CARNICOM INSTITUTE LEGACY PROJECT

A Release of Internal Original Research Documents

Authored
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TOP GRADE

Chemistry Vol XV

120

3 Subject

Perforated Sheets

• 10-1/2 x 8" • 26.7 x 20.3 cm

NOTEBOOK

College Ruled

6 94186 88890 5

MADE IN VIETNAM

[illegible]

[illegible] Road, Singapore [illegible]

Chemistry Vol XV

May 2016

May 27 2016

Regroup again. Good work w/ the mystery sample
[redacted]

We also did some good work w/ boiling points and melting points. We learned error with index of refraction is ± .02 not .001
We also did some intriguing work w/ solubility. We also found a great database on organics! I would like to:

1. Interpret the solubility results from the organic-chromatography batch.

✓ 2. Post biological photos on the rain page.
[redacted]
4. We have established a unique method of extracting the filament sample-network from rainwater.

And our general list is:
1. DNA work
2. Env. filament
3. CDB, Protein & Lipids work, lipids distillation
4. Complete rainfall studies
5. IR recovery - looking better
6. Electrophoresis
[redacted]
8. Hair analysis, pyrolysis
9. GC skill development, application
10. Davis, Organic Chemistry

Organic Database Search Journal.

The organic database @
www.colby.edu/chemistry/cmp/cmp.html
is incredibly valuable.

Search on
Melting point
Boiling point
Index of refraction
Some functional group info
how do you know if something is Unsaturated or not?
Permanganate.

There is a Merck database also, but it did
not find our compound.
And it Costs money.

To search Colby, you must allow error of

Boiling & Melting (Freezing Points) ± 5°C
Index of Refraction ± .03

You Can Combine
1. Melting Point (Freezing)
2. Boiling Point
3. Index of Refraction
4. Basic IR Functional Groups

This is all very good. 2500 Compounds.

ChemExp has some value also in that it allows error.
Melting Point, Boiling Point & Density

Chemspider & PDC may have some utility but
appears less beneficial.

Page 3

Lets try to identify Xylene

IOR = 82.4 IOR ≅ 1.495
(actual may be 1.496)

Pure xylene quite good. ~1.497

Hardware Store Variety.
IOR = 76.0 = ~~1.495~~ 1.478 Δ = .017
Pure 82.4 ~~1.478~~ 1.495

Refractometer is a lot more powerful than you knew it was. Error w/ pure xylene is nil.

So we can see there is some error.

Our Lipids have a IOR of 82.5 (= 1.495) but there is w/ Hardware Xylene.
We therefore have a Δ also of .017 from the original solvent. That is significant.

The refractometer is very accurate

Page 4

Try our lipids:

Brix = 82.5 Estimate 290°F ≅ 140°C

First level BP:	°C	°F	
	72	265°C	
first bubbles	90°C		
	104	280°	
2nd bubble	108°C		
bubble	108°C	295°C	
steady	112°C		
	115°C		
Steady	118°C		
Steady.	122°C		1st level about 1/2

You must provide greater volume

Then Xylene 1/3

	144°C	325°F
	154°C	350° ~~C~~ F
	168°C	365° ~~C~~ F
	168°C	400° ~~C~~ F
80° <1%	187°C	415° ~~C~~ F
		425° F

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Brix CDB Liquids = 82.5 (1.495)
Trying to get a bigger drop for IOR readings

°C   °F

1st layer 88°C ~~89°C~~   No wonder it was so volatile
Could be acetone? No, acetone is 56°C
Seems to evaporate very slowly. ~25%

Now it is increasing.

96°C is next

You need to use a more open heater.

108°

122°C

We have the 122°C & the 187°C fractions left.
Lets try to get IOR of 122°C

	T	IOR	
Fraction	122°C	82.5	Same as before?
	187°C BP	74.3 ~~74.5~~	
	Brix	74.5	= 1.475

Get more material tomorrow by condensing
and also melting point.

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Need MP

It would be interesting to know if the 122°C fraction can freeze?

