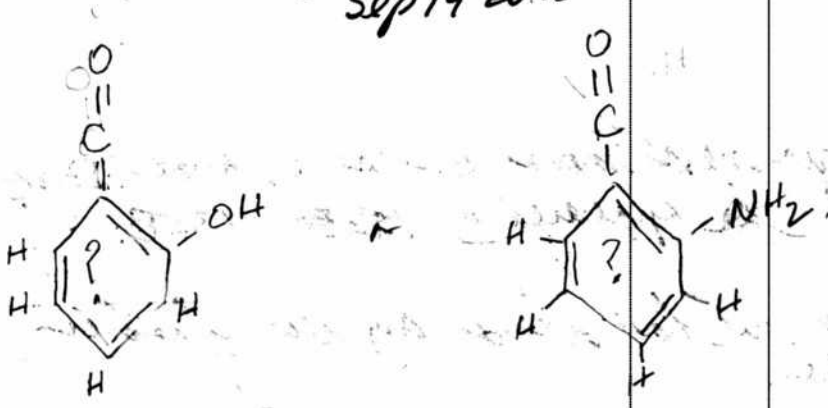


What does ortho mean?
Ortho means Δ=1
so it would have to be



The rest look reasonable
doesn't it?

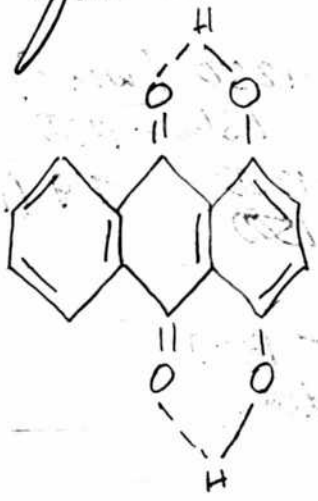
Should have CH_2 here.
Why would it not be
He did not say this. He said not COC_6H_4OH



Oxygen make a double bond.

A very interesting presentation takes place on p379 Avram.

1627 is shown under the "hydroxy quinones". Sound familiar?



This is a solid phase of a "hydroxy anthraquinone"

* Looks like good work here

She also states that the OH group "involved in chelation" gives rise to broad absorption between 2500 & 3200 which is certainly what we have.

This seems to fit like a glove

Page 99

Now, our next move is that a ~~huge~~ strong peak ha appeared @ ~~1350~~. 1358.

We did not have to look any real further before.

IR Spec

Nitro is strong @ 1350.

Not only that but it is a nitro aromatic.

Nothing else is really compete w/ it.

IR Pal also had a nitro aromatic & aromatic amine.

Koji Res

not good		alcohol	1500 - 1250 (weak)	
		Aromatic Amines	1360 - 1250.	T7 (6)
1537	1358	Nitros	1370 - 1250	T9 (1-3)
	1628	and alco:	1650 - 1500	

SO_2 1440 - 1310

not to be neglected.

Page 100

What a shame we no alcohols w/ amines mixed in. hydroxy quinones

and now nitros and/or aromatic amines.

Koji has a thorough discussion on the "ammonium band" on p 41. Also tests are also described. Amine salts are also discussed - this is an interesting discussion.

The aromatic amine is on the extreme edge of the band, so this is more questionable.

The nitro band seems a little strange to me since it overlaps on 2 sections 1650-1500, 1370-1250

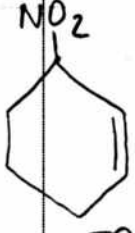
Aram shows phenols @ 1350 strongly.
also NO_2, but not amine.

Aram p 306 shows

$(CH_3)_2CX-NO_2$ but this is too active for me.

On p 307, we seem to be getting closer.
Nitroalkane shows.

The suggests an aromatic nitro.



1550 vs 1537
1363 1358

Nitrobenzene

vs

1530 1537
1353 1358

After reviewing Avram p 309
Our assessment is that we have
an aromatic nitro.

Our best call at this point is a

and our best estimate for
X is COOH
but we do not show
an acid here.

Now let's look @ nitrographs in Koji

This looks like a respectable match
w/ Koji example on page 170 (Problem #45)

Catalyzed Env. Filament.

We are showing that we have

1. Alcohol
2. Amides
3. hydroxy anthraquinone
4. Substituted aromatic nitro

Next, we have a lot of activity clustered @ ~ 1055.

But we have additional activity @ 1184 alcohol
1099 alcohol
1055 alcohol
985 Alkene

IR Spec @ 1055:
Alcohol comes out dead center @ 1040-~~1060~~ 1060

1184 also shows an alcohol @ 1150-1200
Also 1099 has an alcohol, strong
985 has alkene.

None of them shown a surprise.
Not effect of catalysis.
Not effect that it seems to show the products of nitrate & amine compounds, & alcohols.

The data look like good work.

You have made some great progress therefore. You now have made a very serious review of:

1. The Lipids
2. The Protein Complex
3. The Environmental Filament
   The Catalyzed Environmental Filament.

We are now in a position to review the Hench paper now and to draw parallels.

You are also in a position to begin writing a paper. I am recommending that you only show group frequencies.

These are all very important findings and analyses.

Next is most likely biological samples.

Urine
Serum
Hair
Saliva

Page 105

We can now look @ the culture.

There is a very clear cooky spectrum.

3308 a very smoot peak
Certainly look like an alcohol.

Alcohols & Amines are noticed.
Alcohol 3200-3650 strong & broad.
Koji - polymeric alcohol is dead center of 3410-3200.

Avram is also very direct on this, as we have seen before. The hydroxyl group on phenols absorbs from 3330-3500, dimers @ ca. (here ca) 3500 and polymers @ ca (circa!) 3320.

* There you go. ~3320 vs 3308.

* Once again, you have a polymeric phenol.

Now we go to hydrocarbons. 2919 & 2853
CH_2 CH_2
(2926) (2853)

So now we have
Polymeric phenols
CH_2

CDB Culture Filaments
Sep 20 205 Analysis
Olympic N.P.

1606 is next.

IR spec gives Amine 1640-1560
Then is carbonyl region.

Koji: Alkene 1610-1600
RNH_2, $ArNH_2$ 1640-1560 T1(3)

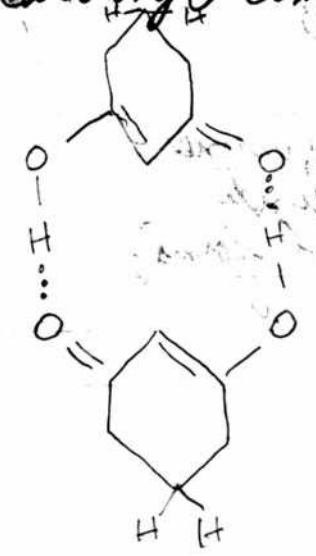
Carbonyl Compounds 1950-1600 T8
NO_2 1650-1500 T9
N=O 1600-1480

The problem I see w/ amine is that we have no activity from 3300-3500.

P380-383 Avram is of interest w/ carbonyls.

It is explicitly stated as a

Dimeric Enol Form and they are listed under the category of 1,3 Dicarbonyl Compounds under Carbonyl Compounds. It looks very specific

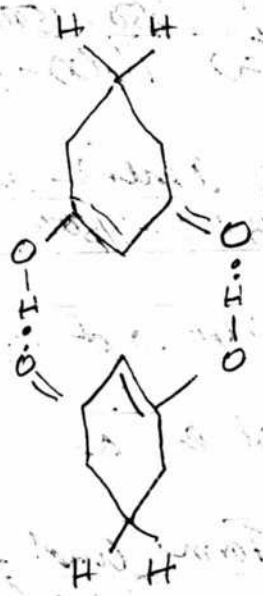


Notice the amazing similarity of this to the catalyzed environmental pollutant. Which came up one page away on p 379 under hydroxy anthraquinones. Then the rather amazing.

Therefore we now have:

1. Polymeric phenols
2. CH_2
3. 1,3 Dicarbonyl Compounds, ie Dimeric Enol form

and the last structure is highly connected to the previous.



What is the significance of these Compounds?

1. Polymeric phenols
2. Hydroxy anthraquinones
3. Dicarbonyl Compounds (Dimeric Enol forms)
4. Proline
5. Phenolic Lipids

4 main Tools:
1. IR spec
2. Chem Toolbox
3. Koji
4. Avram – the final gun.
5. IR pal w/ PC if needed.

Dicarbonyls may be much more acidic than monocarbonyls.

Yes, the hydrogens of dicarbonyl compounds are especially acidic.

Dicarbonyl compounds may be important as in vivo contributors to protein crosslinking.

Enols are compounds that have alcohol groups substituted onto alkenes.
"alkene – ols"

The next major peak is @ 1061.
This may have some similarities to 1038 of the protein complex.
IR spec graphs

Alcohol is indeed a candidate 1040–1060 strong, broad
Chem Toolbox has been underutilized.
There also a Silane Si–O–C aliphatic
Alcohol is also listed as 1030 – 1100.

Koji does give a primary alcohol at 1050 ± 15 cm⁻¹.
That's a good match.
Silicates are listed in Koji, obscured in Table 13
on p 51. It is also listed as strong. So
it is possible but alcohol seems the higher
probability.

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Now to Avram (bypass IR lab for now)
1061
Stone Avram p258.
Avram once again goes into the level of detail that is actually needed.
While she says that indeed conventionally it is taken as

1050-1100	primary alcohol
1100	Secondary alcohols
1150	Tertiary alcohols

which would place us in the primary alcohol range she tells us again that life is not always so straightforward a sample.

She says
"A band between 1050 and 1085 may arise from:

1. a primary straight chain aliphatic alcohol
2. an unsaturated secondary alcohol
3. a secondary 5 or 6 membered cyclic alcohol.

Note this.

How is that for a reality check?

We seem to be, quite possibly right square in the center of category 3. Let's read further. With the primary alcohols, our candidates would be

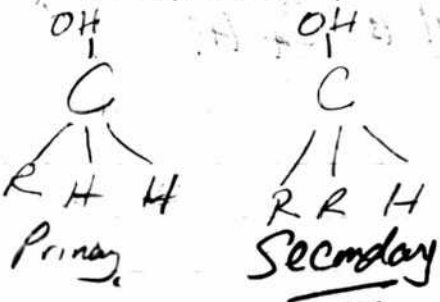
- n propanol
- n Pentanol
- n Hexanol
- n Heptanol
- n Octanol
- 3 Methylbutanol

} these are aliphatic alcohols (straight chain)

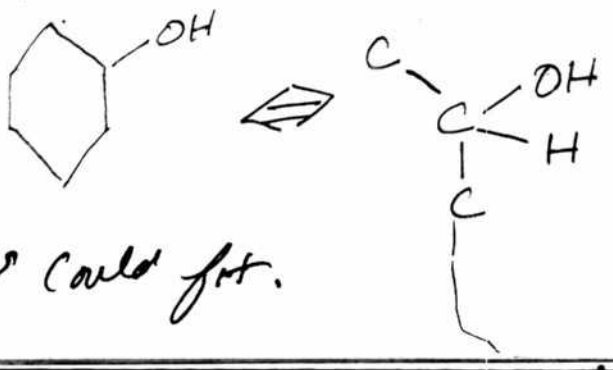
But in terms of the cyclic structure we see, cyclohexanol is in our range @ 1064. Then it lists on p 259 under cyclic secondary alcohols. The matches category #3.

Cyclohexanol. Note that it is cyclic but not aromatic.

but secondary mean



Secondary cyclic would mean:



So this could fit.

So this fits as a secondary cyclic alcohol by no problem.

We see that she could get reasonably well with our polycyclic alcohols. She is not the case, however, as an aromatic alcohol.

Now we see that Avian has another option up her sleeve. On p 260 we have unsaturated and aromatic alcohols.

Remember that an aromatic alcohol is a phenol. Given that we have a phenol she could be right on track.

We have the form:

$$R\text{-}CHOH\text{-}R'$$

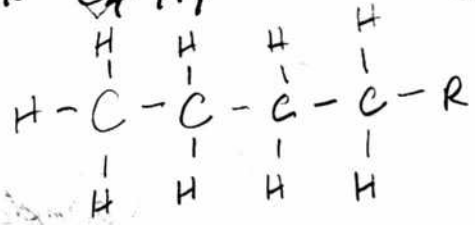
~~With a aromatic this is the form~~

and we choose the selections.

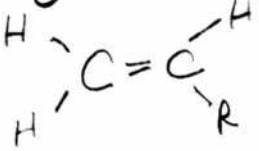
R = butyl — What is butyl?

R' = Vinyl — What is Vinyl?

Butyl is C_4H_9



Vinyl is ethylene minus one hydrogen C_2H_3



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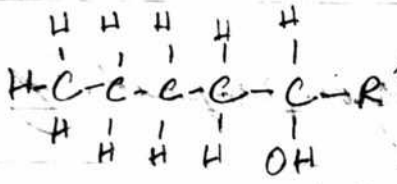
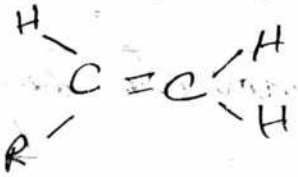
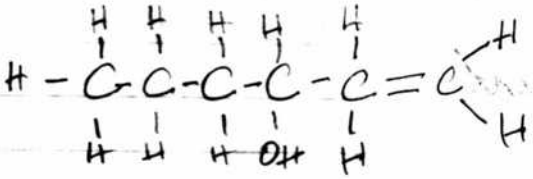
So w.r.t. an aromatic, that means what?

We supposedly have

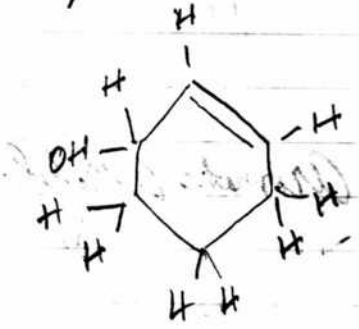






As you recall, a vinyl group means that the chain ends in an alkene & 2 H's.

Now, how to turn this into an aromatic:



That would seem to be the aromatic form.

CDB Culture Filament:
Sep 20 2015

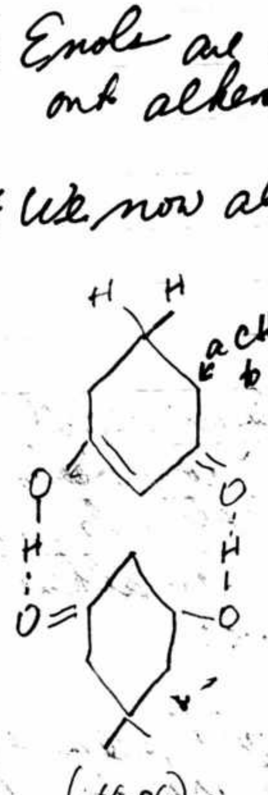
An aliphatic chain seems to be less likely under the circumstances. The means that our current set is.

3308 1. Polymeric phenols (these are aromatics)
2919, 2853 2. Alkanes, CH_2
1606 3. 1,3 Dicarbonyl Compounds Dimeric enol form — I wonder where you found this?

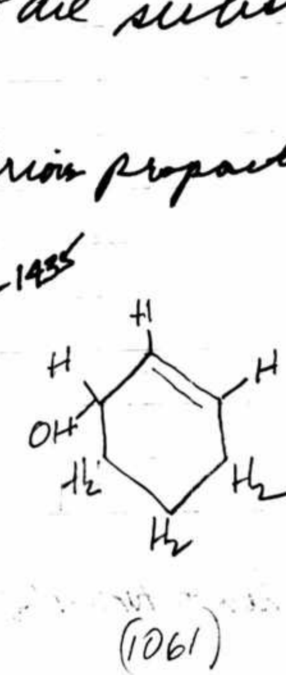
It was p300-303 Arnam. This was within carbonyl compounds — Enols are alcohols that are substituted onto alkenes.

1606 & 1061 4. We now also show the series proportionally both:

See Note Note! page 4 pages ahead.



and



DiCarbonyl Compound
Dimeric Enol
(alcohol substituted onto alkenes)

Aromatic Alcohol

1431 5. We now have a methylene group adjacent to a carbonyl (1431) and the smaller perfect sense. See note in drawing above. (a Ketone)

1717 6. a Ketone. Straight forward.

Now we return to pick up 1431

IR spec: No perfect match.
Benzene ring is our closest. Not good.
Chem Toolbox:
Phosphine is in the center of this range.
Thio seems to be our best match. P-C
Alcohol is on the extreme end of the range 1430-1320
Sulfate is only fair 1450-1340.
also near the edge of the range.

But if there is a phosphine, we also expect
Another @ 1350-1250
Thio is slightly outside the range.
Arsen is not listing Phosphorus.

1430 is alkane from Arsen, there is more
water like?
Alcohols + ~~Phenol~~ hit 1430
Tertiary Amines are also there (not important
as single peak)
The latter may be a lot more obvious.

Check Koji on these first.
Koji documents the alkane here.

Even in Koji, the case for alkane and even alkene is very strong. In the paragraph, he has:

2950-2850	2919, 2853
~ 1465 (1400-1460)	1431
1380	1370

You essentially have all of these

I would say that this is a good match and all we need to do is test it further. Refinement of alkane-alkene seems to be the greatest benefit.

~ 1431, the most likely seems to be $-CH_2CO-$ (1440-1404)

1370 tends toward $O-CO-CH_3$ (1380-1365)

This may serve as a starter. Now Avram.

Here is a question. What is a single C-O bond called?

It appears to be an ether, but only when it bridge two carbons.

Avram does not seem to be handling the in the hydrocarbon section the way Koji is. What is Avram handling this?

We are on 1431 & we're 1370.

Avram does have 1460 & 1380 as a vibration of the methyl group appears to the naked to me. She may be calling it scissoring.

A methine group is CH
methylene is CH_2
methyl group is CH_3
methane is CH_4

Avram definitely covers methyl vibrations on 1375 on p 131 & 132

She specifically says methyl vibration 1365-1380. Then is us.

Now she also mentions the methyl @ 1460 but then notes the methylene group @ 1467 so that further separation cannot be made.

But I notice that we are @ 1431. We are far enough far away that we should be able to tease them down.

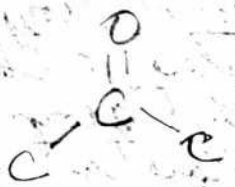
There is something else happening here & Koji may have it.

The note @ top of p133 Avram is over clue that she knows what's going on.

Avram then suggests the variations at least some of to explore..

1. strained cycloalkanes p 152
   I see no match of 1431 occurring here.

2. Next is ketones.
   What is a ketone?



"

Well, there are some "cycloanones" on p 366 that are falling into this range but they seem to show two absorptions close together that we do not have so it is not identifiable @ this stage.

Nothing is clear on this yet.
Let's look @ IR Pal.
~~Before that~~ IR Pal gave S=O & Aromatics

What we can try here is to look at Avram in a correlative sense.

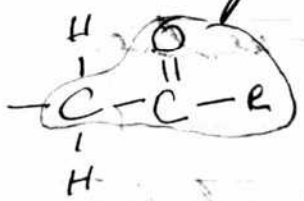
Well, then, that our 1st candidate under this method is a cycloalkane w/ n=4.
$(CH_3)_n$

Note: Note Page!

Methylene adjacent to a Carbonyl identified.

Koji seems to be the most on track here.
I cannot find the equivalent section in Avram.

One last alternate is a methyl group
w/a Co attached.
"a bending of a methylene group"



It appears that this is a Carbonyl group that bends the methyl.

If R = a Carbon, then there is a ketone group!

Great, it looks like we have figured it out.
The answer comes from AVRAM p 364 at the bottom and from Koji @ the end of the alkane section.

What we appear to have is a methylene group adjacent to a Carbonyl, which makes perfect sense. See 4 pages back.

Everything remains entirely consistent now.

The takes care of 1431.

We now go to 1717.

IR spec. A. Very clear ketone identified 1715-1720

Dead center.

A ketone is C-C(=O)-C

and then we defended what we have. So there's a motor.

This is really all that we should do.

I think that we have succeeded well.

Sep 21 2015 Monday Olympic National Park
A sunny day! After raining for 1 week
across the board.

You have a lot of spectra that have been produced.
Unfortunately only a few are of the highest quality.
You have not subtracted the background properly
for the majority of them. You need the actual conditions,
ie two KCl discs in place to complete the background.

It will help to again sort the spectra by quality and
priority. Around Sep 8, whatever the latest data
on file is, the best work should have been done.

Eg, the env. filament was repeated. What we
are uncertain of is whether headspace was used or
not. When you get back you should be able to tell better
but you may have to repeat again.

I now have the spectra & papers organized into a
much more logical fashion.
On the spectra, the organizational criteria are now:

1. Priority
2. Quality
3. Sample type, Disc or headspace
4. Background Removal Complete?
5. Sample Prep Method: Easy, dried, etc
6. Notes

And the research collection is actually quite useful,
interesting & worthy of continuous familiarity & access.

Page 121

What I must to do now is review another IR source that has been acquired but not so far not utilized.

The book is written by Alpert.