Our conditions are

BP ≅ 187

IOR ≅ 1.475

Cycloheptanol $C_7H_{14}O$ BP = 185°C

OH or NH IOR = 1.476

saturated CH

MW = 114 gm/mol

density = .948

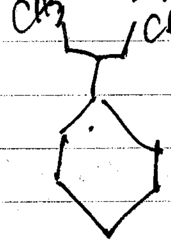
* 2 Amino 1 Hexanol BP = 190°C IOR 1.452 Saturated OH, NH

Dibutyl Sulfide

8 Choices. 1.49

9 3' Phenylpentane BP 191°C 1.488

Aryl, saturated & unsaturated CH



May 28 2016

Fortunately I have 1-2 drop left of Condensate from previous run. Lets try to get IOR again

We get 81.5 Brix = 1.493

(As a side note, 70% Isopropanol is Brix 25.0)

We still have another drop left to test freezing, melting point.

Now we believe the Condensate is the final temperature of 187°C but this has not actually been confirmed.

We therefore have 2 conflicting sets of data

Boiling Successively: BP = 187°C
IOR = 1.475

Condensate Method: BP = 187°C (presumed)
IOR = 1.493

This set seems pretty solid therefore

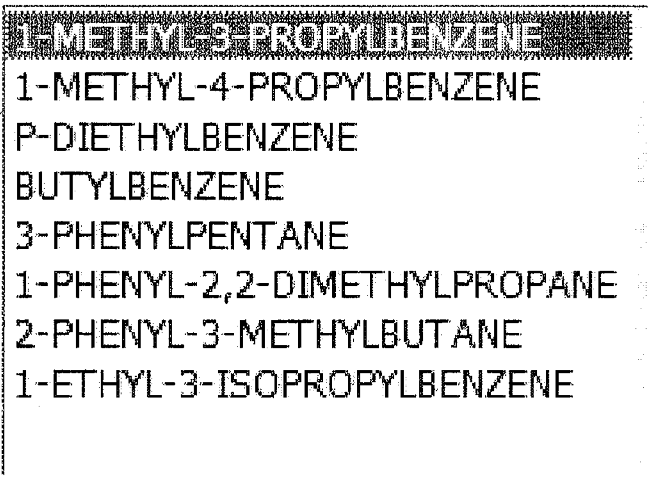
We also have an IR plot that goes along with this. It strongly indicates Alkanes, Aromatics & we believe saturated and unsaturated CH

Page 8

We have lists of candidates here circle the preferred set:

Untitled - 1

alias



1-METHYL-3-PROPYLBENZENE

propyl toluene 1 1-METHYL-4-PROPYLBENZENE

2 P-DIETHYLBENZENE

3 BUTYLBENZENE

4 3-PHENYLPENTANE

5 1-PHENYL-2,2-DIMETHYLPROPANE

6 2-PHENYL-3-METHYLBUTANE

7 1-ETHYL-3-ISOPROPYLBENZENE

This come from www.colby.edu/Chemistry/cmp/fd.cgi

Now, what we really need now is the freezing (melting point)

You can also look @ IR Spectra

You do not need to disconnect the ethernet cable. (maybe)

(1) Melting point -60°C BP 186°C IOR 1.496 MP -60°C

It will be worth it to buy the NIST database. 5200 spectra

Functional types: Aryl, saturated + unsaturated CH

It is also called by many names, such as 2 methyl 1 phenylpropane

JCamp is a .jdx format

CDB Lipids 107° average matches:

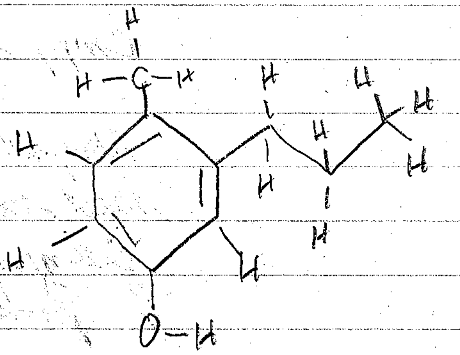
Methyl Propylbenzene

& Campho-Phenique spectrum surprisingly well

(11% Camphor, 5% phenol)

Methyl Propyl Benzene
is a "VOC" Volatile Organic Carbon

Methyl Propyl Benzene looks to be
a decent match other than phenol group



Our name created is
4 methyl 3 propylphenol

LogP is calculated by Chemsketch
as 3.46 +/- 0.20

LogP calculated by
chemicalize.org is
3.59 rather close!

BP ~ 187°C
IOR ~ 1.493
LogP ~ 3.52
Pub Chem ID is 61783-87-7

is a suspected endocrine toxicant
(PCB's & DDT are endocrine disruptors)

Page 10

you will need to prove the existence of phenols

~~It certainly looks like the lipids have phenols in them. A Fe3+ test for phenols in alcohol works, but it creates a yellow color, not purple like in water,~~

Careful here Fe^{+3} + Ethanol creates a bright yellow complex all by itself. No phenol required.

But, we do have a deep orange complex being formed! w/107°deg lipids ~~with raw lipids~~, some acetone & $FeCl_3$.
We need to trace this reaction down.

There is a precipitate forming also.
Need to do one thing @ a time.

Now I recall. It was the 107° lipid condensate that has formed the complex that is bright orange in color. There is no precipitate. This is telling us that the 107° lipid does have a phenol w/in it.

Now the question is, can we duplicate that same result with rainwater?

Tap water is forming the same color

Tap + Acetone + $FeCl_3$ + heat produce a yellow color.

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Alkene Test

We have not proven that we have phenols but we have a strong indicator via IR spectrum that we do.

What if it were alkynes rather than phenols? You need to check for unsaturation.

What is a test of something as unsaturated

In fact, the tap water is even brighter and deeper orange.

Soybean oil is largely unsaturated

* Permanganate Test for Alkenes

You had everything way off in terms of concentration. The method is

Note
Note
Note

1. Prepare a moderate color solution of $KMnO_4$ eg 2 drops in about 20 ml of acetone see notes or water!
2. Next, place about 2-3 ml of Ethanol in a test tube. Also add 1 drop 1M HCl!

or in water if soluble!

↑ notes

3. Now add 1-2 drops of your sample ~~ie, or~~ eg oil
4. Now add the dilute colored $KMnO_4$. You will see the solution turn clear when you add the $KMnO_4$ if alkenes are present.

The test was performed w/ great success w/ vegetable oil which is soybean oil which has a high unsaturated fat content.

We then performed the same test on the raw lipids. It performed flawlessly and then supports the conclusion that the lipids have alkene in them.

We wonder if we may have alkynes instead of phenols. We do not know that we have phenols yet.
But notice similarity to Campho phenique?

You do not have to use ethanol for the solvent.
1. If sample is water soluble, then use water.

2. If sample is not soluble in water, use ethanol.

The rainwater test is absolutely positive for alkenes. It is not positive yet for phenols.

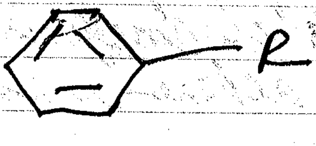
You also are not positive you have an aromatic

Act apparently means friedel craft acetylation

Aryl is any functional group or substituent derived from an aromatic ring, usually an aromatic hydrocarbon, like it phenyl

Aryl is used for abbreviation

A phenyl group is



Ok, our problem is not knowing if we have alkenes or not. We now know that we have alkenes in both the raw lipids and the rainwater.

The question is, do we really have aromatics? Why does COB lipids 187 match Camphor phenyque so closely?

Let's look @ ~~alkenes~~ aromatics in Murray p378
Aromatics or alkenes?

We need: for aromatics
1. 3030 We have 3022 w/ COB 122°
3017 w/ Condensed vapor

We are also supposed to have 1500 & 1600
We do have 1604 COB 122°C S
1604 COB 187°C W
1604 Condensed Vapor

Now for 1500? Condensed Vapor 1490

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Higher than 3000 cm means unsaturated CH.
Could be either alkenes or aromatic.
We know we have alkene. Do we have aromatics?