There is some very detailed information in the book, especially on

1. Hydrocarbons
2. Aromatics
3. Carbonyls
4. Amides & Amines

Also some very good summary charts eg hydrocarbons p 207
p 292 has a major summary chart.

The book is worthy of looking @ as a valuable source.

Blood Serum [redacted]

3289 Sharp but broad peak.

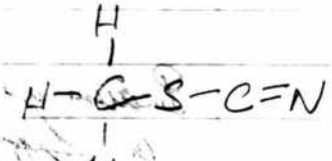
We do however, have an immediate question on Isothiocyanate in blood.

We have a a definite peak @ 2135.
The problem is that the method for saliva test not for blood.

It is also not listed on the screen table.
Koji T4

Then could be either thiocyanate or isothiocyanate
Question: should there be in blood?

It appear to be in the lipids.
It appear to be in the proteins
It appear to be in the cellular filaments



CH_3SCN is a potent toxin

Different versions of SCN are more toxic than others.

Na & K SCN are not considered toxic
Is CH_2SCN also toxic?

Sep 23 2015 near Skykomish WA.

Back to the blood screen. The cyanide groups does seem like it can be a concern. It probably will have to do with concentration levels.
Blood serum does have thiocyanate, but it is probably a matter of how much.
We will research this thoroughly.
Substituent will also be a big factor.

Our first peak @ 3299
We have another source @ 3298.
$\bar{X}$ = 3294.

IR Spec grp alcohol 3200-3650
strong & broad.

also grp amine 3300-4000
weak to medium

Because we have a single pointed signal within a too broad signal (image to determine I believe that we have a tertiary amine within an alcohol.

How does this relate to our phenol interpretation.
Our phenol interpretation was @ ~3400.
Not 3300 where we are now.

3400 is likely different than 3300.
3400 is definitely not a free alcohol and neither is 300.

3400 was in the range of polymeric alcohols
and 3300 definitely is. Dead center there.
Question: How does Koji handle phenols?

Phenols are listed only as 1200 cm⁻¹ in Koji.
I am not sure of this. Check Avram
IR Pal about this issue worked on.
This is incomplete.
Koji does say polymeric but there is more to it.

IR Pal tells us very clearly that Phenols
run from 3200 - 3500.

Buck says phenols are 3620 - 3590.
and acid forms are 2700 - 3300.

Also: Phenyls are not Phenols !! Mistake has been made.
Phe nil Phe nŏl

No wonder there is confusion here. The sources
are not in agreement.

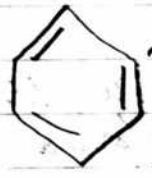
I can tell now that things finally are resolved.
The best source, without any doubt,
seems to be AVRAM.

Lets sort this. I see now why I have been confused.

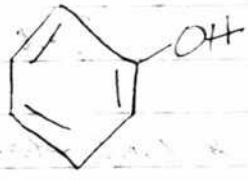
1. First, phenyl is not phenol!

What is phenyl?
The phenyl group is on p 362 & 433 of McMurry Hadia.

Ok, a phenyl group is actually highly related.
Phenyl can consider the aromatic ring as a substituent rather than the primary a phenyl is therefore



While the phenol is



In a sense, a phenol is therefore a phenyl group where R is OH!

So they are actually quite closely related to one another.

I think that Avram and IRPal are going to end up being better source here.

Avram.

Avram explains to us a lot about alcohols. Alcohols were conventionally explained as OH from 3500 to 3700. This was wrong and was due to method problems.

Free OH is actually much more restricted, from 3650-3600

However! she then goes on to say that free alcohols seldom if ever exist in the real world.

What actually does exist is a broad band from 3400-3200 that is then to be subdivided into 3 Bands!

Then then, is the heart of the problem and of the examination to be made

This is not the case of phenols (or of phenyls for that matter; it is the case of alcohols free or otherwise. We shall look @ phenols again later when we finish this section of analysis.

The three sub bands are:

1. Free hydroxyl (OH) ~3620, 3620, 3620, 3615

2. Hydroxyl dimers ~3485, 3435, sometimes 3620.

3. Polymeric OH 3360, 3350, 3320, 3380-3300

! This is the heart of the matter and an entirely different story.

OK, Avram is really going to town for us. Her discussion on p 249 is very valuable in that it shows that we can distinguish type and structures of alcohols.

It does not need to be a battery now. We can distinguish between

1. Free
2. Dimers
3. Polymers

] alcohols

Free 3620 - 3615
Dimers 3485, sometimes 3620
Polymers 3360 - 3300

4. Saturated & unsaturated alcohols.

3500 - 3300

5. Phenols - Alcohols on a aromatic ring
Free Phenol ~ 3605 (does not exist) phenol dimer 3500
phenol polymer ~ 3320
6. Enols (alcohols substituted onto alkenes)

It also is all fantastic.

The above is the real world and this is where we are headed. Alcohol form a variation are really important to be able to discern between.

Back to Alcohols.

We can see now that the human - rat serum table does indeed mask the band. There is indeed an OH element. There may well be an ether amine as speculated but this does not negate the alcohol band.

So clearly we have an alcohol @ 3300 and it looks like it tends toward a polymeric form. But now we can through #4, 5, & 6 on the table and have (list) on the previous page.

Barbara Stuart is also going to be a very good book for us.

Page 129

The Alcohol Table

Continuing to develop the table at the group frequency level since it does not seem to be expressed well.

Types of Alcohols

~3600 1. Free 3620 - 3615 (doesn't really exist apparently)

~3500 2. Dimers 3485, ~~Sometimes~~ 3620

~3400 3. Polymers 3380 - 3300

4. Saturated vs Unsaturated (p260) & Aromatic (p260)

4b. CH_2 group influence (lowers freq)

4c. Primary, Secondary, Tertiary (p258 p259 p260) (Primary Sec, Tertiary Aromatics p261)

5. Phenols (3500 - 3300)

~3600 Free 3605

~3500 Dimer 3500

~3300 Polymers 3320

6. Enols

Apparently under Carbonyl section of Avram 380-393

Alcohol Table.

The table finally helps us quite a bit and it took some time to sort this out.

Now it seems to appear properly in books.

There are many variation on the alcohol theme. See if you can set the group frequency correct to work up and then a little above from there.

So, back to the blood extract.

The reference blood is given as 3400 and is assigned to NH.

Now we also have a graph, but it actually shows a peak @ ~ 3270, so this not that different than your own. Look @ other source here I think to not table is skipping to OH ~3300 and is focusing on the tertiary amine @ ~ 3400.

I have 3 source to consider. 2 papers & Strat - Maybe also to greek book.

In our paper by James, we do give a spectra of erthrocyte. It does have a peak @ about ~ 3330. On our other plot, we have a peak @ appx 3280. The mean of these is 3305. Our peak average is 3294. This is right in range of what is expected.

The way to me that we do have an alcohol in blood, most likely a polymeric complex of some kind, but not likely to be ethanol.

secondary!

Now let's look @ tertiary amines.
We believe we have an alcohol, even a polymeric alcohol a polymeric phenol.
I also suspect an tertiary amine because of singular peak & the last page claims a primary amine. Let's see.

IR spec already has a amine.
Ko; next for 3294

secondary!

Ko; Sure, still enough, a primary amine, plus a double-bond C=NH @ 3300-3400

The spec on the low end of polymeric alcohols or polymeric phenols and the low end of primary amines.

Now for Aston.

From a net source electron donating substituents stabilize the molecule and therefore decrease the stretchy freq. Electron withdrawing (or electrophiles) increase the stretchy frequency.

At this point 3294 does seem best assigned to a secondary amine (3350 – 3300)
the possible ~~bbb~~ there is more to the story, however.

Assignment in the Jones paper do look correct, ie

3300 ν(NH), ν(OH)
notice hydroxyl is indeed added here.
Jones diagram / spectra is pretty accurate here
there is absorption as you want spectra, not transfer

Next: Jones gives a peak @ 3017 for ν(CH)

I do not have it.
The rat paper does not have it.
What does Stuart do here?
3017 is common on alkene.
I have no evidence of this band.

Next Jones has 2959
Rat paper has 2956
Stuart has 2956 for lipids
We have 2950 + 2952 = 2951 Mean vs 2956

But before we go there! We have a 3056
that nobody shows

3067 could be aromatic, amine, alkene

A very good start on the pyrolysis of hair today
We can now see that we are clearly beyond
of ~ 180°C which is just about perfect.
The darker forms of hair are produced @ other
temperatures.

Chromatogram shows that volatiles are produced
@ over 50°C.

We see the air peak @ 0.433 min.

We may have a shoulder peak over given to that,
what is unknown.

What you are really here is that hair has a lot of
components within it. Remember that there is absorbance current.

You would like control of CO_2, O_2
then NO_2

Page 135

GC Hair Comparison

[redacted] m	mV	[redacted] m	mV
Ø.35	Ø.82		
Ø.43	356.8	.44	376.4
Ø.69	30.65	Ø.71	5.8
2.73	1.2	2.84	Ø.6
3.88	4.8	4.11	7.7
4.56	1.3		
		6.56	.02
9.56	1.6		
		9.86	2.9
11.4	.05	11.27	2.4
13.26	1.8	13.59	Ø.7
17.14	.04	17.7	Ø.2
		23.0	Ø.2
29.52	.4	28.9	Ø.2
30.3	46.4		

Sep 28 2015 Monday.

[redacted]

3. Very interesting how analyses to take place
   1. Thiocyanate strong in both samples
   2. Spectral analyses almost identical
   3. GC is vastly different.

4. Can we create an IR thiocyanate sample?

5. Proline sample?

6. CO_2 GC sample?
7. O_2 GC sample
8. Car exhaust sample
9. Room air sample
10. Hydrogen sample?

We have a good CO_2 record from Healy cooler/ Virgin test.

@ 50°C: Low Current

Air Peak	Ø.47 m	87.2 mV	
CO_2	3.33 m	68.8 mV	
@ 100°C Air	Ø.43 m	143.2 mV	Air comparison off by 10 mm
CO_2	1.25 m m	210.6 mV	

The excellent work.

Page 137

IR Work leaded toward proline spectra

You have analyzed acetone evaporated on a single KBr disk. It has worked perfectly.
CO_2 Comes in @ 2300.

You now have a perfect spectrum of proline.
You have a match.

Sep 30 2015

1. Thiocyanate reaction w/ Fe^{+3} is what we are seeking.

Black green reaction w/ Fe^{+3} when hair volatiles are pumped into water solution.

2. [redacted] sample

What is the pH of the hair solution.
It is alkaline @ 9.0
so Fe^{+3}
green-black
alkaline
reaction
chemical

The combination of condensed tannins and Fe^{+3} produce green black complexes.

A tannin is a polyphenol.

Polyphenols are organic chemicals usually found in plants.

There is the ferric chloride test for phenols.

formation of a red, blue, green or purple coloration

This means hair has a polyphenol & thiocyanate component to it.
polyphenols affect mineral balance in the hair (and therefore likely the body)
Polyphenols inhibit heme iron absorption. Vit C helps to reverse this.

Remember the red wine reaction?
Anthocyanins are involved.

Anthocyanins are phenolic compounds.

So polyphenols react w/ Fe +3 to produce a dark green complex of alkaline ptt.

Search phrase: Polyphenol iron complex

Polyphenols inhibit iron absorption

The complexes formed between Fe+3 and polyphenols are very stable; those w/ Fe+2 are much weaker.

Polyphenols oxidize the iron from Fe+2 to Fe+3 to form thermodynamically stable complexes.

Major Findings

There are mechanisms at interface

1. Oxidation of Fe+2 to Fe+3 (polyphenols do this)

2. Presence of thiocyanate complexes
   O2 interference

3. Presence of prolin in the protein
   Protein linkage
   Joints & collagen

4. Try to identify the phenol peak USP
   1. Camphor phenique
   2. Phenol extract
   3. Hair

Boiling point is 187°C This is high.

We have a problem showing up w/ Camphor Phenique.
We are getting no result beyond an air peak.
How is the sample?
Let's charge to 200°C. for 15 min.

OK, we have mild success now.
We've heated the solution to a much higher level and oven at @ 200°C.

We actually have up to 6 peaks now but they are very small.
We must look up to high-current.
Most of the peaks are w/in 5 min.

Page 141

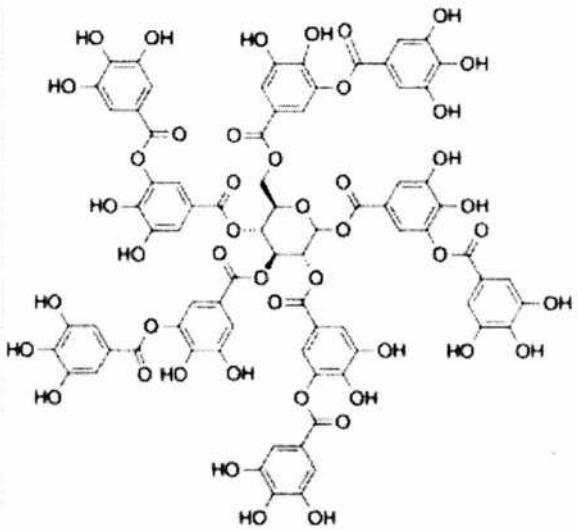
Polyphenol

From Wikipedia, the free encyclopedia

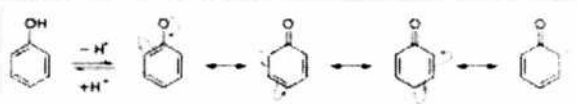
This article is about larger phenolic substances. For smaller molecules, see natural phenol.

Polyphenols[1][2] (noun, pronunciation of the singular /pɑli'finəl/[3] or /pɑli'fɛnəl/, also known as **polyhydroxyphenols**) are a structural class of mainly natural, but also synthetic or semisynthetic, organic chemicals characterized by the presence of large multiples of phenol structural units. The number and characteristics of these phenol structures underlie the unique physical, chemical, and biological (metabolic, toxic, therapeutic, etc.) properties of particular members of the class. Examples include tannic acid (image at right), and ellagitannin (image below). The historically important chemical class of tannins is a subset of the polyphenols.[1][4]

The name derives from the ancient Greek word πολύς (*polus*, meaning "many, much") and the word phenol which refers to a chemical structure formed by attaching to an aromatic benzenoid (phenyl) ring, an hydroxyl (-OH) group akin to that found in alcohols (hence the "-ol" suffix). The term polyphenol appears to have been in use since 1894.[3]



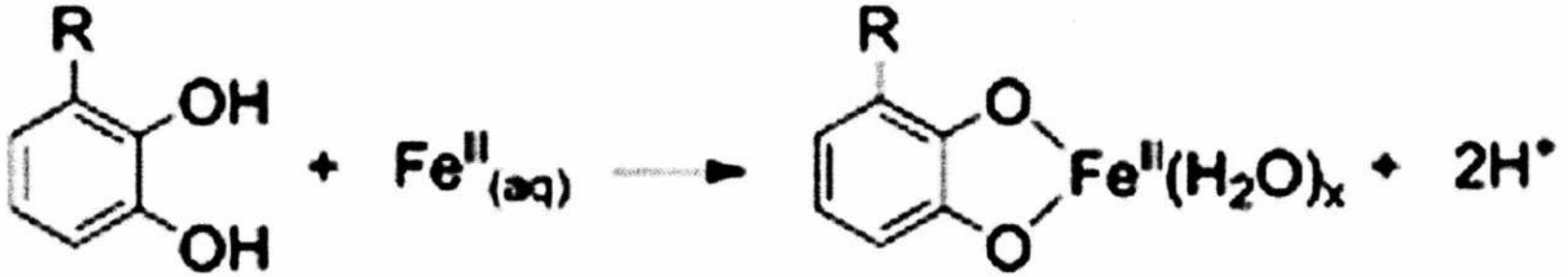
Plant-derived polyphenol, tannic acid, formed by esterification of ten equivalents of the phenylpropanoid-derived gallic acid to a monosaccharide (glucose) core from primary metabolism.

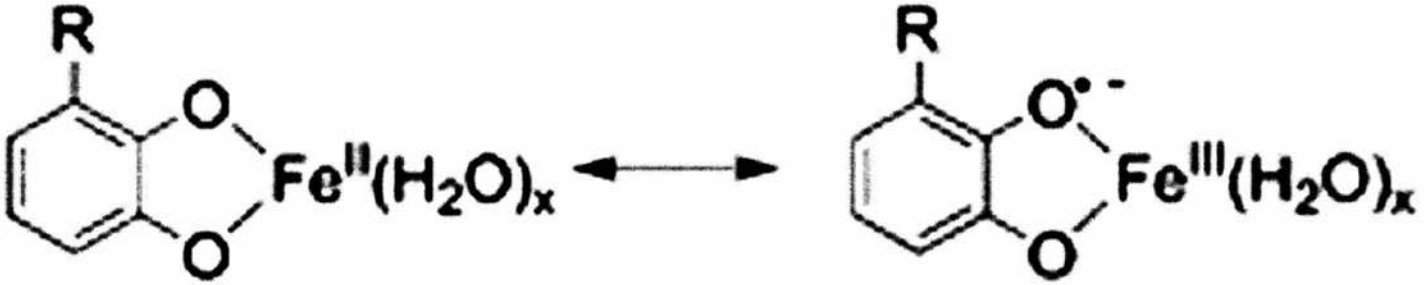


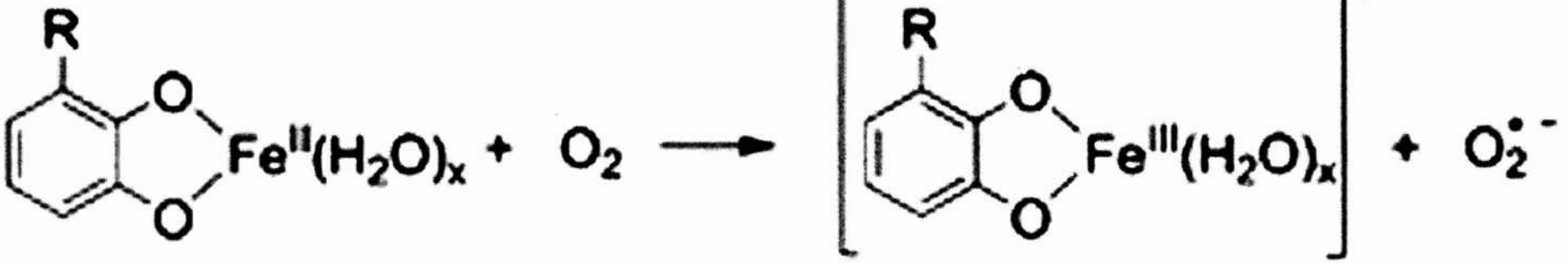
Phenol-phenolate equilibrium, and resonance structures giving rise to phenol aromatic reactivity.

Contents

Untitled - 1

A 

B 

C 

Lets flip to high current
And repeat.
Heat to ~ 200°C
Oven 200°C, 15 min

We are going to investigate CO_2 found.

200°C Holding Breath 10 min, low Current
I see a double peak, notch, else
afterwards. The must be repeated w/
the large balloon.

Ok we have figured it out with the
large balloon & the ~~start~~ tube baby.
soda method. Very good results.
The secondary close peak is CO_2.

@ 200°C, 10 min, low current.

Air	D. 423 mM	~260 mV
O_2	D. 596 mM	~400 mV

The sample was about 10% CO_2. Good work.

Now we will go back to the Carapho phenyl

Notice how radical this is compared to
80°C run.

Phenol Prospective
GC Determination

OK, we are learning to get decent prints now. Camphor Phenique

Air Peak	Ø.40 m	3062 mV
	Ø.66	525
	Ø.94	27.1
	1.16	237.6
Shoulder		~8.
	2.07	68.5
	3.60	19.95
	5.29	.80
	6.66	7.61

We are getting good data. We are comparing
Camphor Phenique
Purchased Phenol
Hair

We conclude that the most likely case for a phenol identification by GC is 6.66 - 6.77 m @ 200°C Hi Current 10M.

Sep 30 2015 Wednesday

[redacted]

3. A new IR spectrum of hair

4. A proof of phenol retention in the GC.

[redacted]

6. Photographs of essential oil trials

We are now examining cocoa powder w/ GC for polyphenols. Cocoa is one of the foodstuffs that is highest in polyphenols.
Running @ 200°C, 15 min, low current.

We clearly have some water in the sample. Water gives a massive broad peak starting @ 8.5 min. We are no longer surprised. This means that we dry the sample just like with infrared.

You could dry it on a watch glass first!

We have established a novel way of learning what the retention time of a compound is.

1. Inject a substance of the suspected compound & record the Chromatogram.

2. Do it for as many similar substances as you can

3. Perform a freq. analysis, ie histogram.

From the method & your sample you are associating the following two retention times of phenols:
t = 1.20 min @ 200°C 150 thermal
t = 2.14 min
t = 6.62 min.

Now, the relative % should give you a likely value or t value of the compound.
For t = 1.20 min

Hair	29.04/1989	=	1.46%
Phenol Purchased	3.256/2662	=	Ø,12%
Campho Phenique	237.6/3062	=	7.8%
Cocoa Powder	10.3/638	=	1.6%

This suggests that the purchased phenol may be much less concentrated than you think.