We positively have an aromatic.
Look @ Koji Fig 7a Page 126
We match

Koji	measured
3020	3022, 3017
1608, 1615	1604
~~19~~ 1494	1490
1380	1376
761, 792	792, 766
692	691

So it is definite that we do have an aromatic.

We do not have a phenol. Koji Fig 17 p130.
OH is a very strong signal.
Your test for phenols w/ $FeCl_3$ fails also.

So remove this

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We have some very important IR plots now from NIST. We need the database as soon as is possible.

May 29 2016 NIST data is requiring google back door access.

We have some important plot differences available now.
We know that COB 122 very closely matches xylene.
But we know that COB 107 has some very important difference.
Namely @ 3026, ~~2849~~, 1735, ~~1606~~, 1494, 1188, 765 & 700

Now, what happens is:

3026 is removed.	These are ~~alkenes~~ aromatics
~~2849 is removed.~~	~~These are alkanes.~~
1735 is added	These are esters - major
~~1606 is removed reduced~~	~~These are?~~
1494 actually holds - 1457	These are alkanes
1188-1174 is a major addition	This is phosphorus
765 is a major reduction	These are aromatics
700 is a major reduction.	These are aromatics.

~~Interesting that NIST xylene is~~
Go after the big player.

Therefore we know that COB 107:

1. Removes aromatics from the xylene (3026, 765, 700)
2. Maintains alkanes
3. Adds esters (this means bacteria)
4. Adds phosphorus, this means phospholipids

The power bacteria.

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You have done some good work today.

1. You have clarified the relation of CDB 122 to CDB 127 and the ester linkage of phospholipid existence. Bacterial note posted.

2. You have Spekwin working on the work laptop and you can do some very useful things w/ it.

3. You have a back door, likely temporary through Google image for NIST plots. You need the database.

4. You have cleaned up your set of IR plots for the current set of plots on CDB lipids, rainwater, etc.

5. You have some very useful reference plots
eg benzene
ethanol
xylene, etc.

6. You have posted all Categories on the Demise of Rainwater paper. Now you need to fill it in and we will start w/ pH.

We can now regroup on the priorities.

1. DNA work
2. Env. filament
3. CDB / Protein & Lipids work
4. Write up rainfall studies
5. IR recovery - looking better
6. Electrophoresis

[redacted] Book

8. Hair analysis & Pyrolysis
9. Gas C. development of skills, applications
10. Davis, organic Chemistry
11. Citizen samples

May 30 2016

I am going to study Xylene (commercial version) boiling point & compare it to the raw liquid using the local hot plate.

Also will try to determine the molecular wt of xylene as a control (both control of reagent grade)?

~~55.09~~ Weight of Erlenmeyer flask,
55.06
55.04 foil & wire
.04
.05 If you cannot get things
.08 hot enough you switch
$\bar{x} = 55.05$ to sand & the torch.

Let's learn what thermometer placement gives the best results. I think it will be in the oil but the pan heat tells you what is possible.

Torch idea works great
Set 2" away.

Comm. Xylene:
Initial boiling @
First bubbles @ 144°C - Not steady
Keep probe away from torch. It makes a difference.

163°C

Page 20

It almost looks like a more viscous fluid remained.

56.47 after cooling

$$\begin{array}{r} 56.47 \\ -\,55.85 \\ \hline .62 \text{ gms} \end{array}$$

56.57
56.53
56.57
56.54
56.55 ~~56.55~~
~~56.55~~ 56.57
56.56

Oil has made it to 189° good, 192°, 198°

So

$$\begin{array}{r} 56.56 \\ -\,55.85 \\ \hline .71 \text{ gms} \end{array}$$

$$\frac{.71\text{ gms}}{137\text{ ml}} = \frac{X}{1000} \quad X = 5.18 \frac{\text{gms}}{\text{liter}} \quad MW = \frac{\frac{\text{gms}}{\text{liter}}(.08206)\,T°(K)}{\text{atm pressure}}$$

$$\text{so } MW = \frac{5.18(.08206)(163° + 273°)}{.9} = \underline{\underline{206}}$$

So we have BP = 163°
MW = 206
IOR = 1.490

what is it?