Bottle of Campho phenique is undoubtedly 5-10% Concentration.

Lets compare with iron test.

The conclusion, of weak perchloric phenol does not bear out. Qualitative chemical testing of camphaphenique vs perchloric phenol shows a strong Fe+3 reaction with the perchloric phenol. The camphaphenique shows no reaction.

So how is this result possible?

Why would the perchloric phenol have such a weak peak @ 1.20 min? Compared to Camphaphenique. This does not make sense!

This really does not make sense value it was not volatilized properly.

The iron test shows us that something is wrong.

The answer might be not Compare to air!

Compare to something else that is common to all
How about 2.14 m? So 1.20/(1.20 + 2.14) ratio is

			1.20/2.14
Hair	29.04/(29.04+7.35) =	79.8%	3.95
Phenol Purchased	3.26/(3.26+1.16) =	73.8%	2.81
Camphe Phenique	237.6/(237.6+68.5) =	77.6%	3.47
Cocoa Powder	1.261/(1.261+.70) =	64.3%	1.80
		$\bar{X}$ = 75.9%	

to close intensity. The ratio is relatively constant.
The ratio are inderectly but I am not sure what
they mean.

Oct 02 2015 Friday

Oct 08 Trip Planning

Desires today:

1. Hair samples have come in

[redacted] sample needs a repeat.

3. Phenol- Continued testing?

4. Other reference, such as purified alcohols?

5. CO_2 checking

There is a question as to whether the volume of the hair sample affects the thiocyanate CO_2 ratio.

t_r				
0.413	Av	Δt (.55-.34)	~~413~~ 550.1	57.8
12.03		(13.55-10.1)	.89	1.20

$\frac{1.20}{1.20+57.8} = 2.03\%$ Could be CO?

Why odor?

Oct 03 2015

1. What are the main gases in car exhaust?

OK 2. How about phenol film in IR?
I think you have SDBS version which is adequate

3. Water vapor in GC?

4. Pure alcohol in GC?

5. How do you know what phenol is in GC?

You have a very good phenol spectrum and your sample material is quite pure.

Gases in car exhaust are primarily nitrogen (air peak) CO_2 & H_2O. Nitrogen oxides are very small.
You say leave for tonight & that car exhaust changes if it sits in the balloon for 24 hrs.
It is interesting that you can smell it.
This indicates that it is not water or CO_2? ...?

You could do car exhaust in it & you would be all set.

Oct 04 2015 Sunday

1. Phenol test repeat w/ Fe+3

[redacted]

3. Water vapor in GC?
What is the second gas? in Cane extract

4. ___

I have succeeded w/ the B glarola hair samples.

The hair hypothesis is solidified. There appear to be a correlation between age & thiocyanate / altered hair toxicity accumulation.

	~2070 Thiocyanate	100 − Thio
13	71.3	28.7
43	44.4	55.6
62	Ø	100

100 − Thio

$y = 1.40(100 - \text{Thio age}) + 6.2 \quad r^2 = 0.93$

Hair from [redacted]

I now have a method of collecting aromatics from hair and placing them into solution but it seem too weak.

I think you will have to do it using only a few drops of acetone.

The first trick is to concentrate the aromatics in only about 1 ml of solution, like it acetone & water.
Method is to

1. Pyrolyze the hair to 150-160°C
2. Place (bubble with long needle syringe) the aromatics into approx 1 ml of solution (acetone)
3. Make a film and let dry.

The hair worked very well. Interesting that the the closest spectral match is that of [redacted] blood serum.

In addition, the hair aromatics placed into solution and subjected to $FeCl_3$ produces a blue precipitate. There is an iron cyanide complex and apparently a Prussian blue.

Cyanide is CN^- "ferrocyanide" $[Fe(CN)_6]^{-4}$ C≡N

Sodium Nitroprusside is a cyanide compound.

Let's examine the water vapor curve on the GC column.

We have put in water @ 200°C.

It looks like we have a double sharp peak

We have a very strong peak showing up @ 4 min.

And it has a very broad tail which matches our expectation.

We now know that the earliest peak @ ~ 11 min IS NOT WATER.

Then a good & very useful experiment.

Since it matches up & the column the confirms our assessment that this is a Polar column. Non polar substances will not react with it.

We have a 40 min period to work with.

The indicates that the compound in the exhaust @ ~ 11 min should be ~~non polar~~ with a high boiling point. or it could be polar since it takes as long.

The broad tailing peak is perfect to show the influence of water in the column.

Advantage of liquid solution is that the air peak is dramatically smaller.

But the fact that it tails is clear, as the peak for well formed indicates that it is non polar w/ a high boiling point.

We are now testing pure xylene, a highly non polar solvent w/ a fairly high boiling point. 200°C low advert

Notice your car exhaust peak does not come out until 31 min in.

Some type of trace compound has come in @ ~ 21.5 min.

Now @ 29 min we have a defined peak coming in.

It is a well shaped peak as we anticipated. Peak height is only about 0.8 mV. I would expect higher than that.

Here come our peak!

Our peak is coming in @ 34 min!

We anticipated this perfectly!

A highly polar compound with a high boiling point.

Now the peak is broader than I would like.

It is going now for a second peak @ 36 min.
Now it is descending @ 37 min

You needed to extend the time to 50 min+.

Magnitude is 30 mV.
Good work.
Repeat a Clemhouse for 60 min.
Then repeat for 60 min.

Practically is giving a very broad peak which is not so good.
That part is very confusing. You may actually need up to 3 x 60 min.

Now 100 mm!

Producing Carbon Monoxide

Now testing isopropanol.
A poor response. Low Magnitude.
Combined peaks
Long tail.

The column does not seem well suited to the alcohol. Even water looks better than the. Alcohols do not look favorable here.

To produce carbon monoxide (and carbon dioxide) and to measure the smoke:

1. Put some chunks of a wood pencil in an 250ml Erlenmeyer flask, (break pieces with pliers)
2. Cover the top w/ several layers aluminum foil & seal top w/ a large neck balloon.
3. Heat up on hotplate until smoking & it fills container. The will not take very long.
4. Withdraw the gas with a syringe & set to 1/2 ml. The balloon acts a septa.
5. Inject into GC. Our column produces a CO_2 & CO peak @ ~ 1.8 min @ 200°C w/ low current. We also have a CO_2 peak @ ~ 0.6 m followed by the O_2-N_2 peak @ 0.44m

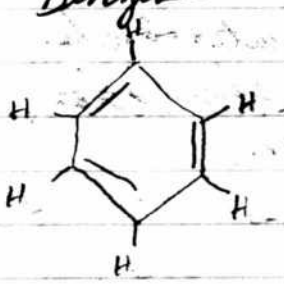
Learn to control your smoke ahead of time!
Here comes our peak!

The worked very well!

Oct 05 2015

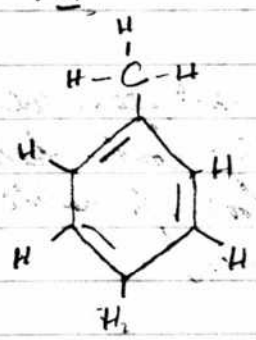
Considering the prospect of homologous series and retention times

Benzene



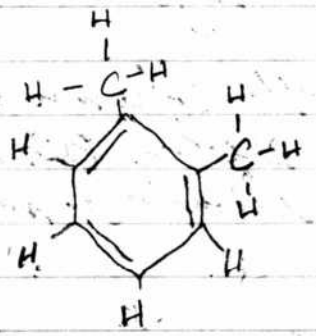
C_6H_6

Toluene



C_7H_8

Xylene



C_8H_{10}

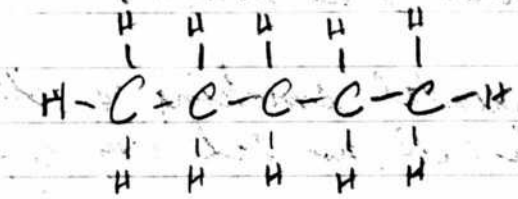
So the series is

$C_{n+5}H_{2(n-1)+6}$

n	F
1	C_6H_6
2	C_7H_8
3	C_8H_{10}

What if $n=0$

C_5H_{11}



This is C_5H_{12}

Let's work w/ Benzene & Xylene

n	C	H
1	6	6
2	7	8
3	8	10
	$=n+5$	$2(n-1)+6$

So does mean that the Obstruction between aliphatic & aromatic structures is crucial.

To use a predictive homologous series you must know if a compound is aliphatic, aromatic, or both.

How do you do this?

Surfaced in certainly a good way!

Propane & butane are a perfect study of alkanes $n=3$ & $n=4$

Charcoal Briquette Study:

We have a very interesting situation going on. We lost no air peak. How is this possible? Thought experiment may have been lost.

The first peak well formed, came in @ 13 min @ 120°C. Then we have a string of very small peaks.

Recommend

200°C high current, 60 min.

Why no air peak?

Another peak @ 38 min.

Running again after bakeout (very significant difference)
@ 200°C High Current / 60 min.

We have a clear air peak again.
We also have the CO_2 peak.

A tertiary peak, very small, right after CO_2.
Could this be methane?

A great sample, not overheated & remarkably different.

6 peaks in 5 minutes
8 in 8 min.
9 in 13 min.

Aromatic Homologous Series Model.

y	x
log(t)	n
~~log(T)~~	
log(7.85) [2.06]	1
log(36.43) [3.595]	3

$y = a \cdot \ln(x) + b$

$\log(t) = .7675 \cdot n + 1.2925$

$t = e^{(.7675n + 1.2925)}$

Good $n=1$ $t = 7.84$ min

good $n=3$ $t = 36.42$

(n) x	(t) min y
1	7.85
predicted. 2	25.9 min
3	36.43

$y = 7.85 + 26.01 \cdot \ln(n)$ OK

How does a phenol fit into this situation.

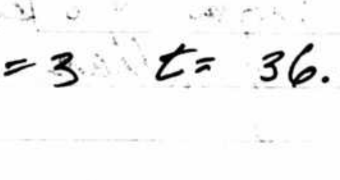
$C_{n+5} H_{2(n-1)+6}$

$n=1$ C_6H_6

$n=2$ C_7H_8

$n=3$ C_8H_{10}

C_5H_4 of $n = \varnothing$



Cannot and does not work
Aliphatic entirely different.

Page 161 Aliphatic Proposal

The briquette experiment to produce some interesting results.

You all now that they are a "match light" version. Does mean that they have a fuel added to them?

It appear to me that they may have butane added.

It is possible that we have

n	Name	t_r
1	Methane	.66
2	Propane	.99
4	Butane	1.21Ø

* $t_r \approx 0.65 + 0.37 \ln(n)$ $r^2 = 0.96$

This is your aliphatic proposal

* $n \approx 3.05 + 4.95 \ln(t_r)$ $r^2 = 1.00$

Oct 06 2015 Tuesday

We are starting to learn the advantages of calibrating a homologous series, eg alkanes, alkenes, aromatics, alcohols, etc. If you set two in a series the model can be developed and intermediates & externals to the model can be estimated.

Eg

alkanes: We may have a great model estimate. We only know propane w/ high certainty. We are questioning if methane overlays w/ CO_2 gas. We also know that something was added to the charcoal briquettes & we suspect that is butane.

the gases: We have quite a few. We believe we have O_2 & N_2, CO_2 & even CO. I would like to consider hydrogen (negative peak?) and also pure oxygen generation.

Aromatics: You have benzene as a known. You also have xylene. The give you a model on aromatics w/ successive substitution of methyl groups.

We would like alcohol or phenol groups.

We would like to verify either butane or methane.

We have water now.

We have an unknown gas @ a 36 min a 200°C from the car. What is it?

We want to study the calculations - GC Chapter in detail

We would like to know how to approach metals. Flame test idea - burn sticks.

I have done some work here.

1. First I recognized that I had some butane in a lighter fuel container & I successfully transferred it into a syringe

2. I deduced that Charcoal briquette purchased had an additive to it, that methane was apparently NOT within, and that the additive was butane (C_4).

Furthermore I proved that it was butane.

We also see that the butane is contaminated w/ a little bit of propane in it.

and parameters I have now developed a model for aliphatic chains

Predicted Assumed	C_1 Methane	1	0.00	CH_4
	C_2 Ethane?	2	0.65	C_2H_6
		n	t_r	
	C_3 Propane	3	0.94	C_3H_8
	C_4 Butane	4	1.213	C_4H_{10}
		x	y	

$t_r = 0.08 + 0.806 \ln(n)$ $r^2 = .994$

$n = 3.32 + 3.17 \ln(t_r)$ $r^2 = .989$

207°C: Air Peak 0.43 min

CO_2 Peak 0.65 min

CO? 11.8 min

Exhaust Gas ? 37.4

I believe we have ethane & CO_2 very close to one another. Best estimate for ethane is 0.65 & CO_2 is 0.62. We will check CO_2.

You need to see if you can determine the difference between CO_2 & Ethane.

Model Predictions: min

C_5	1.38 min	C_9	1.85
C_6	1.52	C_{10}	1.94
C_7	1.65	C_{11}	2.01
C_8	1.76	C_{12}	2.09

Octane.

The work is really important and valuable. It shows you that any well formed plate within the first two minutes are likely to be alkanes / aliphatic alkanes, no less.

You can even estimate which one.

Now, let's do the aromatic model

		methyl groups	t_r
Benzene	C_6H_6	$n=1$	7.85 min
Xylene	C_8H_{10}	$n=3$	36.43 min

$t_r = 7.85 + 26.01 \ln(n)$

$n = -1.68 + 1.30 \ln(t_r)$

Predicted Toluene	C_7H_8	$n=2$	$t_r \approx 25.88$ Predicted

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Next, figure out O_2 + methane (like propane)

Notes:

1. Any plot a file must have operating conditions selected to be useful.
2. Each temperature has its own retention model.
3. Long tails distort the actual retention time.

A. Saturated Aliphatic Model 200°C:

$t_r \cong 0.08 + 0.006 \ln(n)$

$n \cong 3.32 + 3.17 \ln(t_r)$

	methane	propane
$n=1$	CH_4	C_3H_8 $n=3$
$n=2$	C_2H_6	C_4H_{10} $n=4$
	ethane	butane

B. Aromatic methyl Model 200°C

$t_r \cong 7.85 + 26.01 \ln(n)$

$n \cong -1.68 + 1.30 \ln(t_r)$

C_6H_6	benzene $n=1$
C_7H_8	toluene $n=2$
C_8H_{10}	xylene $n=3$

C. Saturated Aliphatic Model 80°C

You can tell now that the trailing peaks require an adjustment. Split the base from straight line extension.

80°C 20 min

	x		y
Butane	n=4	i=1	t_r = 14.5 min
Propane	n=3	i=1	t_r = ~~5.23~~ min 5.5
ethene	n=2	i=1	t_r = 1.7

~~$\hat{t}_r = -11.0 + 16.9 \ln(n)$~~ $\hat{t}_r = .197\, n^{3.08}$

~~$\hat{n} = 1.47 + 0.95 \ln(t_r)$~~ $r^2 = .999$

$\hat{n} = 2$: The result is only modest. ethene

The may not be the best solution. It is covely a pretty broad range in time so the error is going high.

It looks like you need at least 3 pts in a series to have any hope of approximating time.

Tracky peaks also indicate an error.

What we see is that a power model looks to be more better and that tracky peaks must be adjusted.

80°C 20 min

	x		y
Butane	$n=4$	$i=1$	$t_r = 14.5$ min
Propane	$n=3$	$i=1$	$t_r = 5.23$ min 5.5
ethane	$n=2$	$i=1$	$t_r = 1.7$

$\hat{t}_r = -11.0 + 16.9 \ln(n)$ $\hat{t}_r = .197\, n^{3.08}$

$\hat{n} = 1.47 + 0.95 \ln(t_r)$ $r^2 = .999$

$\hat{n} = 2$: The result is only modest.
ethane

The may not be the best solution.
It is covering a pretty broad range of time
so the error is going high.

It looks like you need at least 3 pts
in a series to have any hope
of approximating time.

Tailing peaks also indicate an error.

What we see is that a power model
looks to be more better and that
tailing peaks must be adjusted.

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Aliphatic 80°C & 200°C

Aleylatre model 80°C @ 20min High current

* $t_r \cong 0.197 n^{3.08}$ $r^2 = .999$

Notice this is close to Cn^3

The model looks good for n=1 to 5

Now for 200°C

		t_r
propane	n=3	~~0.93~~ 1.05
ethane	n=2	0.67
butane	n=4	1.4

* $t_r \cong 0.32 n^{1.066}$ $r^2 = 0.999$

almost linear

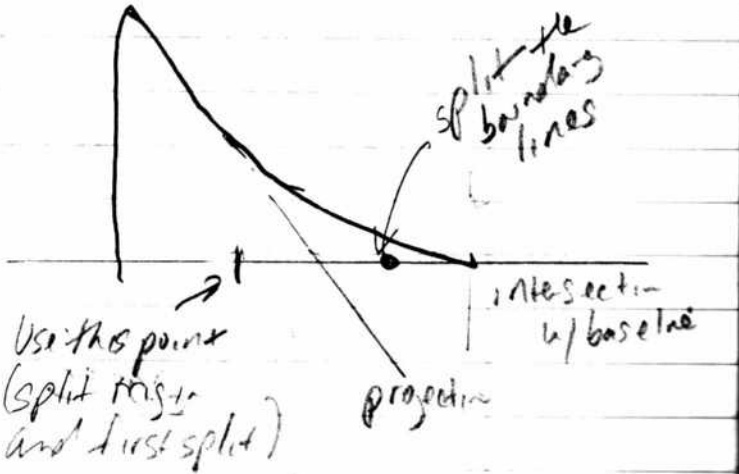
n=1 methane 0.32 min

n=5 pentane 1.78 min

This looks like very good work.

To adjust trailing peaks

Fit points to a power line.



Aromatic 200°C

200°C

Next ya work on the aromatic models

We get

n	tr
1	9.4 min
2	25.6 min (est.)
3	46 min
4	69.7 min (est.)

1.445

$t_r \cong 9.4\,n$

where n = the no. of methyl groups attached to the ring + 1.

benzene
toulene
xylene

Ya also have some gases clarified.

CO_2 80°C $t_r = 2.00$ min
200°C $t_r \cong 0.67$ min (same as ethane)

Ethane 80°C 1.1 min
200°C 0.67 min

$O_2 + N_2$ 80°C ~~0.45~~
200°C 0.43

CO 200°C ~12.0 min

H_2O 200°C 9.2 min

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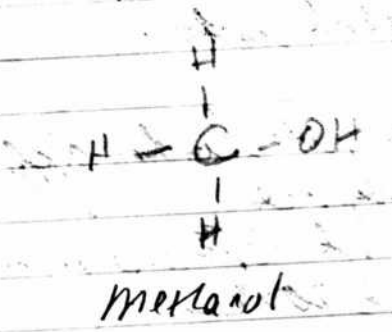
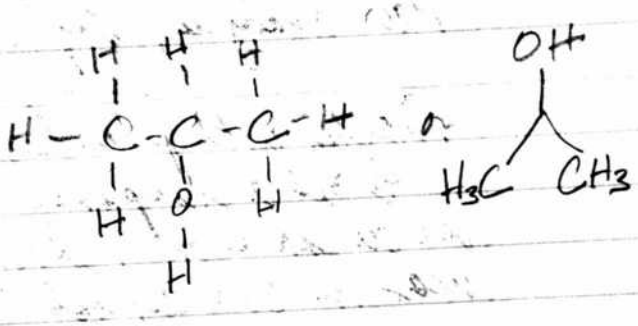
Next we can work with
1. Phenols
2. Ca- Exhaust
3. Alcohols

We have covered
1. Saturated aliphatics 80°C & 200°C
2. Methylated Aromatics & Benzene 200°C
3. Water 200°C
4. Some gases such as O_2, N_2, CO_2, CO

Has a good week.
Can we do alcohols next?
Do alcohols & phenols work on headspace?
It did not seem so.



Methanol



Oct 01 2015 Wednesday

The last field day + the lab before Sunday test.

What would you like to do today?

Phenols are a group.

A Random sample, like soil, would be ~~interesting~~.

Both today?
You have seen what you can do w/ soil already.

How analyses, dep. w/ phenol info?

Starting with phenol analyses, it looks difficult to get a response w/ headspace.
We do have one, however, at $t_r \cong 1.6$ min
What is corresponding temp @ 200°C

200°C model for alkanes

$t_r = 0.32\, n^{1.066}$

$n^{1.066} = \frac{0.32}{t_r}$

$1.066 \ln(n) = \frac{0.32}{1.066 t_r}$

$n = e^{\left(\frac{0.32}{1.066 \cdot t_r}\right)}$

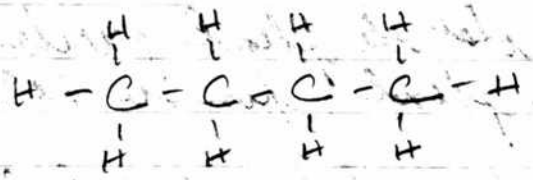
Notice linear + not + to be removed.

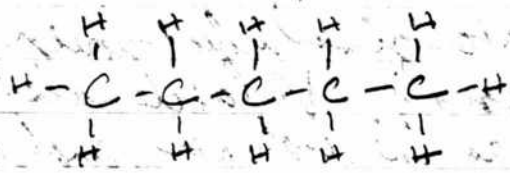
$n = \left(\frac{.32}{t_r}\right)^{\frac{1}{1.066}}$

NO

If $t_r = 1.6$ $n = 4.52$

This suggests that there is a compound somewhere between $n = 4$ & 5

$n = 4$  C_4H_{10}

$n = 5$  C_5H_{12}

Where is interesting because I do not see how it relates to a phenol yet. There is a quarter of volatility here also.

Benzene 120° bent, 3 bents = 360° = a closed circle.

Alcohols? Benzene Combustion?

No, it is 1.49 There is reasonably close to 1.4. This indicates $n = 4$ or butane??

Test for volatility:

Here is a real world problem:

We know that the phenol purchased solution is high in phenol, from the $FeCl_3$ test.

But it fails a volatility test (there is residue on the watch glass which would damage the GC column). Volatile substances should be clear as one indicator, and the solution is not.

Conducting a volatility test, if in any doubt, is indeed imperative.

It also fails the headspace test. Why? Noticed where it is, in the GC applicability chart - not good.

Why do phenols not vaporize?
Why does headspace fail?
Does the sample have to be alkalinized?
It seems like you will not want to be here. GC does not work for everything, even headspace.

Indeed research indicate that phenols are very problematic due to

high polarity
high vapor pressure

How does vapor pressure relate to GC?

Volatile substances have a low vapor pressure.

high vapor pressure normally mean a relatively low boiling point.

In a table, phenols are highly polar.

Higher temperature will increase a compounds vapor pressure.
& the vapor pressure that determines retention time.

Aliphatic model - alkane 200°C $t_R = 0.32n^{1.066}$

n		t_R (min)
1	methane	0.32
2	ethane	0.67
3	propane	1.03
4	butane	1.40
5	pentane	1.78
6	hexane	2.16

Up V have some real quantum about phenol. Headspace can't treat them down so do we expect it to be like a charge stripe.

Up have some peaks produced that are not a part of any model. They seem to be @

1.20 min
2.14
6.62

Just only under heated headspace conditions. Let's repeat:
200°C High Current 20 min.
Twice now, with commercial heated phenol you set a defensive well formed peak @ 1.49m. The does not match anything previously.

Compare all three w/ GC, IR & SDBS.

Something strange happening. We heated it more and we lost our peak, but 1.49 peak but we have a peak @ 2.02 which is what we had before?

Also the baseline is incredibly flat! Simple problem? The baseline dropped 5 mV - why?

200°C 10 min. No drift?
On sufficient heating the there is no air peak.
Why!? How can that be.

Yet you have a very strong peak @ ~2 min.
That suggests that you might be producing hexene?

The peak is incredibly smooth?
Now you are having a run like an alcohol.

The is really strange. No oxygen or nitrogen?
The peak has tailed and require adjustment.

The is fascinating!

Heating phenol sufficiently (a cloud slowly moved up the tube until reached the top with a sealed balloon - a new one)

IS APPARENTLY & POSITIVELY creating a vacuum!
There is no air peak.
The is reproducible.

You get a very clean clean and clear
Consistent peak. The is phenol.
I believe that it is producing hexene.

I also see my peak that time @ 6.5!
Your work is repeating itself.

We have a match!

Confirmation of Phenols

Oct 07 2015

high in phenols
- Phenol 90%
- Campho Phenique
- Cocoa Powder
- [redacted] Hair

All have a match @ 200°C
2.14 min major
6.62 min minor
w/ Headspace Methods.

You have proven the existence of phenols and the thiocyanate components in human hair!

? (Poly) B12 Amino Acids Anti Oxidants & Precursors
- Phenols
- Thiocyanate
- Proline
- Iron Oxidation

the set human health impacts.

- Citrate
- Antioxidants
- B-12
- Proline
- Enzymes

I have therefore proven the existence of a compound within human hair using Gas Chromatography. The above will warrant the cost of the treatment.

OCT 20 2015 [redacted] Conf Close

Conference is ending. We are trying to pin down the thiocyanate complex. That could be different forms of/or different compounds.

In hair
We have
2090
There is a match to a test w/ sodium thiocyanate but only at the 2090 peak so that does not mean that it is NaSCN, might not share 2090

Candidates are
NaCN (sodium cyanide) 2080. Strong.
This actually is our best match.

The blood spectrum shows the peak @ ~ 2120.
Notice the room 6 test match w/
~~KCN~~ KOCN @ 2130.
There is potassium cyanate
not cyanide, not thiocyanate, but cyanate.

We also measured actual sodium thiocyanate solution from our ~~the~~ lab in acetone based fabric & it came out @ 2063 NaSCN

Interesting that Mueller's was @ 2020
So there is actually quite a bit of difference
$\Delta = 43$ and it considered to be a lot.

Oct 21 2015

Back to Spokane. Lots done
Pavia in tablet class have a very
valuable section.

Essentially the molecular mass of any liquid
or gas should be able to be determined
if it is a solid, and you can get it into
a liquid form, you should also be able
to do this. There is much of what you learned
during this trip for sample prep.

In addition, there should be pre-fabricated
glassware available for CHN determination
by combustion.

Gas chromatography should also have
some capability here, at least in terms
of a ratio.

A really good thing to try this work is sugars.
It is straight forward & it can also be
dissolved into or a liquid form.

To get enough material w/ CDB may be tricky
but it should be possible

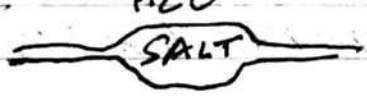
A question — at what point is bonding
actually occurring? First bubble or
linearly?

[redacted]

*

Carbon
Weigh
Initial
& end.

Plastic tubing? Not as good.
You need it hot.



small enough to weigh.

binding agent?

So many ways & approaches now.

1. Solubility
2. GC *
3. IR - Mid & Partial NIR *
4. CHN Analyzer *
5. Molecular Mass Analyzer
6. Boiling Point analyzer
7. Refractive index
8. Numerous qualitative tests
9. Crippen GC-qualitative combination.
10. Voltammetry *

Voltammetry

Using E only:
Start recording new reference value.

1. Limits are set @ ± 1.5V
2. App E is set @ +1.3V
3. dE/dt rate is set @ 0.10 V/s
4. dE/dt rate is 100 mV/s
5. Gain is 0.01
6. Output is I out
7. I polarity is +
8. E polarity is +
9. Filter is .001

We learned a very important lesson today!
The electrode must be completely INSULATED
You have done great work.

Page 192

You have a water reference & the gain is 0.02

Now you added ferrous sulfate (& no salt!)

and you had to reduce the gain from .02 to .1

So there is a dramatic difference in the voltage difference as well as the shape.

The water peaks were at ~ 0.4 & −.8

You are now, w/ the same gain >1.2 & <−1.5

This mean Δ = +.8 and −.7

Exactly what you saw before.

Sure enough, there is iron again.

You are running @ 2s/div.

Now let's salt again.

$\left((.337)^2 + (.11)^2\right)^{1/2} \cdot .84 \overset{?}{=} \left(\frac{\Delta \cdot}{\text{Gain}}\right) \cdot .2$

$\frac{\frac{31.5}{.2} = 1.57}{100}$ $\quad 1.57(2) = 3.2$

[redacted]

$\frac{\frac{51.5}{.2} = 2.57}{100}$ $\quad 2.57(2) = 5.15$

$5.15 + 3.2 = 8.35$?

Water

Gain Ø.2

~~+ Ø.~~

+ 62.0 ~~mA~~ mV

− 65.0 ~~mA~~ mV

$\bar{X} = 63.5$ mV

Δ = 31.5

I added 2-3 drops of Ø.5M $CuSO_4$.

It has risen to + 90 mV $\quad \bar{X} = 95$ mV

− 100 mV

Gain = Ø.2

Water:

~~I (max) = 95E-3~~

So it is defiently detecting it.

Δ = 51.5

A few grains of $FeSO_4$ have now been added.

It has risen to

+ 110 mV $\quad \bar{X} = 115$ mV

− 120 mV

They've definitely detected both substances.
But we can't tell which one & how many
components there are.

$E_1 = 1.40V$

$\frac{1.40 * 100}{0.2} = 700$

Gain in mA per Volt = $\frac{\text{App E}}{\text{I output in Volts}}$ $\quad \frac{1.4}{.115} = 12.17$

$10mV = \frac{1 \text{ Volt}}{100} * \text{Gain}$

meas V $\quad 10mV = \frac{1 \text{ Volt}}{\text{Gain}}$ applie E

I do not know how to interpret voltage

meas mV $\quad 20mV = \frac{2V}{\text{Gain}} * 100$

$(0.9 + 1.1)/2 =$

$40mV = \frac{2V}{5}$

meas mV $= \frac{\text{Applied E}}{\text{Gain}} * 100$

Oct 24 2015 Saturday

1. Today we are going to refine some voltammetry work.

Primary Operating Conditions are

1. Applied E +1.40V
   + Lim +1.50V
   - Lim -1.50V
   Scan V/s 1.00
   Scan Rate 1.00
   Output is I
   Gain Ø.2

We have a water sample.
The output looks very clean w

Max = +47.5 mV DC
Min = -50.0

Water $\bar{X}$ = 48.75 mV

Approx 5 min later the values are

Max = 44 mV
Min = 47.5

Water $\bar{X}$ = 45.75

We now add Ø.08 gms $FeSO_4$ to rm level line of water and stir
We now measure

Max +97 mV
Min -98 mV

X = 97.5 mV

after 5 min it has decreased to.
+82 mV $\bar{X} = 83.5$ mV
−84 mV

From initial sets:
$\Delta = 97.5 - 48.75$ mV $= 48.75$ mV) OK

NO Now if gain is 0.2 we anticipate that we have a factor of $\frac{48.75}{0.2} = 243.75$ mV
and what exactly does the mean in the question?

From the latter set we have
$\Delta = 83.5 - 45.75 = 37.75$ mV) OK
and w/ gain of .2 we anticipate
NO $\frac{37.75}{.2} = 188.75$ mV
and there is actually a big difference.

I notice that it continues to decline & is now @ approx 80. Notice also the solution is turning brown instead of green. So it is oxidizing.

Now we add 0.139 mV It rises to
Max = +130 $\bar{X} = 125$
Min = −120

So now we see that it decreases with time as the material is oxidized, most likely by the oxygen in the water.

Let switch to E
reads approx 120mV peak.

We have flipped to E.
It is a somewhat triangular wave with peaks @ approx 150mV.

The gain know knob does not affect the voltage graph @ all.

Now Notice that we have E limit set to 1.50
Very interesting. This is a factor of

$$\frac{1.50V}{150E-3V} = \frac{10}{}$$

Lets set E limit to 1.60V and see. Absolutely. It goes to +0.160mV.

Now lets go to 2.00 mV
Absolutely!

Crucial Discovery: Output is Reduced by a factor of 10.

Here is the crucial.
When we measure the actual voltage from what is supposedly applied it is reduced by a factor of 10 exactly !!

This means that your readings apparently are to be multiplied by a factor of 10.

Therefore for us for example, we

$$\Delta = 37.75(10) = 377.5\,mV = 0.38V$$

If this is half potential, then we have

$$0.38(2) = 0.76V$$

And guess what:
$Fe^{3+} + 1e^- \rightarrow Fe^{+2}$ has a reduction potential of 0.771 Volts!!!

Sound realistic? Notice this is after solution stabilizes for a few minutes.

Let my Copper. Reset Element to 1.5V

Since the reference solution is the same gain as the sample solution it cancels out in the calculations

Let the copper. Maybe a different sandpaper.

It looks like a table gate for 5 min is important.

Now for $CuSO_4$

Water

Initial readings: +37, −43 $\bar{x} = 40$ mV

after 5 min: +36, −42 $\bar{x} = 39$ mV

Now add a few drops of $CuSO_4$

Initial reading: +48, −62 $\bar{x} = 55$

after 5 min: 52, 62 $\bar{x} = 57$

$\Delta = 57mV - 39mV = 18.mV\ (10) = 180mV$

$= .18V\ (2) = 0.36V$
(half E^o)

Actual: $Cu^{2+} + 2e^- \rightarrow Cu \quad = +0.34$

It looks to me like we have it. Ok to here

The are fantastic.

So now what if Fe + Cu are mixed together?

The copper cell is still stable. $(53+62)/2 =$

Add Iron:

First of all you have a double peak. The telling on that we have at least 2 components.

Max is @ $+66$ $\quad \bar{X} = 75.5$
-85

~~75.5 (10 factor) =~~

Δ $75.5 - 39\ mV = 36.5\ mV$

$\Delta_{10} = 36.5\ mV\ (10\ factor) = 365\ mV = .365\ V$

Half Voltage: $.365\ (2) = 0.\underline{73V}$ Combined Copper & Iron.

How would you know?
How to interpret this?

It certainly is closest to iron. Assume this is a max.

So we could declare that we have iron and then ya would need to precipitate it out.

So what if you did not apply enough voltage to oxidize the iron but enough to oxidize the copper ya use a max of Ø.5V.

What happens then.

Now since we really only need ½ voltage this means Ø.25V

We have at Elim @ ± Ø.5V

The means we are actually outputty Ø.05V = 50mV max.

Our copper signal is coming in @ $\begin{matrix} +34 \\ -44 \end{matrix} \cong 39$

But we can see that is the same as water.

We have to increase it to about 60.

We seem to have copper alone with limits of
$E_{lim} = \pm 1.10V$

This gives us ~~+48~~ +50 $\bar{X} = 60$
−70

Still may be a little high.

Now we have $E_{lim} = \pm 1.00$
with readings of +46 $= \bar{X} = 55.5$
−65

Notice the offsets in Iron + Copper are different.

In Copper, the reference line is at $(46-65)/2 = -9.5$ mV

but in Iron, the reference line is @ $(130-120)/2 = +5$

There may be another way of distinguishing the species.

Our mean now is 55.5
$\Delta = 55.5 - 39\text{ mV} = 16.5\text{ mV}$
16.5 mV (10 facta) = 165 mV = 0.165V
Half cell factor: 0.165 (2) = 0.33 Volts

And actual value is
$Cu^{2+} + 2e^- \rightarrow Cu = 0.34V$

I believe that we have isolated the Cu species.

You now see a method to isolate species within a mixture.

1. Develop reference electropectra.

2. Notice & record the reference line for each species. The reference line looks to be very important.

3. Variation in peaks mean different species.

4. The actual voltage out in E_{out} is actually 1/10 exactly of the applied voltage (many lemeta a directly ($\frac{1}{2}$)) Do not ask me why but it is.

5. The Δ determined is from the reference line, but the reference line itself is an important attribute.

6. Elim of ± 1.0 V identified copper and Elim of ± 1.5 V identified iron. I am not sure why yet.

The method of measurement:

1. Determine the reference line & $E'_{1/2}$ for a reference solution.
   Set App E close to E limit
2. Add the sample w/ limits imposed and observe the behavior and number of peaks.
3. Multiple peaks will require further separation by E range if required.
4. Determine $E'_{1/2}$ and the reference line for the species under examination.
5. Determine ΔE by:
   a) take mean $E'_{1/2}$ difference between sample & reference. Note the reference line value.
   b) multiply ΔE by 10 to get actual voltage in mV
   c) Convert to Volts
   d) Multiply $E'_{1/2}$ by 2 to get full redox potential
6. Compare to tables.

Starting to think now about the Assays of metals.

Al, Ba, Sr, Mg, Ca, Cu, Fe should all be in range.

Even ~~Ba~~, K, Li, Na should also be in range now.

$Al^{3+} + 3e^- \rightarrow Al \quad -1.66$

$\overset{+3}{Al_2}\overset{-2}{(SO_4)_3} \rightarrow$

$SO_4 + 2e^- \rightarrow SO_4^{2-} \quad +1.90$

You only need to change E_{lim} to get it running.

We have set E_{lim} to ± 2.50 V

Gain = 0.2 Water Sample:

$+114 \quad \bar{X} = 113$

-112

Notice zero line is essentially zero.

$E'_{1/2}$ ~~494~~ 480 $E'_{1/2}\bar{X} = 454.5$

$-430 \quad -429 \quad \bar{X} = 25.5$

Therefore
$\Delta E = 454.5 - 113 = 341.5\ mV$

10 Factor: $341.5\ mV = 3415\ mV = 3.415\ V$
and the does not work at all.

Get E output.

Eout has peaks of 250mV so this is right on.

Lets set back E_{lim} to 1.5V

+129 $\quad \bar{X} = 131.5$
−134

$\Delta = 131.5$ − Assume Water is still @ 45.75 $= 85.75\ mV$
10 Factor $= 85.75(10) = 857.5\ mV = .86\ V$
$E_{1/2}$ factor $= .86(2) = 1.72\ V$ for Al

Actual:
$Al^{3+} + 3e^- \rightarrow Al \quad -1.66\ V$
This is not bad.

Now, we have a strange circumstance here.

Why did it not work when we set $E_{lim} \pm 2.50V$ instead of $E_{lim} \pm 1.50V$?

Why would this matter?

What is the 10 factor about?
Why should it depend upon E lim?

	Water 5min	Al_2SO_4 3 5mn
Elim ±1.5	45.75	131.5
Elim ±2.5	113	454.5
Ratio	2.47	3.46

$$\text{Gain} = \frac{.2\ mA}{\text{Volt}}$$

1 Volt is 1.5, G

$$\frac{.2\ mA}{1} = \frac{X}{1.5} \qquad X = \frac{0.3\ mA}{V}$$

1 Volt is 2.5

$$\frac{.2\ mA}{1} = \frac{X}{2.5} \qquad X = \frac{0.5\ mA}{V}$$

Let's try it again...

Water: Clean signal.
Start out @ +60, −65 $\bar{X} = 62.5$

5 min: Max +54 $E^1/_2 = 51mV$
Min −60 $\bar{X} = -3$

Now add $Al_2(SO_4)_3$:
Max ~~+160~~
Min ~~+100~~

5 min Max +135 $E^1/_2 = 131.5$
Min −128 $\bar{X} = +3.5$

Δ: 131.5 − 51 = 74.5
10 factor: 74.5 (10) = 745 mV = .745 mV
$E^1/_2$ factor .745 (2) = 1.49 Not good.

Then is not OK.
Work @ set @ 1.4 will have
Max = 142 141 $E^1/_2 = 138.5$
−136

Δ 138.5 − 51 = 81.5
81.5 (10) = 815 mV = .815 V
.815 V (2) = 1.63 and this is good. vs 1.66

So raising App E to 1.4V did make a difference!

Ok, We still have some mysteries
last we did get A/+3.
Lets do it again. All original conditions
must be satisfied.

App E 1.40V
+/Lim 1.50V
Scan V/s 1.00
Scan Rate 1.00
Gain 10.2
Output is I

Water 5min: +62 $\Sigma' = 65$
-68 $\bar{X} = -3$

Al_2SO_4 ~~146~~ 147 149 $\Sigma' = 144.5$
~~137~~ 141 -140 $\bar{X} = +4.5$

$\Delta = (144.5 - 65) = 79.5$
~~79.5~~ (10) = 795 = .795V
.795V(2) = 1.59V

vs 1.66 Actual Not bad again.
Concentration is reasonably low.
There is nothing else close & you
have two points.
1.59 $\bar{X} = 1.61$ vs 1.66
1.63
This is quite good.

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You have succeeded w/ ionic Fe, Cu & Al.
There is a major accomplishment.

Who is next? $Ba(OH)_2$

Water 5min 55.5 $E'_{1/2} = 58.2$
-61 $\bar{X} = -2.75$

$Ba(OH)_2$ does not appear to be working even under extreme limits.

+249 $E'_{1/2} = 241.5$
-234 $\bar{X} = +7.5$

$\Delta E = 241.5 - 58.2 = 183.3$
$183.3\ (10) = 1833\ mV = 1.833\ V$
$1.833\ V\ (2) = 3.67\ V$

Actual is 2.90V

So this did not seem to work.
You had it set on 4.5V - very high
I see no basis for success.
It is not very soluble. Is it soluble in acid?

Yes it is! In fairly weak HCl
it is soluble. This is therefore
your next test.

On to Magnesium.
Using Epsom Salt.

Water 5 min 58
-63 $\varepsilon^1/_2 = 60.5$

Let's also get time on water peaks.

12.7
8.86 6.74
-2.8 = 6.0 sec

+120 $\varepsilon^1/_2 = 125.5$
-131

125.5 - 60.5 = 65 mV
65 mV (10) = 650 mV = .65 V
.65 V (2) = 1.30 V

Mg = 2.37

+100 $\varepsilon^1/_2 = 173.5$
-167

173.5 - 60.5 = 113
113 (10) = 1130 mV = 1.130 V
1.13 V (2) = 2.26 V OK VS 2.37

The concentration had to be higher.

The concentration is changing it.

~~+218~~ 213 $\Sigma^{1/2} = 201.5$
~~-190~~ -190

201.5 − 60.5 = 141 → 1410 = 1.410V
1.410V(2) = 2.82 vs 2.37

It is too high now. What gives here?

If concentration affects this it causes a problem.

210 → 202.5
-195
202.5 − 60.5 = 142 → (10) 1420 = 1.42V
1.42V(2) = 2.84V

Should be 2.4 What gives here? Overload?

Maybe more time? Because you loaded it up so much? Then a closer to Calibration.

Magnesium Agar:

Water 5 min: Max +77 Min −86 $E_{1/2} = 81.5$ $\bar{X} = -4.5$

$MgSO_4$ Max 160 Min −176 $E_{1/2} = +168$ $\bar{X} = -8$

$160 - 81.5 = 79.5$

$79.5 (10) = 795\ mV = .795\ V$

½ cell $.795 (2) = 1.59$

Should be 2.37

This is no good.

Added Mn: Solution

Mg 211 Min −198 $E_{1/2} = 204$

$204.5 - 81.5 = 123$

$123 (10) = 1230\ mV = 1.23\ V$

$2 (1.23) = 2.46\ V$ vs 2.37

And the cell works. But why does it vary by concentration?

* How would you know that it varies & how to vary it?

Notice it has nice sharp peaks.

If you change the gain it simply changes the y-scaling by that same factor eg if you went from G2 to G1 then
80 → 160
200 → 400.

The

Another run
~~199-81.5~~ 199-81.5 = 117.5
117.5(10) = 1175 mV = 1.175 V
1.175(2) = 2.35 vs 2.37 and this actually is phenomenal.

Lets try to do tot peak.

When I add more it still looks good.
Is there a time factor?
I do not know how much to add.

Lets start looking @ the curve more closely.

Water is very smooth.

Water 5 min +80 $E^{1/2} = 83.5$
-87

~~155~~ 213 $X = 208.5$
~~-114~~ 204

$208.5 - 83.5 = 125$
$125(10) = 1250 mV = 1.25 V$
$1.25(2) = 2.50 V$ vs 2.37

Notice that the peaks are asymmetric the more concentrated it is

There seems to be an issue of asymmetry that is involved. It seems like we are seeing a measurement symmetric a to a a maximum as the measurement slope change.

Libs look @ sodium.

Water is as smooth as can be.

Notice the
Water 5 min. 82 $\bar{X} = 84.5$
-87

Still symmetrical @ +132 strong offset.
-153

Higher Concentration +213 $X = 227$
Asymmetry starts. -241

227 - 84.5 = 142.5 → (10) 1425 mV = 1.425
(2) = 2.85 V vs 2.71 using Elim 1.50 V

Not terrible.

So there is an interesting lesson here.
The concentration needs to be high enough to maximize the volume of asymmetry.

Notice the current under I output is uA, not mA!

If you drop the voltge a little, it gets rid of the sharp peak & increases symmetry
+193 $X = 210.5$ ~~→ 2105 mV = 2.105~~
-228
210.5 - 84.5 = 126 → 126(10) = 1260 mV = 1.26 V
1.26 V (2) = 2.52 vs 2.71
using Elim 1.40 V

It may be that you just want to start creating the sharp peak.
Now @ $E_{lim} \pm 1.45V$.

+203 $\bar{x} = 220.5$
-238

220.5 - 84.5 = 136
136(10) = 1360 mV = 1.36 V
2(1.36) = 2.72 V
vs 2.71 Olla!

It look like I have found the trick. It appear to set E_{lim} as it just falls in to peak out.

You might be able to use different concentrations w/ the method.

We now have metals in an ionizable form.
Iron
Copper
Magnesium
Sodium
Aluminum
Potassium
Great Work

Barium requires an acid solution.

The a looky very good.. Now we know we are looking to peak out.

Now for Potassium.

Water. Reliable as can be.
Gain = 82 +82 $\bar{X}$ = 85
Elim = ±1.5 -88

You have adjusted Elim to +1.80 V to introduce peak out.

+170 +161 $\bar{X}$ = 174.5
-182

174.5 - 85 = 89.5
89.5(10) = 895 mV = .895 V
.895 V (2) = 1.79
The a way off. Must not have enough concentration.

$\frac{.02gm}{125ml} = \frac{X}{1000ml}$ X = 0.16 gms

KCl : MW = 74 gms/mole .16/74 = .002 Molar
or 0.1% solution
so it is indeed very weak.
You are allowed to concentrate it more

We have increased concentration to about 0.06 gms.

Elim ± 1.7 V looks good.
+198 X = 204.5
−211

204.5 − 85 = 119.5
119.5 (10) = 1195 mV = 1.195
1.195 (2) = 2.39 V

It is 2.92 still short.
We have now added about 0.1 gms.
We have a peak.
Elim ± 1.40 V +200 X = 219.5
−239

219.5 − 85 = 134.5
134.5 (10) = 1345 mV = 1.345 V
1.345 (2) = 2.69 vs 2.92

Close but could be a little better

Elim = ± 1.50 V
It looks like you want a little peak showing up about 3/4 of curve length.
Drop to 1.45 Elim
+221 X = 236
−251

236 − 85 = 151
10 (151) = 1510 mV = 1.510 V
1.510 (2) = 3.02 vs 2.92

Then means that we now know that it is likely either potassium or lithium.

2.69] $\bar{x}$ = 2.86 vs 2.92
3.02]

We know that it is likely lithium, potassium barium or calcium.

These top 4 metals are so close that it would be hard to tell them apart.

[redacted]

$\frac{0.1 \text{ gms}}{125 \text{ ml}} \cdot \frac{x}{1000}$ $x = 0.80$ gms

$\frac{0.80 \text{ gms}}{74 \text{ gms}}$ = ~~1 M Solution~~ or = .01 Molar Solution

0.1

or ~~0.80~~ gms = ~~0.64%~~ solution
125 ml

or ~~close to 0.5%~~ solution

or 1 part in ~~200~~ 250

or about 1 part in 1000.

or about 1000 ppm.

It would be more if we could increase the a little bit.

Oct 25 2015

1. Combustion Chambers available?
2. Drainage water test
3. Other metals
4. Continuous bar analysis
5. GC Reset oven
6. Determine the CH ratio of fructose!

stainless steel container measures
11.06 gms

add 5.31 gms fructose
Σ = 16.43 gms

16.41 gms after approx 40 min
16.33 gms after 90 min = 0.6% moisture
16.33 gms after 150 min looks like we have it.
16.28 gms after 180 min 240 = 0.15 gms = 0.91%
16.16 = 0.27 = 1.64%

Estimate given .039 gms per 100 gms.
Not matching very well.
15.43

Drainage Water Test:

Water reference 5 mm.
Elim = ± 1.50V
Gain = 0.2
Scan V/S 1.00
Scan Rate 1.00
I output

@ a gain of 0.2
Voltage = 1/10 of App E

Water Max 65.5 mV $\bar{X}$ = 70.00 mV reference.
Min −74.5 mV

New Drainage Water: Max ~~+168~~ 165 $\bar{X}$ = 169.5
Min −176 −174

Peak induced @ Elim ± 1.70V

Therefore (169.5 − 70.0) = 99.5
(99.5) 10 factor = 995 mV = .995V
dc .995V (2) = 1.99V ~~estimated~~ redox potential.

The ~~p~~ somewhere between magnesium and aluminum. That is certainly interesting. It did come from a lot of leaves in the drainage ditch.

Mag = −2.37V
Al = −1.68V

This says it is magnesium.

Revise to ~~1.95V~~ Elim ± 1.90V
Max = 175 $\bar{X}$ = 177.5 ~~=~~
10

Closest to Magnesium

177.5 − 10 = 167.5 (10) = 1675 mV = 1.675V (2) = 2.15V

Since I changed Elim to ±2.00V
from 1.50 V
let remeasure difference.

Well indeed it is a lot higher.
so your results are longer.

Waterway @ Elim ±2.00V

Elim ±2.0V (2) 108 $\bar{x}$ = 113 Max +106 $\bar{X}$ = 110.5 (1)
+118 Min −115

And we had Drainage rate @ $\bar{X}$ = 169.5

169.5 − 110.5 = 59
59(10) = 590 mV = 0.59V
2(0.59V) = 1.18V

And this corresponds to manganese.

This is very interesting. Ideas entirely different.

Elim ±2.0V (2) 151 $\bar{x}$ = 154.5
158

Δ = 154.5 − 113 = 41.5 ⇒ 415mV = .415V
.415(2) = 0.83V

And this is totally different.
So your work is not reproduced.

Let's repeat our work @ two different voltages. The only work calculated thus far is @ Elim ± 1.50V so let's hold there for now.

You also need to wash the electrodes!

Water Elim ± 1.5V

Max +86 $\bar{X}$ 89
Min −92

Drainage: 111 $\bar{X}$ = 116.5
−122

Δ = 116.5 − 89 = 27.5 27.5 = 275 = .275V
.275(V)(2) = 0.55V

Twice now

Again ether is closest to manganese = 0.5B

Let's do the entire test again @ Elim ± 2.0V

Elim ± ~~1.5~~ 2.0V

~~Drainage~~ ~~water~~ Max +109 $\bar{X}$ = 113.5 Water/groundwater
−118

~~113.5~~

Drainage/water 146 $\bar{X}$ = 149.5
−153

149.5 − 113.5 = 36 → 360 = 0.36V
.36V(2) = 0.72V Closest to chromium & iron but these values are not calibrated

You seem to have some problems with your work.

Two tests do reproduce themselves

You stated that this work for 5 metals a was calibrated is to use

Water @ Elim ± 1.50V
then to peak out solution w/ modified Elim

One test result which duplicate the process here is

Elim ± 1.50 Water $\bar{X} = 70$

Drainage sample Peak ordered @ ± 1.90V
with a $\bar{X}$ of 177.5

The solve to
$177.5 - 70 = 107.5\,(10) = 1075 = 1.075$
$1.075V\,(2) = 2.15V$
which is closest to magnesium.

Let's repeat.

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Then is our last work w/ the drainage water.

Ref water @ 1.5V

Max 89. $\bar{X} = 92$
Min -95

Drainage: @ ±1.5V Question: Is there a peak?
NO.

5. increase voltage until you see a peak.
± 1.6V very small.
± 1.7V NO.
± 1.8V NO
± 1.9V Yes, small peak.
± 1.95V Yes, small peak at now.

Drainage +150 $\bar{X} = 154$
-158

Δ 154 - 92 = 62 $\Rightarrow$ 62(10) = 620mV = .62V
@ 62V(2) = 1.24V

Also closest to Manganese. 1.24 VS 1.18
on 1.24 VS 1.21

The hypothesis is that there is manganese in the drainage water.

We have 1.18 with $\bar{X} = 1.21$ V
1.24

The matches quite closely as you see.

So now the question
What is a qualitative test
for manganese in water?

We all now that we have a real problem.
Voltametry determine twice, by what we
believe is an acceptable & calibrated
method, that manganese appear to be
in the drainage water.

But! We cannot qualitatively prove or
verify it!
The qualitative test fails and
we have no salt to work with.

NaOH fails
Ammonia? - Guess What!
Ammonia does indeed give
a very weak but visible reaction!

While precipitate is formed.

SUCCESS!!!

You have your first successful trace
determination of a metal ion in
an environmental sample.

Indeed a precipitate is formed in both NaOH & Ammonia Hydroxide additions to the water sample.

Indeed the color is "off white" per Doc Brown. It should get successfully darker upon further oxidation from Mn^{+2} to Mn^{+3} to Mn^{+4}.

The laboratory is all set out. The precipitate is extremely weak & requires close magnification. The reaction also takes time. Both are successful but the ammonia may be more readily visible.

You never would have noticed it except for magnification & time.

Voltammetry determined its existence!

Hurrah! Hurrah! Hurrah!

TDS is 92
pH is on alkaline side ~ 8.5
The was great work.

Manganese culture started.
Apparently closely related w/ iron bacteria.

Next we are testing distilled water vs drinking water. There is a huge difference!!

Distilled! ~~Drinking~~ water has a very symmetrical profile, low magnitude, perfectly bidynatic waveform.

Distilled Water:
$E_{1/2} = \pm 1.5V$ $+6$ $\bar{X} = 6mV$
-6
incredibly uniform

Drinking water we have B2
-86
but let's peak it out!

$E_{1/2}$ ± 2.00 peak is just starting to show.

Ok, peak comes in by 3.00V
Peak appears to be in @ $\pm 2.8V$ $E_{1/2}$
Therefore:
$+147$ $\bar{X} = 145.5$
-144

Δ $145.5 - 6 = 139.5 \rightarrow 1395mV = 1.395V$
$1.395(2) = 2.79V$

Indicates Calcium 2.79 vs 2.84

seems reasonable.

Now, how to test for Calcium

Oct 26 2015

1. CO_2 in sugar headspace?

2 Why can't you force the sugar?
& then weigh what is left?

3. [redacted] hair

OK! 4. E+I on two channels for voltammetry

5. New baseline & calibrate w/ distilled water.

6. Only positive voltage means a linear sweep

7. Crushed mixture

8. Four different env. samples.
IR analyzer also in highly polluted waters?

[redacted]

Very good work $r^2 = 0.95$ with $n=4$

Concentration
Determination

Distilled water Calibration:

Gain 0.2 +5.1 4.1 Creating line
Elim ± 1.5V -3.8 -4.1 $\bar{x} = 4.3$ mV

Gain 0.1 +8.6 +8.4 $\bar{x} = 9.0$
-9.7

So the gain is a direct multiplication factor

Gain = 0.05 +17.4 $\bar{x} = 18.85$
-20.3

$\frac{.2}{.05} = 4$ $4(4.3) = 17.2$ vs 18.9

so gain is a straight forward factor that can be changed @ any time.

Now, for Calibration, you should add to distilled water. Then add my sulfate. & Calibrate it.

1st Concentration: 0.05 gms / 200 ml 200ml
Gain = 0.2 Elim ± 1.5V Max = +216 $\bar{x} = 229.5$ mV
-243

Now dilute in 1/2 or .05 / 400 ml 400ml
.025 gms / 200 ml
max = 166 $\bar{x} = 166$ mV
min -172

Now dilute by 200 ml again .05 gms / 600 ml 600ml
Max = 131 $\bar{x} = 140$ mV
Min -144

Now by 200 ml again .05 gms / 800 ml 800ml
Max +110 $\bar{x} = 110$ mV
Min -110

Now by 200 ml again: .05 gms / 1000 ml 1000 ml

Max +109 101 $\bar{X}$ = 101

Min − 101

This means the previous set was my limit of detection as it will not go any lower.

What if you diluted it radically. By 1000 ml or so?

3500 ml total Max 58 $\bar{X}$ = 58

Min 58

n = 5 This is not linear It is a power rule: $r^2 = .995$

So Concentrations are:

Molecular mass of $MgSO_4$ is: 120.37 gms mol

So

$$\frac{.05 \text{ gms}}{200 \text{ ml}} = \frac{X}{1000 \text{ ml}} \qquad X = \frac{0.25 \text{ gms}}{1000 \text{ ml}}$$

and $\frac{0.25 \text{ gms}}{120.37 \text{ gms/mol}}$ = .002071 M for 200 ml

.001038 M 400

.000692 M 600

.000519 M 800

.000119 M 3500

So our Calibration data is

Y Moles	NOTE Reversal! X mV
@2.077E-3M	229.5
1.038E-3M	166
6.92E-4M	140
5.19E-4M	110
1.19E-4M	58

$\frac{\text{Moles}}{\text{liter}} = 3.0144E\text{-}8 \cdot mV^{2.048}$ $r^2 = 0.995$

Now if we have moles per liter
to get grams per liter you multiply by 120.37
so.

$\frac{\text{gms}}{\text{liter}} = 3.628E\text{-}6 \cdot mV^{2.048}$

gms/liter is PPThousands.
to get PPM we multiply by 1000
So

PPM $\frac{\text{milligrams}}{\text{liter}} = 3.628E\text{-}3 \cdot mV^{2.048}$

MgSO4 Calibration Curve Great Work.

PPT And to get PPT parts per trillion we have

PPT $\frac{\text{micrograms}}{\text{liter}} = 3.628 \cdot mV^{2.048}$

A useable range is PPM
eg $mV = 58$; $PPM = 15$ PPM Superb Work.

Now the big question is, Can we still determine if/if not is magnesium @ this extremely low concentration?

We need to see what happens when we try to peak it out.

Our reference is distilled water @ 4.3mV

We are now @ approx 58 mV @ Gain = 0.2 and Elim = ± 1.5

Go + 2.0V
This only brought it to 80.
No peak.

Now 2.5 V	~ 90mV	No peak
3.0V	120mV	No Peak
4.0V	150mV	no Peak.
5.0V	170mV	no Peak.

So negative, you can not identify the metal @ the 15 PPM level. You would need it more concentrated.

Try it for kicks. 170mV - 4 = 166mV → 1660mV = 1.66V and 1.66(2) = 3.32V
Magnesium is only 2.37
so it never peaked @ this low concentration.

to identify the metal we need a higher concentration. How high.

Lets go to 0.1 gm in 1000 ml.

But before we do that, lets look @ filtered water for a reference material.

Reset Elim to ±1.5V Gain = 02

Filtered Water measure @ Max 60
Min -60

So it is only slightly better than tap water

You also see that your lower threshold of measurement was about 60 mV so there really is not much of an incentive to use distilled water unless you are really very very low concentrations, eg ppm range.
Therefore tap water will work ok.
Notice you do have a very nice waveform w/ filtered water, however, so there might be some improvement.

Ref filtered water:

+65 $\bar{X}=65$

-65

Peak in corner @ 2.0

A little better @ 2.2

Look best @ 2.40

Max 211 $\bar{X}=211$

Min -211

211 - 65 = 146 → 1460 → 1.460V

1.46V (2) = 2.92V.

Actual is 2.4V

so you are high

Clean water may be making a difference.

@ 2.1 clean & no distortion

(max reached before distortion

+179 $\bar{X}=182$

-185

182 - 65 = 117 → 1170 → 1.170V

1.170V (2) = 2.34V vs 2.37

So when the water is clean it looks like your goal is to. ~~see~~ reach maximum symmetry & magnitude of the peaks. If you introduce a peak (i.e., asymmetry) it'll look like you have gone to far.

The says to me that we probably have one part in 10,000 or so to actually identify the metal.

We have now completed our first metal analysis with flying colors.

Magnesium.

Detection @ 15 ppm range

Identification @ 1 in 10,000 range

Tap water ID:

We have peak starting @ 1.65V Elim: Gain 0.2
Max 198 X=208
Min 216

(Filtered)

208 − 65 = 143 ⇒ 1430 ⇒ 1.430V

1.43V(2) = 2.86V

Sure enough, that is Calcium
2.86 vs Actual 2.8 Great work
Results have been repeated w/ an arbitrary sample.

Final voltage @ 1.65V. Reset to @ 1.5V

We could now calculate a solution to get the Concentration.

Need 17x150 test tubes

Oct 28 2015 CHO analysis

~~2.00 gms fructose~~

measure salt & tube

2.07 Salt 24.65 w/ Salt ~~Cap~~ & tube
No Caps & Cap only.

Note this. Tube alone is 22.58

measure KOH & tube & cap

KOH 2.03 gms 25.35 No KOH added
end cap & tube

Check w/ KOH ~~27.40~~ ~~27.42~~ 27.36

so 25.37
+ 2.03
27.40 vs 27.36 Ok

22.35 Sample Tube No Caps ~~theoretical~~ is
2.02 Fructose 0 2

Check 24.37 Total : 22.35
measured + 2.02
24.37 Match
measure measure measured

Sample tube after experiment 22.72

Theoretical is 24.65
- 2.07
22.58 Not bad @ all

The means you burned up all but .04
22.72
- 25.58
0.14 gms - not bad.

Salt tube after weighing 25.23
Started w/ 24.65
.58 much better

KOH Tube weighs 27.63
VS 27.38
Δ = .25 so there is better, but how much

.58 + .25 = .83 total recaptured. better.
2.07 - .14 = 1.93
So we assume
1.93 - .83 = 1.10 due to oxygen
our measurements are

H_2O .58 gms
CO_2 .25 gms
O_2 1.10 gms
Σ = 1.93
+ 0.14 unburned
2.07 original.

There are therefore our measurements. They be seen much better than the first run.

H_2O 0.50 gms $\Sigma = 1.93$ gms
CO_2 0.25 gms
O_2 1.10 gms

H_2O: $0.25\left(\frac{2}{18}\right) = \frac{.064}{.058}$ gms

CO_2: $0.25\left(\frac{12}{44}\right) = 0.068$

O_2: $1.10\left(\frac{16}{32}\right) = 0.559$ gms

Should have been 2

Moles:

H: $\frac{0.064}{1 \text{ gm/mol}} = .064$ moles (3.76) (4)

1

C: $\frac{0.068}{12 \text{ gms/mol}} + 0.14$ direct carbon $= .203$ gms
$= .0054$ moles $= .017$ moles. (1)

1 0

O_2: $\frac{0.55 \text{ gms}}{16 \text{ gms/mol}} = .034$ moles (2)

Should have been
CH_2O

Now fructose is $C_6H_{12}O_6$

Now, you did some really good work here.
But you totally messed out on carbon. Why?

Did you weigh the new tube that you made?
No, you did not need to. The CO_2 tube did not break.

H & O came out perfect.
How could CO_2 be off so much?

The only way she could have worked out is if

What if you add the remainder on C instead of subtracting it?

H_2O 0.5895 ms
C

Well, there is something wrong
and something is right.

Salt Tube: Start 24.65
End: 25.23
Δ = + .58 gms @ H_2O

KOH Tube. Start 27.36
End 27.63
Δ = + .27 gms @ CO_2

Now assume C, H, O

~~C: .58~~ (

H: $.58\left(\frac{2}{18}\right)$ = .064 gms

this is H_2

C: $.27\left(\frac{12}{44}\right)$ = .074 + residual $\begin{array}{r} 22.72 \\ -22.35 \\ \hline .37 \end{array}$

= .444 gms

O: 2.02 gms − (.064 + .444) = 1.512 gms

Good!

30 H: $\frac{.064}{1 \text{ gm/mol}}$ = .064 moles 1.73 ≅ 2

C: $\frac{.444}{12 \text{ gms/mol}}$ = .037 moles 1

O: $\frac{1.512 \text{ gms}}{16 \text{ gms/mol}}$ = .094 moles 2.5

Problem here Should be x 2

So we have C & H roughly correct!

$C_1 H_2 O_x$ has an error

We actually did find the Carbon - Hydrogen ratio!!!!

So we know sugar has a ratio of CH_2 empirical as

$C_n H_{2n}$

So Oxygen is way too high.
You have a problem of Oxygen
being the difference but you have
hydrogen & Carbon correct.
This is the most crucial factor!

You cannot assume the amount of oxygen.
Somehow is wrong of that assumption

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Combustion - Note by Lipids.

Sample tube, no caps ~~22.45~~ 22.46

Sample tube w/ Lipids 24.69

Salt Tube No Caps 23.00

Salt 3.15

Sample Salt w/ Tube NO Caps 26.14 vs 26.150k

KOH Tube No Caps! 22.58

Sample KOH w Tube No Caps! 25.57

~~2=300~~ 2.99 gms

Measured 3.00 gms KOH

WORKED BEAUTIFULLY!

HEAT SLOWLY!

Use 2.5 gms absorbant

CO_2 KOH Sample Tube: 25.65

H_2O H_2O Sample tube: 26.60

Raw Sample Tube 22.44 GOOD!

Great Work, All consumed.

Assume CH only:

CO_2 Δ KOH = 25.65
(CO_2) − 22.58
Δ = 0.07 gms

ΔH_2O = 26.60
− 26.14
= 0.46 gms

So

H: $0.46\left(\frac{2}{18}\right) = 0.051$ gms

C: $0.07\left(\frac{12}{44}\right) = 0.0191$ gms

			Ratio
H moles:	$\frac{0.051}{1 \text{ gm/mole}}$	= 0.051 moles	32.1
C moles	$\frac{0.0191}{12 \text{ gms/mol}}$	= .00159 moles	= 1

C_1H_{32} is the ratio we set. How is that possible

Assume Δ is Oxygen:
(24.60 − 22.46)
~~3.15~~ − (.051 + .0191) = 2.070

$\frac{2.070}{16 \text{ gms/mol}}$ = 0.129 moles → 81.1

Can you have C_1H_{32}? It would mean that there is something else

How can ga be off so much w/ the lab.

No CO_2 was absorbed. Why?

Lets use xylene as a test case.

XYLENE

Sample Tube 22.41

Loaded Sample Tube ~~24.45~~ 24.46

w/ spot Salt Tube (H_2O) No Cops ~~22.49~~ 23.00

+ 1.46 gms

24.46 gms

Δ = 1.47 gms Measure (24.47) gms OK

KOH Tube (CO_2) 22.59

1.42

24.01 (23.95) meas

OK

Δ = 1.36 gms

KOH Tube Post op 24.04 Δ = 0.09

Start 23.00 Δ = 0.53

These numbers are very similar to the lipid

Post SAMPLE TUBE: 22.37 vs 22.41 to start.

KOH (CO_2)

C: $.09 \text{ gms}\left(\frac{12}{44}\right) = .0245 \text{ gms}$

H: $.53 \text{ gms}\left(\frac{1}{18}\right) = .059 \text{ gms}$

Moles:

C: $\frac{.0245 \text{ gms}}{12 \text{ gms/mol}} = .00204 \text{ mol}$ 1

H: $\frac{.059 \text{ gms}}{1 \text{ gm/mol}} = .059 \text{ mol}$ 29

Essentially the same results.
So how is this possible?
No CO_2 is being picked up. A lot of water is.
30 times as much.
You added left over Carbon in the tube and
that is the only way you made it before

You are not picking up the CO_2.
A strong base. Should it be liquid? Maybe a paste

$2 KOH + CO_2 \rightarrow K_2CO_3 + H_2O$

↑ soluble while salt.

Answer is a solution!
Concentrated.

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Again w/ Xylene. KOH must be a concentrated solution!

Sample Tube 22.37
Loaded Sample ~~24.54~~ 24.36
Δ = ~~2.17 gms~~ 1.99

NaCl Salt Tube (H_2O) 23.01 gms
+ 2.09
25.10 gms meas (25.03)

Δ = 2.01 gms (25.03)

KOH (CO_2) Solution:
Sample tube: 22.58 gms
Conc. KOH Solution 25.16
Δ = 2.58 gms

Post KOH 25.15 No Change

NO GOOD.
Try again
filled tube made seal

KOH = 27.76

KOH 27.74
No EFFECT!

Why!?

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Sample tube again Pre 22.37
24.54
Δ = 2.17 gms

We are picking up nothing or CO_2 - why?

Salt Tube Post = 27.12
vs original 25.03
Δ = 2.09 this is a lot

Sample Tube Post = 2236 Complete

Oct 29 2015 Thursday

We now have a visible and metric method of determining the existence of CO_2.
Calcium Hydroxide
We had a problem as we were unable to detect it.

Exhaled air is 4-5.3% CO_2
But air alone is only 0.03%.

$\frac{300}{1E6} = \frac{x}{100}$ $x = .03\%$

So there is not a significant error source for us in our combustion analyzer.
We do not need to use <u>oxygen</u> !(|

The method of
Calcium Hydroxide (hydrated lime)
(Calcium oxide should also work) exposed to breathing will turn the solution cloudy (cloudy white into a straw color to solution).
You have repeatedly filtered a hydrated lime solution to create your control solution. It is reasonably clear and more than sufficient for testing purposes. What is indicated is that the solution LOSES weight after being exposed to CO_2 ?
Formed the precipitate $CaCO_3$
Calcium Carbonate.

OK we go. Let's try again.

Monitor visually the effect of air flow now. Use xylene a tiff confirm

Sample tube 22.36
24.55
Δ = 2.19

NaCl (H_2O) Tube (pre) 2.08 gms
23.01
25.09
Δ = 2.08 ✓

$Ca(OH)_2$ tube: 22.59
27.75
Δ = 5.16 gms

An important lesson to take place. CO_2 is not being produced. Why? Does NaCl react w/ CO_2?

Guess what? Orange tea (no cooling tube) immediately ~~produced~~ reacted CO_2. What is the difference?

but I have no proof that it is CO_2.

We are going to use the $CuSO_4$ instead of salt.

We are going to use steam.

Sample Tube

Pre	22.38
Post	22.92
Δ	0.54
Post	22.43

Δ = .05 Carbon

Water Tube ($CuSO_4$)

Tube only	23.00
Tube w/ $CuSO_4$	25.88
Δ =	2.88
Post	25.91

Δ = .03

CO_2 tube only	22.59
CO_2 tube w/ $Ca(OH)_2$	27.79
Δ =	5.20
Post	27.70

Δ = 0.09!

decreased as expected

Ok this is at least a different story

$C: +.05 + .09\left(\frac{12}{44}\right) = .0145 \text{ gms}$

$H = .03\left(\frac{2}{18}\right) = .00339 \text{ gms}$

Moles H $\quad \frac{.00339 \text{ gms}}{1 \text{ gm/mol}} = .0033 \text{ moles} = 1.0$

C $\quad \frac{.0145 \text{ gms}}{12 \text{ gms/mol}} = .0062 \text{ moles} = 1.88$

This strongly suggest a ratio of $C_2 C_{2n} H_n$

but we could be on the right track now.
What salt do you have that absorbs moisture?

What about baby soda?

It does not make sense that salt will not work. Do not use Corderay tube.

What about magnesium sulfate?

Again w/ $MgSO_4$

Sample tube 22.36
Post 23.42
Δ = 1.06

22.49 Δ = 0.13

$MgSO_4(H_2O)$

Tube: 23.00
Post 26.39
Δ = 3.39
26.58 Δ = 0.19

$Ca(OH)_2$ [CO_2:]

Tube: ~~22.61~~ ~~22.40~~ 22.41
Post, ~~27.65~~ 27.70
~~Δ = 5.04~~ Δ = 5.29

27.46
a couple of drops spilled Δ = 0.24

C: $0.13 + 0.24\left(\frac{12}{44}\right) = .1954$ gms

H: $.19\left(\frac{2}{18}\right) = .0211$ gms

			Ratio	
Moles				
C:	$\frac{.1954 \text{ gms}}{12 \text{ gms/mol}}$	= .0163 moles	1	3
H	$\frac{.0211 \text{ gms}}{1 \text{ gm/mol}}$	= .0211 moles	1.3	3.9

~~CH_3~~ CH_4

$C_1H_{1.3}$
$C_2H_{2.6}$
$C_3H_{3.9} \rightarrow C_3H_4$

~~to the first time, they seem~~ Very reasonable.

Now we can try sugar again:

You may have your first empirical formula that make sense,

You need only about 1.5 gms of $MgSO_4$ & 5 gms of $Ca(OH)_2$

Again w/ Fructose

Sample Pre 22.39
Post 24.02
Δ = 2.43 . .34
Post mean 22.73 Δ = ~~.09~~

$MgSO_4(H_2O)$
Pre 23.00
Post 24.12
Δ = 1.12
Post mean 24.77 Δ = .65

$Ca(OH)_2$ CO_2 22.40
Pre 27.47
Post Δ = 5.07

Post mean 27.33 - Δ = .14

C: .34 + .14 $\left(\frac{12}{44}\right)$ = .3782 ~~.0382~~ gms

H: .65 $\left(\frac{2}{18}\right)$ = .072 gms

Moles H $\frac{.072 \text{ gms}}{1 \text{ gm/mol}}$ = .072 moles 225

Moles C $\frac{.3782 \text{ gms}}{12 \text{ gms/mol}}$ = .032 moles 1

Use 2 gms $MgSO_4$

2 gms Sample — 55 ms $Ca(OH_2)$

$C_1H_{2.25}$ Very good!

2.25! You are in range.

a little high on Carbon

a little short in water, Cneed more $MgSO_4$

I am in range now!!!!

your actual answer is ~~C_2H_5~~

$C_1H_{2.25}$ instead of ~~C_2H_6~~

Now, what of then was oxygen (which there is)

Diff = 2.43 − (.34 + .65 + .14) = 1.30 gms

This is way too high.

~~1.30 gms~~

Moles O = $\frac{1.30 \text{ gms}}{16 \text{ gms/mol}}$ = .08125 way too high = 2.5 moles

So we see that oxygen is also somewhat in range

And now your actual answer is

$C_1H_{2.25}O_{2.5}$

H & O a little high relative to Carbon.

- $C_6H_{13.5}O_{15}$ way too high
- vs $C_6H_{12}O_6$ in range!

Lipids Now:

Sample Pre 22.41
25.26
Δ= 2.85
Post Meas 22.41

$MgSO_4$ (H_2O) Pre 23.00
Post 24.83
Δ 1.83
27.22
Post Meas Δ= 2.39?

$Ca(OH_2)$ CO_2 Pre 22.40
Post 27.99
Δ 5.59

Post Measure 27.52
Δ= 0.47?

Very strange. Lipids & Xylene appear to present difficulties.

Ratio

1 C: $0.41\left(\frac{12}{44}\right) = 0.1282\text{gm} \rightarrow \frac{.1282}{12\text{gms/mol}} = .01068\text{ mol}$

25 H: $2.39\left(\frac{2}{18}\right) = 0.2656\text{gms} \rightarrow \frac{.2656\text{gms}}{1\text{gm/mol}} = 0.2656\text{gms}$

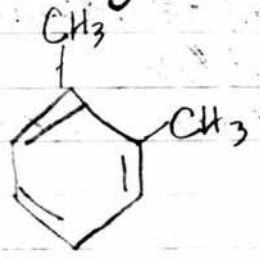
So Once again we get

$C_1 H_{25}$

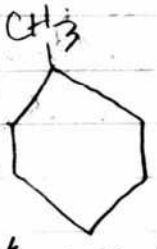
Last time using Xylene w/ KOH we got $C_1 H_{29}$
And w/ lipids we got $C_1 H_{32}$

which shows an interesting level of repeatability.

Xylene is $C_8 H_{10}$
So this means we are in this same range.
And maybe a better clean like $C_8 H_8$ or so.



So we might have something like



With something else substituted.

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Rainwater - Not Concentrated

Oct 30 2015 Voltammetry Testing:

Rainwater Testing Gain = Ø.2 $E_{Lim} \pm 1.5V$

Clear Distilled Water: +3 mV $\bar{X} = 3mV$
-3

Rainwater: +16 mV $\bar{X} = 16mV$
-16 mV

Assuming peak reached (not evident) we have

16 - 3 = 13mV ⇒ 13mV(10) = 130mV
130mV(2) = 260 mV = Ø.26V

If this was a peak, it would be nickel. but not likely.

Try to peak out This brings it to 22mV
± 2.0V
22mV - 3 = 19mV ⇒ 190mV
190mV(2) = 380mV = Ø.38V
This brings it up to Cadmium - Chromium level.

Now big to 2.5V
This brings to 26mV ⇒ 260mV = .26V
.26(2) = Ø.52V
This brings it to Cu(1)

You must concentrate the carbonate. Measure carefully.

Now to ± 3V
This brings to ~~Ø~~ 31mV ⇒ 310mV = Ø.31V
.31V (2) = Ø.62V

This brings to manganese level. Mn IV-VII not a very likely ~~state~~.

The suggest chromium III but this is not as likely also.
Iron & aluminum are approaching the last.

Now to 3.5V 350mV Ø.35V
Brings to 35mV = ~~100mV~~ = ~~.1V~~
~~.1V (2)~~ (2) Ø.35V = Ø.70V

The is setting on the zinc iron range.

Now ± 4V is 40mV ⇒ 400mV = Ø.40V
.40(V)(2) = Ø.80V

The is @ iron II-III level. The will belong to last for w/ ions and is unlikely.

The suggests manganese & aluminum are on the horizon.

± 4.5V This is 45mV = 450mV = Ø.45V
.45(2) = Ø.90V
Manganese (II) and Aluminum are indeed on the horizon.

± 5.0V leads to 50mV ⇒ 500mV = .5V ⇒ .5V(2) ≥ 1.00V
Mn & Al remain on list.

Nine - Mile Crk - Concentrated
Sample is condensed approx 60x
We will need more rain water to concentrate.

for the interim, Nine mile Crk sample.

Assume distilled water remains @ 3mV

Nine mile shows overpeak @ ± 1.50V Elim
Drop it lower. from approx 125 mV signal
w/ sharp peak.

1.20 does not show the strong peak.
I call it @ 1.35 Elim ±V.

Readings +114 $\bar{X}$=125
= 136

125mV ⇒ 125mV(10) = 1.25V
1.25V (2) = 2.50V

The measure is in magnesium

2.50 vs 2.37
The is a reasonable candidate
We may easily have more than one species

In Process of

Rainwater – Concentrating

2nd sample of rainwater.

1st sample, approx 50 ml, has been
1. examined under microscope
2. video made
3. preliminary metal analysis
(concentration not sufficient to plate)

2nd sample volume determination.

Glass jar (small) = 190 ml
w/ sample 421 ml Δ= 231 ml

Large glass jar = 455 ml
w/ sample = 484 ml Δ= 29 ml
Σ= 260 ml

Now evaporate.

These will combined into one small jar.

Oct 31 2015 Saturday
You must weigh all the rain water or ya
Well have no way of determing your results!

Original Small glass jar is 190 ml (gms)

This is WITH the cap
(It is 176 gms (ml) without the cap)

The original large glass jar is 454 gms (ml)
&
This is with the cap
(It is 434 gms (ml) without the cap)

Original Total 1430:
Glass Jar 1 w/ lid now weighs 312 gms
Δ = 421 − 312 = 109 evaporated.

260 ml Total water now available is 312 − 190 = 122 ml (gm)

Glass Jar 2(l) now weighs (with lid & sample) 1308 gms
854 ml This means sample size is 1308 − 454 gms = 854 gms.
This is before evaporation.

Glass Jar 3(l) w/ lid now weighs 1249 ml
795 ml This means sample size is 1249 − 454 = 795 ml

Glass Jar 4 w/ lid weighs 1267 ml
813 ml This means sample size is 1267 − 454 =

Now merged @ 1100 → Glass Jar 5 w/ lid weighs 751 ml
291 ml The amount sample size is 751 − 454 = 297 ml
1900 Rosel @ 1341: 1341 − 454 = 893
#1
Σ = 3019 ml
+ 893 ml
Σ = 3912 ml
The sample in the last twelve is preliminary & separate.

Nine Mile Ct - Revisit to
Select additional Metal

Let's recalculate the distilled water
but let's do it at the new reference
of $E_{lim} \pm 1.35V$ vs $\pm 1.50V$.

It is really high - Why?
It was at the test mode!
± 4.0 reference distilled water.

Ok it is @ $\pm$ ~~4.5~~ mV Ok It was set a little high
assume 4.0.

121 ~~122~~ 131
142 ~~-147~~ $\bar{X} = $ ~~134~~ vs 125
So a little higher
w/ a definite peak.

Let's remove the first peak, manually
identified a ~~magnesium~~ to seek a second.

@ $\pm$ 0.85 The second curve is
a smooth peak with a new curvature

+88 $\Rightarrow \bar{X} = 93$ $93(10) = 930 = .93V$
-98

$.93(2) = 1.86V$
That isn't really anything here.

$\pm$ 0.55 The bottom symmetry is in.
+76 $= \bar{X}$ 73 $73(10) = .730$ mV
-76

$.73V(2) = 1.46V$
In between the two was aluminum?

$\pm$ 0.6 +72 $\bar{X} = 76.5 \Rightarrow$ 765mV = .765V $\Rightarrow$ (2) = 1.53
-81

The closest is aluminum. As the possible

The steps you need a qualitative test for aluminum. The suggests magnesium & aluminum in the Mine Mile Creek water.

What happens if we drop to 0.4?

Every thing is really smooth.
Readings are 58 = 59.5 ⇒ 595mV
-61
.595V(2) = 1.19V
and this is manganese.
I would call this a smooth and more orderly fit. Two scenarios are

1. Magnesium & Magnesium
2. Aluminum Manganese

(Slight preference)

Lets move on with CDA South Fork Rock water.
The sample is Condensed Approx x 80%.

Doubled reference:

How can that read 0.8V?
Because once again it was in test mode!
± 5.5 $\bar{X} = 55$

You need to set in the habit of using the electrode.

We definitely have at least two metals.
We are already peaking @ ± 1.50 Elim.

Conditions: Gain = 0.2 Elim = ± 1.5
Careful, the top peak may be interfered with by the lower peak. Narrow the lower peak first ± 1.0V. Still way too high.

0.6 is looking decent. 0.8 is too high.
$\pm$ 0.1 is too high.
You 0.6. Measure +90 $\bar{X} = 100$
-110
100 (10) = 1000 = 1.00V ⇒ 1.00 (2) = 2.00V
Nothing really fits here.
Manganese is 1.2 Mag is 2.4

forget to subtract 5.5

Maybe you can justify $\pm$ 0.7V max. 98 = 111.5
-125
⇒ 1115 = 1.115V ⇒ 2.23V vs 2.37
Best call is magnesium again.

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There are actually 3 peaks that you can see by setting 6 lin ± 2.5
One of them is beneath magnesium
and one seems a likely yellow.

I cannot detect this @ 3.5V
measure 1.000V ⇒ 10V ⇒ ÷2 = 5V
not possible.

Maybe the problem is there is actually only two peaks and you have already picked up the higher one.

Remember that your water is concentrated.
Your measurements are correspondingly high.
You could reduce gain

What seems to be happening is that you get some distortion @ ~ Ø.7 and that Ø.4 is clean. These may be the two points.

± Ø.4 measures +78 / −89 ≈ 83.5 ⇒ 835V

Potentially very important: .835 (2) = 1.67 aye aye but this is aluminum!

± Ø.7 measures 105 / −122 113.5 ⇒ 1135

1.135V (2) = 2.27 vs 2.37 Mag.

We might have an issue here of detecting aluminum.
As your water is concentrated, the voltage required to produce the peak potential required.

The means the concentration could efficiently be measured.

There are some filaments showing up in the COA fourth fast sample tube. Not excessive, but it does appear beyond that of random contamination.
Approx 45 filaments have been found.

Hwy Drainage

Sodium & Aluminum?

Next, we go to highway drainage.

The concentration has worked well on the rose water samples.

Detailed water reference $\pm \frac{5}{2}$ mV $\rightarrow \bar{x} = \frac{5}{2}$ mV

Need to scale to $\pm$ 1.5V! It was @ 0.4V $\pm$!

Look like two co-elements again.
Sharp peak (too high) w/ secondary humps

Attempting to identify high peak first.

Seems to be @ Ø.85 Elm $\pm$
Measure: +122 $\bar{x}$ = 136 $\Rightarrow$ 1360
-150

1.36V(2) = 2.72V Equals Sodium.
2.72 vs 2.71 actual.
That is interesting. That is different.

Now go for lower peak:
I take it @ Ø.5
measure: +91 99.5 $\Rightarrow$ 995
-106

.995(2) = 1.99V
Nothing fits here. ??

@ 0.4 (possible to justify, more likely to lower than to raise. Measure +82 $\bar{x}$ = 87.5 $\Rightarrow$ 875
-93

.875(2) = 1.75V vs 1.68 Aluminum is closest.?

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You will need to run calibration demo with sodium & aluminum combined to see your behavior like this.

—

We now have a mix of aluminum & Sodium.
Reset to ±1.5
Why when I set it to ±1.5 does it read higher on LED

Turning on Channel B get rid of distortion
+12 $\bar{X} = -11$ (do not dismiss!)
−10

Resetting scale from distortion in signal.

We do clearly get a double peak.
And we can see that 1.50V is indeed way too high
Drop to 1.0:

Set for appx 3 peaks in full screen.

Indeed it is @ 0.85. The first indicates highest peak, just like before. Now measure:

+133 $\bar{X} = 146$
−159

We have a match here.

$146 - 11 = 135 \Rightarrow 1350 \Rightarrow 1.35V$
$1.35V(2) = 2.70V$ vs 2.71 Perfect
for Sodium

Now since we have a discontinuity in the curvature, we know that we have a secondary component of lower voltage.

Set E_{lim} @ $\pm$ 0.5V
Set for 3 peaks.

The discontinuity is removed @ ~~0.30~~ 0.37V

measure: +85 $\bar{x}$ = 92.5 ~~= 89~~
−10V

92.5 − 11 = 81.5 ⇒ 815 = 0.815V
0.815V(2) = 1.63V vs 1.66

Superb work. You have identified a 2 component solution w/ perfect scores within error limits.

The validator gave method.

This is really quite amazing work.

If your signal gets too weak, you can just change the gain. I believe instead of multiplying by 10 you will multiply by 5.

eg now it reads +170. $\bar{x}$ = 184 vs 92.5
@ Gain = Ø.1 -198
instead of Ø.2
And our reference will be 22 instead of 11
so we have
184 - 22 = 162 ⇒ 162(5) = 810 = Ø.810V
and Ø.810V (2) = 1.62 vs 1.63
vs Actual 1.68

so you then work fine.

Notice we still have a slight discontinuity.
I believe this means a 3rd component.
Reduce further w/ the adjusted gain of Ø.1
We are now @ Ø.32V

Set to Ø.25V± Keep at 3 peaks/screen!
This looks very smooth.

Ø.30 introduces a slight discontinuity.
Down to Ø.28 This is it.

You must be very good w/ gain measurements here
Ø.28: Measure 149 $\bar{x}$ = 161 ~~161(5)~~
-173
161 - 22 = 139 139(5) = 695 = .695V
pile
.695V(2) = 1.39V Closest is dichromate?
Does Aluminum sulphate fertilizer contain dichromate?

Manic is 1.36. This is it!

3912 ml | Additional Jars (large)

Glass Jar 6 w/ lid weighs 1331 gms
1331 - 454 = 877 ml

877 ml

Glass Jar 7 w/ lid weighs 1251 gms
1251 - 454 = 797 ml

797 ml

Σ = 5586 ml

Let's go back to calibrate.
Concentrated by approx ½+.

Reference distilled @ 1.5Ø ± Elim ± 18mV
+

There is no peak yet.
Raise to 2V.±

2.0V
Small peak is come in @ ~~1.5V~~ -sacrificial
Gain is @ Ø.1!
Measure: !

+175 $\bar{X}$ = 182.5 ⇒ ~~@ (5) = 912.5~~
−190

Actualy
~~= .9125 V~~ 182.5 − 18mV = 164.5
164.5 (5) = 822.5 = .8225V
Gain = Ø.1 .8225 (2) = 1.645 V

This is indeed aluminum
1.645 vs 1.68
There is no other Competitor.

There is no secondary peak detectable.
Lets weigh the final jar.

You should be able to determine the Concentration.

Question: Do you always subtract the actual background?

This is the relationship

Actual mV = (50 · gain) * Measured mV

and $E = 2 \cdot E_{1/2}$

Let determine how the multiplicative factor works w.r.t. gain.

Gain	Factor
.2	10
.1	5

$y = ax + b$

Factor = 5 * gain · 10

n Factor = 50 · gain

It is slightly more detectable @ 2.1 V

Gain = 0.1 Elim = ±2.1 V

Measure ±200

$200 - \frac{2.1}{1.5}(18) = 200 - 25.2 = 174.8$

$174.8(5) = 874 = .874V$

$.874V(2) = 1.75V$ vs 1.60

still the same Conclusion

Actual Voltage will be 2.05 V

+195 $\bar{x} = 197.5$

−200

$197.5 - \left(\frac{2.05}{1.5}\right)18 = 173.5$

$173.5(5) = 867.5 = .8675V$

$.8675V(2) = 1.73V$ vs 1.60

1.64 1.73 $\bar{x} = 1.71$ vs 1.68 V = 1.7% error

1.75

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Your next equipment is to weigh the samples.

Weight of final sample = 275 gms. w/lid

Original sample was 260 gms.
We know the small jar w/ lid weighs 190 ml

So 190 gms
+260 gms
450 grams – original weight

Now it weighs 275 gms. So 450
−275
= 175 gms (ml) now remains in jar

vs the original of 260
Therefore full sample is $\frac{175}{260}$ = 67.3% of the original
(Remember you combined 2 jar portions into 1).

This is wrong. Jar holds 190: You have about 40% left
or about 75 ml left
$\frac{75 ml}{260}$ = 29% approx is left.

Let figure out what we did wrong.

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Empty jar weighs 190 gms

It now weighs, with the assay sample 275 gms
so in the jar there is now

$$\begin{array}{r} 275 \\ -\ 190 \\ \hline 85 \text{ ml} \end{array}$$

There was originally 260 ml in the
original sample.
Therefore we have
$\frac{85}{260} = 33\%$ of original sample left

or, it has been concentrated by a factor of

$$\frac{260}{85} = 3.06 \quad \simeq 3$$

(concentration factor of Glass Jar 1 (small)

I think you would like to keep
this separate.

Now, what I would like to do is to determine the concentration level.
The require distilled water.
But it is worth it.

You will need at least 4 samples.

100 ml
200 ml
400 ml
800 ml

Σ = 1500 ml should be OK.

Use .05 gms $AlSO_4$ in 100 ml of H_2O.
1.0

Elim ± 1.5 V Gain .1 Meas ± ~~6.7~~ mV $\bar{X}$
You have a peak so it is too high.

1.3 looks close. Meas 193 $\bar{X}$ = 205.5
-216

$205.5 - \frac{1.3}{1.5}(7) = 199.5$ $199.5(5) = 997.5$

$= .9975(2) = 2.0$V then is too high.

Adjust to ± 1.10 V Measure +165 $\bar{X}$ = 175
170 -165

$175 - \frac{1.1}{1.5}(7) = \cancel{160}$ $\Rightarrow \cancel{160}(5) = \cancel{840}$ 850
850 1.70 170
$\cancel{840}$V $(2) = \underline{1.60}$ vs 1.68 This is it
Since Gain is so
high, you pick the max of the smooth curve

All at 1.10V Gain = 0.1 +/-

Next we dilute by a factor of 2.

200 ml

You should not need to recalibrate it.
You should be at the right voltage now.
We should simply measure. Correct.
+143 $\bar{X}$ = 147
-151

Now dilute by 200 ml:

400 ml: ± 110 $\bar{X}$ = 110

800 ml ± 89 $\bar{X}$ = 89

Now measure ~~rainwater~~ Concentrated again:

We measure: ± 101

Then is essentially a perfect measurement.
Right within range of the final dilution.

We now have all we need to figure out concentration

The readings are

100 ml ~~205.5~~ - 175 - $(1.1/1.5)7$ = ~~160~~ 170
200 ml 147 + $(1.1/1.5)7$ = 142
400 ml 110 - $(1.1/1.5)7$ = 105
800 ml 89 - $(1.1/1.5)7$ = 84

Now, the molecular weight of $AlSO_4$ is $Al_2(SO_4)_3$ (Al^{+3}, SO_4^{-2})

is 342.15 gms/mol

We have for 100 ml solution

$\frac{.05 \text{ gms}}{100 \text{ ml}} = (.05/342.15)\frac{\text{moles}}{100 \text{ ml}} = .00146 \frac{\text{moles}}{\text{liter}}$

for 200 ml: .000731 moles/liter
for 400 ml: .000365 " "
for 800 ml: .000183 " "

	So y	x	Power Relation
100 ml	.00146 M	170	Molar Conc. = $6.295E\text{-}10\,X^{2.8405}$
200 ml	.00073 M	142	$r^2 = 0.992$
400 ml	.000365 M	105	
800 ml	.000183 M	84	

? (10+5) = 96

So our concentration is .000269 moles/liter

(.000269 * 342.15)

$= \frac{.092 \text{ gms}}{\text{liter}} = \frac{\sim\sim.092\sim\sim}{} \frac{.092 \text{ gms}}{1000 \text{ ml}} = \frac{92 \text{ gms}}{1000000 \text{ gms}}$

$\approx$ 92 PPM of Al_2SO_4 However,

for the aluminum ion alone

Al has a molecular weight of $\frac{26.98 \text{ gms}}{\text{mole}}$

and we have 2 atom so.
our ratio is

$$\frac{2(26.98)}{342.15} = .158$$

So our actual Concentration of Al
is estimated at

$$.158\,(92 \text{ PPM}) = 14.54 \text{ PPM}$$
$$\approx 15 \text{ PPM}$$

EPA limits are
.05 to .2 mg/Liter Use higher limit

$$\frac{.2 \text{ mg}}{l} = \frac{.2E\text{-}3 \text{ gms}}{1000 \text{ ml}}$$

But we have an estimate $.158\left(\frac{.092 \text{ gms}}{\text{liter}}\right) = .0145 \frac{\text{gms}}{\text{liter}}$

and the a ratio of $\frac{.0145 \text{ gms}}{.2E\text{-}3 \text{ gms}} = 72.5$

times the EPA limit.

$\frac{1\text{ mg}}{\text{liter}} = 1\text{ PPM}$

We set ~ 7 PPM recommended 0.2 PPM.

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Now lets back up and assume a hydrated form of Al_2SO_4.

Lets use the octadecahydrate form

$Al_2(SO_4)_3 \cdot 18H_2O$ MW = 666.43 gms/mol

Now our molar concentration is determined as $\frac{.092\text{ gms}}{\text{liter}}$ of $Al_2(SO_4)_3 \cdot 18H_2O$.

Therefore the aluminum contribution is $\frac{2(26.98)}{666.43}\left(\frac{.092\text{ gm}}{\text{liter}}\right) =$

$\approx \frac{.0074\text{ gms}}{1000\text{ ml}} = \frac{7.4\text{ gms}}{1{,}000{,}000\text{ ml Gms}}$ = 7.4 PPM

Choose 1 PPM

= 7.4 PPM

(.2 PPM) Now the EPA limit is set at $\frac{0.2\text{ mg}}{\text{liter}} = \frac{0.2\text{E-3 gms}}{\text{liter}}$

So we have $\frac{.0074\text{ gms}}{0.2\text{E-3 gms}} = \frac{20}{37} \approx 40$ times

and the actual range is ~~3.7 to 14.0~~ 40 to 150 times

~~4 to 15~~ a times

$\frac{\text{mg}}{\text{litre}} = \frac{\text{grams}}{1{,}000{,}000} =$

Most conservative estimate is 40 time EPA recommendation

NOTE NEXT PAGE!

Now Recall that the sample was concentrated by evaporation by a factor of 3.06

This means our final estimate of Al^{+3} concentration is

$$\frac{7.4\text{PPM}}{3.06} = 2.42\text{PPM}$$

And the EPA upper standard is 0.2PPM

Therefore we have a ratio of $\frac{2.42}{.2} \cong 12$ Times

Important factor

Nov 01 2015

Carry forward 5586 ml

Glass Jar #1 (l) has now been folded into the sample set & is now available as an empty jar.

Glass Jar #4 (l) has also been folded into the sample set & is now available as an empty jar.

We will remove these jars from consideration & create two additional jars #8 #9

+864 Glass Jar #8 (l) w/ lid weighs 1318 gms
Therefore 1318 - 454 = 864

Glass Jar #9 (l) w/ lid weighs 1038 gms
This is my partially filled #
1038 - 454 = 584

+584

Σ = 7034 ml We are now going to remove Jar #1 (s) from the large glass jar sample set since it has been analyzed independently.

11/01/15 1020
Adj. Vol 6774 ml

Therefore our adjusted total volume is now 7034 - 260 ml = 6774 ml

11/02 #5 Folded In 297,893 1030

Our active jars are: ~~#2, #3, #5, #7, #8, #9~~
or ~~#2 864~~ ~~#8 864~~
~~#3 795~~ ~~#9 584~~
~~#5 297,893~~
~~#6 811~~
Σ = 6774 ml Check ✓

#4 Folded In 813
#1 Folded In 797
#9 Folded In 584 @ 1430
#2 Folded In 864 @ 1930
11/02 #3 Folded In 795 @ 1000

260 ml Glass Jar # 1 small
to carry to a close.

Let's centrifuge.

IR specimen taken on solid remains w/ ATR
Drops.

Under guard w/ Sam J 10.

Phenols & CN present.
Major implications to all of this.

Rain
Water (Man sample has been concentrated)

Nov 02 2015 - Monday Page 289

Lots of projects to work on of course.

1. Is there free software that will read spc files?
Yes!!! Specwin

Yes, I now have access to my data.

You have many many interesting project now.

1 Soil
2. Water
3 Air

1. Your man water sample is closing in.
3 days of prep now.

2. CH methods need clarification w/ Xylene

3. You now have access to your IR data!

4. Soil data has a first separation
IR? GC? Volatility?

rain
The water condensation is now complete.
It has been transferred to #10 (small) & #11 small

Wgt of #10	= 388 gms	− 190 gms	= 198
Wgt of #11	= 395 gms	− 190 gms	= 205
	403 = .0595		Σ = 403

Total wax: 6114

Concentration ratio is 16.81 times

We have therefore a total of 403gms (one) that is concentrated by a factor of 16.81 times.

Idea for analyses

0. pH 9.54 Init.
1. Color, Optical Density. Photo taken
2. NIR
3. Visible Light
4. TDS Distilled: 002 #10: 066 #11: 096 $\bar{x}$=81
81/16.81 = 4.8
5. Voltammetry
6. IR (ATR) of any solids.
7. IR (ATR) of pure liquid.
8. Microscopic Analysis

Let's mix #10 & 11 for a uniform sample.

Visible light spectrum indeed shows Color in the yellow green portion of the spectrum as compared to distilled water.

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Voltammetry Elim ± 1.5V Gain = Ø.1
Distilled water in ± 12

We have a peak showing up higher. Elim is ± 1.90V

+144 X = 150.5
−157

(5) = 677.5 = .6775

150.5 − 12(1.90/1.5) = 135.5 ⇒ ~~1355 = 1.355V~~

2(.6775) = 1.35V?

Nothing
But indeed it is most likely closest to aluminum @ 1.68

It is unclear if you really subtract the 15 or not.

+157 X = ~~329~~ 164.5
−172

~~329(5) = 1645~~ 164.5(5) = 822.5 = .8225

.8225 (2) = 1.645 V vs 1.68
Indeed we have a notice show what is the concentration.
Molar Conc = 6.295E-10 X 2.8405 X = 164.5
Mola Concentration = .00124 M
Now for $Al_2(SO_4)_3$ Hydrate we get
.00424 M (342.15 gms/mol) = .4243 gms/1000 ml = 424.3 gms / 1,000,000 gms

= 424.3 PPM Anhydrate!

However!!

But we really want to use Al. sulfate octahydrate
so what I have

$$2\,\frac{(26.98)}{666.43\ \text{gms/mole}} \qquad (424.3\ \text{PPM})$$

(Al$_2$ 3 mol wt)

(hydrate form)

(no. of Al atoms in compound)

16.81 Concentration Lacta

$= 2.04$ PPM

Incredibly good work.

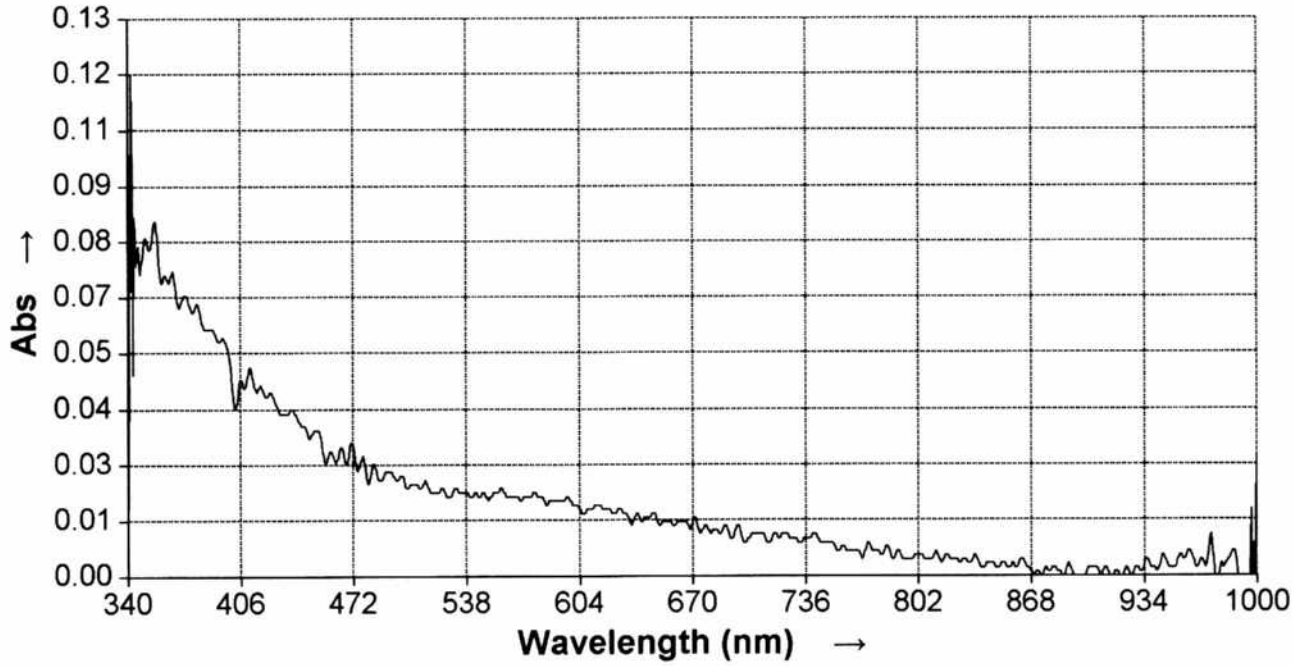
Same results achieved.

Rainwater Nov 02 2015

SPECTRONIC 200

Scan report

Spectrum of :	10	Analysis date :	02 - Nov - 2015
Analyzed by :	CI	Analysis time :	4:25:44 PM
Channel # :	2	Print date :	02 - Nov - 2015
		Print time :	4:27:02 PM



Nov 04 2015 Wednesday

ATR work & questions.

First question:
Background w/ & without Cover, any change?

Bellow?

Water?

Polystyrene?

Reference 1. Case 1: Open Cover ATR Gain 1

Now Compared to: Close Cover Gain 1

All noise (good) w/ more noise @ higher cm⁻¹ also Corresponds to increased noise @ CO2 & H2O locations.

Next is Cover Closed Gain 10.
We have a artifact change here.
Gain 10 introduces 2000 peak —
No idea why .. These are very problematic.

Gain of 10 introduces major spurious peak @ 2000 & 869 No idea why!
What do these peaks correspond to?

2000 is only in IRPal & R-N=C=S
869 is strongly listed as aromatics. 1,2,3,4,5 peaks
What is going on here? Why are these showing up with increased gain?

How could there be that these two functional groups show up under increased gain?
The makes no sense to me

This to me is really bizzaar.
It says the background @ high gain has these groups? Is such a thing possible?

What possibly can this mean?

Next we use open cover at gain of 10.

No Cover, Gain 10 gives an inverse CO_2 peak, the 2000 peak as well as the 869 peak.

This is pretty bizzaar.

Ok you see the problem.
These peaks correspond to the strongest peaks (or weakest?) in the background scan of CO_2 & H_2O
This is what is happening.

It is an inverse relationship of some kind.

I think the reason is that the same setting on background and sample must be the same.

I think that you learned the hyfe.

Also the background must be taken with the exact same condition eg cover, no cover, etc.

ATR background w/ no cover Gain 10 flattops - there is no band.

So it is NOT CN & Aromatic it is CO_2 & Water interference.

Flattopping does not necessary create a problem as long as you do everything the same.

It looks as though as long as you run the background & the sample in the same fashion that it will work out, even w/ ATR & even with gain = 10.

There might be an advantage to ATR w/ gain of 10?

Try the water drop idea

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I have just learned how to use ATR properly!

It is OK to use gain 10 as long as you subtract the reference out correctly especially the water!

Lesson - subtract the background completely & properly and you should be OK even w/ gain of 10 on ATR.

CO_2 can give some trouble but that is OK.

You see how it works!

Subtract the background completely & exactly.

Nov 05 2015

The rain water residual phosphate ~~extraction~~ have been repeated but only through an universal method. Ya get three peaks!

I do not know why but they are quite real and at the same location

Concentration expectant.

Nov 06 2015

We have some serious problems and lessons taking place w/ A+R.

We have a good control w/ distilled water as background as well as sample. None only as sample, where is great.

The problem is we use sugar water and we still end up w/ peaks @ 3290 and @ 1646.

This is our answer! And there is no answer in sugar water. & there should not be.

We also end up w/ a peak @ 1640, you called this a C=C ring Amide! RCONHR

There are potential errors due to this but they do not work!

There is also question of the alkyl halide.

The Amine group actually still looks ok. It is stronger and about 40 higher than sugar water.

But 1640? Why a repeat here?

@ 1640 It appears that both an amine and an alkene
This is why

Now I am down a test w/ distilled water and the balloon and I am having a problem again.

OK, now it is working. You must always note if load to specific background & test if balloons active.

ASR:
1. Background must be exact & loaded
2. Surface must be clean
3. Reference background must be perfectly accurate & identical

Ok, you have a very good result w/ Distilled water reference, balloon, cover.

The answer was the acetone film that you had made on a single KCl cell

Nov 07 2015

You got a great ATR plot of urine today closely to a parallel. Over w/c slide.

Boiled urine creates ammonia carbonate Ammonia carbonate is smelly salt.

Treated fat urine w/ ferric chloride produce Pt ferric phosphate. White, yellow precipitated.

It can be tested for by dissolving the precipitate in any mineral acid, eg HCl.

$FeCl_3$ + Potassium Physphate $KH(PO_4)_2$ → Ferric phosphate + KCl,

Nov 08 2015 Sunday

The lab is on its way to shutdown.
You have some practical time available
but much.

1. IR ATR
2. GC in fan fourier
3. HC Analyzer.

You could do some of all that.

ATR & pellet - what to do?
You have no single pellet worthy yet.
Why didn't a balloon work?
What about polyzeta?

Saran wrap is marginally worthy.

Ok Saran wrap w/ glass slide does not work.

Saran wrap w/ balloon on top and plate is
worthy.
I am not sure why the water band is
library so poorly.

Could there be water somewhere?

Even w/ solid wall drawn as background

Setting up control

1. Balloon w/ pressure plate
2. Leave it and recall it.

3. Now saran wrap w/ balloon & pressure plate.

Very interesting. We picked up very clear isolated peaks. Why?

It's backwards. Why?

Consider it without the balloon. We had picked the wrong background!

OK w/ the right background we are picking up a proper signal.
& still have a reversed peak.

Is the balloon absorbing?
Is the pressure plate absorbing?

Using Straight Air & the last!

We have success - Some ATR correction, some smoothing.

Sugar, however, did not work @ all.

Butter worked great w/ a glass slide.
About 10 yeah.

Try to use the glass slide whenever you can.

Your first real solid, you did some paper
on the Clemmyde.

Camptothecique next

Nov 10 2015

One source lists alum. sulfate fertilizer as 17%, 15% sulfate ($^{+3}$ $^{-2}$)

Assume it is $Al_2(SO_4)_3$

Now assume it is $Al_2(SO_4)_3 \cdot 18H_2O$

H	36.3
O	480.0
Al	54.0
S	96.2
	$\Sigma = 666.43$

$\frac{54}{666.43} = 0.08 = 8\%$ by wgt.

This says 18 H$_2$O. The mean less water. So alum is lower than we say.

$Al_2(SO_4)_3 \cdot 12H_2O = 558.4$

$\frac{54}{558.4} = 10\%$ Al

So the only way they come out even with no water.

$Al_2(SO_4)_3 = 342.15$

and $\frac{54}{342.15} = 16\%$

and they are an exact match w/ the fertilizer statement.

So I believe that I have done the right thing. I have assumed the most water possible.

This mean I am 1/2 of what it might be. I have assumed the least Al possible. This means my result may actually be double but I doubt it. It has to have water in the powder. I think good enough. It might be, however, more like 3PPM vs 2.0 all is good.

[redacted]

Index of Refraction 67.7

Nov 28 2015 Santa Fe NM!

Molecular weight determination of salt.

We have 398.5 ml (22.12 moles) H_2O
boils @ 100°C

Then we have 120 gm of NaCl (2.053 moles)
raise the boiling temperature to 103°C.

Question: What is the molecular wt of NaCl?

Molecular weight of NaCl is 58.44 gms/mol
H_2O is 18.02 gms/mol

Molar Mass (gms)

~~No of moles of substance~~ (salt) = $\frac{120.\text{gms}}{X \text{ moles}}$

$\Delta T = 3°C$

Molality

$m = \frac{\Delta T}{2.53\,(K_b)}$ = $\frac{2.32}{2.53} = .9170K$

Benzene triangle

Benzene problem

Wikipedia table has benzene $K_b = 2.65 \frac{°C \cdot kg}{mol}$

water $K_b = 0.512 \frac{°C \cdot kg}{mol}$

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$\frac{mol}{kg}$ $\quad molality = \frac{\Delta T}{kb} = \frac{3^\circ C}{.512 \frac{C^\circ \cdot kg}{mol}} = 5.859 \frac{mol}{kg}$

Moles of solute:

$398.5\ ml\ H_2O = .3985\ kg$

$5.859 \frac{mol}{kg} \cdot (.3985\ kg) = 2.335\ moles$

$molar\ mass = \frac{120 gms}{2.335\ moles} = 51.4 gms/mol$

Nov 29 2015 Santa Fe.

We are now trying to determine the MW of glycerol monolaurate using ethanol as the solvent.

We have roughly 150 ml of ethanol (bo. is @ 78°C) and we add roughly 50 ml of g.m. And it depends a many a difference.

Temp is going from 78°C to 186°C

Now we want to find the minimum proportional addition of g.m. that produces the temp change.

* It does not help and will not change the ΔT if you add more than the minimum required. It will only dilute the results.

Now we swell to 150 ml ethanol / 10 ml g.m. because we completely swamped the ethanol with 50 ml g.m.

The boiling point is 186°C @ 1mm Hg

but another source has it at 397.9° @ 760mm Hg

the mass change.

Solubility is listed as 12.67 mg / liter @ 25°C

This means 1.267 mg / 100 cc

Then means 1.267E-3 gms / 100 °C = Very small!!
So it is definitely not soluble in water.

Is it soluble in ethanol?

It is also called monolaurin
Density is .991 gm/cm3 $C_{15}H_{30}O_4$
MW 292.4

Specific heat is listed as 441.7 $\frac{J.mol}{K^\circ}$

Lets find something that does not boil as high, like acetone.

Now you are working w/ acetone & ethanol

	MW	BP
Ethanol	46.1	78.4
Acetone	58.1	56.0

So we have all ethanol, we added a little bit of acetone trying to determine the molecular wt. but this is impossible since acetone boils off before ethanol.

You must review the cards.
Use Acetone as the solvent.

Now if something does not dissolve in the solvent we does not work.

My idea of mixing two solvents together is not working.

The program may not be able to handle liquids not solubility.

Salt dissolves in water
salt does not dissolve in ethanol
salt does not dissolve in acetone.

Chem lab was definitely smart enough to figure this out. How did it do that?

Chem lab does not handle two non water solvents w.r.t. solubility. This is the problem.

You can only determine the mix of solids in a solvent, not two liquids apparently.

There is a very important issue here. When you mix two liquids together you do not get a new liquid, you get the combination (but still separate) of both.
This is important.

Now determination of salt again.

25 gms NaCl
87.3 ml H_2O raise temp to 103°.

& $\Delta T = 3°$ C

$$\text{moles} = \text{Molality} = \frac{\Delta T}{K_p} = \frac{3°}{.512} = 5.859 \frac{\text{mol}}{\text{kg}}$$

87.3 ml H_2O
$= .0873$ kg.

$$5.859 \frac{\text{mol}}{\text{kg}} \cdot (.0873 \text{ kg}) = .444 \text{ moles}$$

$$\frac{25 \text{ gm NaCl}}{.444 \text{ moles}} = 56.3 \quad \text{vs } 58.4 \text{ actual}$$

Very Good Work

So the question that is analogous to how do you determine the molecular mass of a liquid using colligative properties?

[redacted]

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Molecular Weight Determination

Nov 30 2015 Santa Fe

Chemlab Support Questions:

1. How to add Dumas? (greyed out)
2. Xylene specification lead to
   1°C w/ hot plate turned on.

Hexane has a BP of 68.
Benzene has a BP of 80

Theoretically you can use hexane as the solvent.

Xylene BP = 138.

Acetic Acid BP = 118.0

Google search phrase:
"Molar mass carolina biological determine"

Acid - Titration!
Volatile Liquid - Dumas
Compound - Freezing Point

$PV = nRT$

$n = \frac{Mass}{MW}$

$\frac{Mass}{MW} = \frac{PV}{RT}$

$MW = \frac{Mass \cdot R \cdot T}{PV}$

[redacted]

Interesting - What they really want to do is boil the water & use a scale!

You are using a lower temperature. This will affect the calculations (~ 60°C)

$^{\circ}K = {}^{\circ}C + 273$

$R = 8.314472 \frac{J}{K^{\circ} mol}$ Mass = .1159 gms (observed)

1 atm = 101325 Pascal

V = 56E-3 ml = =

1 liter = .001 m^3

So

$56 ml = 56E\text{-}3 liter * 1E\text{-}3 \frac{m^3}{liter} = 56E\text{-}6\ m^3$

$T = 60 + 273^{\circ}K = 341^{\circ}K$

$$So\ MW = \frac{.1159\ gms \cdot 8.314472 \frac{J}{K^{\circ} \cdot mol} \cdot 341^{\circ}K}{101325\ Pa \cdot 56E\text{-}6\ m^3}$$

= 57.46 gms Calculated MW for acetone

Actual: 58.08 excellent work!

$\Delta = \frac{58.08 - 57.46}{58.08} = 1.1\%$ error Superb work!

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On Acetic Acid / Xylene attempt.

Acetic Acid is not volatile. It will never work as there are no vapors to condense.

Dimer has to be for a volatile substance.

But the acetic acid should still work?

Question: Why do you have to put it in a double heater.

Let's dry at @ ~~60°C~~ 56°C
Then we have acetone boils away.

$$MW = \frac{Mass \cdot R \cdot T}{P \cdot V}$$

We have 0.120 gms @ @ 56°C

$$MW = 0.120 \text{ gms} \frac{(8.314472)(329)}{(101325)(56E-6)}$$

MW = 57.65 gms

vs 58.08 Fantastic.

The idea worked. You do not need a heavy base.

Error = 0.4%

Now go to a volumetric flask.

OK, the problem is that it has to be a Durran bulb. I do not know why but a volumetric flask does not work. It must be stoppered or contained somehow. With a volumetric flask it all should go.

Sodium Chloride specs are:

GMW 58.44
Density 2.17
BP 1465
MP 800.7
Heat Capacity 50
CAS 7647-14-5
Sol 0° 35.7
100° 39.12
Solid

1M Salt Solution
Conductivity = 111.80 uS
OK ✓ $141.3 \cdot (1) - 29.5 (1)^{1/2} = 111.8$
$141.3 \cdot (.25) - 29.5 (.25)^{1/2} =$
70.65 –

NaCl Conductivity Value are

$$\text{Conductivity} = 141.3 \cdot \text{Molarity} - 29.5 (\text{Molarity})^{1/2}$$

$$\text{g} \frac{1 \text{ gm}}{150 \text{ ml}} = k \left(\frac{58.44}{1000 \text{ ml}} \right) \Rightarrow k = .114 \text{M}$$

Conductivity = 16.11 –

$ax - bx^{1/2}$?

$\tilde{a}x - (bx)^{1/2}$?

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1M NaCl solution is supposed to have a Conductivity of 85,000 uS !!

He has 111.8
What gives?

He must be using a "brine solution"?

.5% NaCl?

1 liter weighs 1000 gms
so .5% = 5 gms

$\frac{5\text{ gms}}{58.44} = .085\text{M}$

$1\text{ ppm} = \frac{1\text{ mg}}{\text{liter}}$. $100\text{ ppm} = \frac{100\text{ mg}}{\text{liter}} = \frac{0.1\text{ gm}}{\text{liter}}$

Water Conductance Chem Lab -

$0.1 = .01(0.1\text{gm}) = \frac{.001\text{gm}}{100\text{ml}}$

so $1\text{ ppm} = \frac{1\text{ mg}}{\text{liter}} = \frac{.001(1\text{mg})}{100\text{ml}} = \frac{.1\text{ mg}}{100\text{ml}}$

$= 1E\text{-}4\frac{\text{gms}}{100\text{ml}}$

PPM	gms/100ml	M	uS
100	.01	1.71E-3 M	0.24
500	.05	8.56E-3 M	1.19
1000	.10	.017 M	2.35
5000	.50	.086 M	11.35
10,000	1.0	0.171 M	22.09

You have theoretically calculated the Brine Solution by PPM

Linear regression is perfect.

$uS = 2.2074E-3\,(ppm) + .1156$

$r^2 = .9998$

So conductivity is clearly linearly proportional to Concentration.

Unknown brine #1 $uS = 19.48$

$PPM = 452.95\,(uS) - 51.79$ $\quad r^2 = .9998$

$PPM = 8771.7$ PPM
Unknown Brine #1

It does not seem to me that the model corresponds to real life but it certainly does correspond to a linear relationship w/ Concentration.

.017

$E_c = 2.402$

His model is close to: $uS = 141.3 \cdot M$ darity gas very close

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Ok, I have figured how out.

the linear relation is actually calculated very closely to

$$uS = 141.3 \cdot \text{Molarity of NaCl Solution}$$

or

$$\text{Molarity of NaCl solution} = \frac{uS}{141.3}$$

and the model linear w.r.t Concentration very well.

The second term in the model is not required..

Now the data not conform to real life value since 1M NaCl = 8000 uS.! (instead of 141.3)

So the values are reduced by approx: $\frac{141.3}{8000}$

$= 1.8\%$ or $\sim 2\%$ of the actual value.

There is no reason to have the $x^{1/2}$ term from what I can see.

Yes, Conductivity is directly proportional to Concentration

The model is in mS not uS

If you want to calibrate in uS you can only use for very weak electrolyte solutions < 1E-4M

so you can increase it to actual value but like the meter is hard to read.

You could reduce it to 10% of the actual so that 1M ≅ 800.

$\frac{8000}{141.3}$ = 56.62 times

but if we want 10% of value but use $\frac{800}{141.3}$ = 5.66

times what he has, so we use 800

This work much better.

From table, a 0.05% NaCl solution @ 25°C should read 1014.90

Therefore $\frac{0.25 gms}{500 ml}$ = 1014.90 uS = 1.015 mS This is a 8.56E-3 M solution

1M/8.56E-3M = 116.82 and 116.82(1.015mS) = 118.6 factor to 1M

mS = 118.63 • Molarity of NaCl

Use meter in Solution Calibrated mS now closely to mS instead of uS

You see the problem.

You have a minimum detection level in mS of $\frac{.003 gms}{500 ml}$

≅ 1E-4 M of NaCl.

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Now what is inherently a true

ms of KCl ≅ 111.8 Molarity of KCl
25°C

Then we extremely close to NaCl

of

ms of NaCl ≅ 118.6 M of NaCl

Not very much difference here!

1.58M

Ferrous Sulfate FeSO4 is 53 ms @ 24°

~~15.06~~ ~~15.44M~~ ~~Solution~~ ~~15.44M~~ Solution ~~15.06~~ 1.58M 151.91

24° = 240 gms / 1000 ml = 120 gms / 500 ml MW = ~~154~~

240 gms ~~FeSO4~~ = ~~15.06~~ M solution

You solved it by trial & error.

Where did this one from?

So for Iron Sulfate:

ms ≅ 33.5 M of FeSO4

(53 ms) (1)

You solved this by trial & error

I am now successful in buying my Chemicals into Chemlab w/ the aged of conductivity now they available to me.

I think we have a problem here

[redacted]

Try a mixture of NaCl + FeSO4

100 ml , 1M solution of each.

We get 122.15 mS

Check: It should be 118.6 + 33.5 = 151.5

But the actual answer is

$$\left[(NaCl)^2 + (FeSO_4)^2\right]^{1/2} \quad !!!$$

$$\left[(118.6)^2 + (33.5)^2\right]^{1/2} = \underline{123.2} \text{ very } \underline{\text{close}}$$

Try for 3 compounds? We have a problem w/ FeSO4

~~OK, we solve it~~ (24% solution)

$53\,mS = C \cdot \frac{15.94}{15.06}\,M\ FeSO_4$

We want $C \cdot \frac{15.94}{15.06} = 1$ $\quad C = .\cancel{063}$ $\quad C = .066$

53 mS ⇔ 1.58M FeSO4

33.5 ⇔ 1.00 M FeSO4 OK

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ChemLab Version 2.6 Changes:

CDL is saved in a new logical location
Separatory Funnel!
Show meniscus
Volume Lab
Caffeine extracts Lab
Periodic table updated.

The TDS of motel tap water is 099.

Calibrate w/ NaCl.

Alka seltzer is 2620 PPM TDS
Tap water is 100 PPM

tap Water $100\,PPM = \frac{100\,mg}{liter} = \frac{0.1\,gms}{liter}$

alka seltzer $2620\,PPM = \frac{2620\,mg}{liter} = \frac{2.620\,gms}{liter}$

pH of tap water is 10.9!
pH of Alka seltzer is 10.4!

Water Test @ [redacted]

Distilled Water from Albertsons TDS = 0.0 !

I have calibrated the pH meter. It was way off. It is now 7.0 for distilled water. Mike Dietter.

Tap water from Hotel:
pH 9.2
TDS 108 ppm

Planning for Calibration of TDS meter.
TDS meter is apparently calibrated @ 320 PPM
$= \frac{320 mg}{liter} = \frac{0.320 gms}{liter}$

And we can not measure this with our scale.
We would like @ least 2 grams or 1 gram
in about 190 ml of H20

$\frac{1000 mg}{190 ml} = \frac{X}{1000 ml}$ X = 5263 PPM

We have $\frac{1.0 gms}{200.5 ml}$ We get TDS = 3870 @ 18.6°C

$\frac{1000 ppm}{200.5 ml} = \frac{X}{1000 p.ml}$ X = 4987 PPM Theoretical
We measure 3870

25°C - 18.6°C = 6.4° Deg C @ 2% per degree $(1.02)^6 = 1.126$
And $(1.126)(3870) = 4358$ vs 4987 Theoretical.

The is not too bad.

We would have

$X(1.126) = 4987$

$X = 4428$

The mean that we would take at 4 read. 443

We have $19.6°$

$25 - 19.6 = \Delta = 5.4°$

~~$1.02(5.4°) =$~~ $5.4° \Rightarrow (1.02)^{5.4} = 1.113$

Theoretical 4987 is the factor

so theoretical ~~5103~~ $= 4480$
1.113

is what we

Should be ready

OK. I have successfully calibrated the TDM meter to 4480. @ 25°C

Therefore a measurement will record the TDS and the temperature.

$(1.022)^{\Delta T^\circ C}$ is the factor to multiply or divide the measured value by to get the theoretical TDS

If temp is below 25°C we increase the TDS reading if temp is above 25°C (Cont Lab) we divide the measured TDS.

If we want to convert to uS, we divide TDS by ~0.6. This is the "conversion factor".

Eg we measure 4480.

$4480\ (1.022)^{5.4^\circ C} = 4985$ PPM Theoretical TDS

$4985\ (.6) = 2991\ uS = 2.99\ mS$ is our solution.

From Table $\frac{2991}{1014.9} = 2.95$ Factor

$2.95\ (.05\%$ Solution Reference from Table$) = .1475^{gms}$ Solution.

This mean we should have .00147S(200.5 gms H_2O) Salt = 0.30 gms salt, but we have 1.0 gms so I am not sure where other error is coming from.