Brix = 80.5

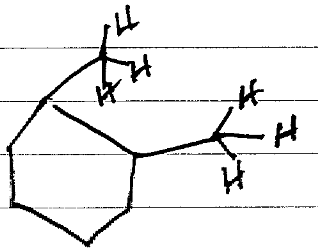
MW of Xylene is 106.2
So that is a big problem.

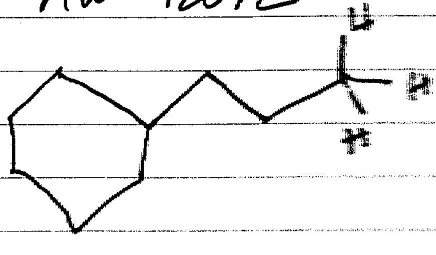
Method still needs work.

We get propyl benzene or Cyclohexene

Proply Benzene is:
MW = 120.2

Xylene is





You must determine BP again until
you get it right.

Boiling Point of Xylene is 138°C

We saw first bubble @ 144°

But we measure BP@ 163°C

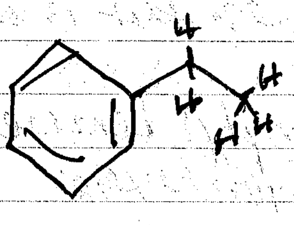
Do we have a mixture here?

Lets do it again.
Melting point is 13°C
There is no way in the world this is correct.
I think we do not really have xylene!

Wallace Hardware is True Value

Guess What?
It Contains xylene *and* ethylbenzene.

Xylene BP= 138°C
Ethyl Benzene BP= 134°C This is not
making sense @ all.
Ethyl benzene is



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How can it have a BP of 163°C?
So first of all you do have a mixture.

Then complicate life

Heating this up may not be the best thing in the world.
Usually contains about 15% ethyl benzene.

You must stop breathing this stuff.
Heating it makes everything a lot worse

You need to think about what you are doing here. Exposure to these solvents is not good or smart.

The less you put in a vial to heat the better! You need good ventilation.
To change your methods
Maybe you should wear a mask or get a respirator?

Way is not much better.

It is looking like my solution (xylene) may have been contaminated w/ the pipette.
Something ungodly was in it.

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Trying again w/ BP of xylene

Heat up quicker.
Some bubbles already @ 125°C
Lots vapor @ ~130°
134°C Close — 138 range

Oil is up
to 190°C
to 200°C
Good

Now it is slowing down @ 145°
Maybe when it is heated it forms
something else.

Ok you ~~boiled~~ ~~boill~~ boiled it too long
You should have stopped after it stopped boiling

56.32 But it is definitely Condensing.
56.35
56.38 Its actually very measurable.
56.38
~~56.42~~
56.38
56.38

IOR is probably different!
now. You are making a
new compound by heating it up

So MW= 56.38
−55.85
= .53gms

$\Rightarrow \frac{.53\text{gms}}{137\text{ml}} = \frac{x}{1000}$ $x = \frac{3.87\text{gms}}{\text{liter}}$

$$MW = \frac{3.87(\cancel{.0830}\ .08206)(136° + 273)}{.9} = \frac{144\text{gms}}{\text{mole}}$$

IOR = 82.2 Brix = 1.494

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So we have a final material

BP = 136°C
IOR = 1.494 (condensate used)
MW estimate = 144 gms/mole
(subject to most error)

Guess what we get!?

~~ety~~ ethylbenzene + Xylene!!!

Exactly what it is, a mix of both.

Ethylbenzene:
BP = 136°C (MW = 106) C_8H_{10}
IOR = 1.496
MP = −95°C

Xylene: BP = 138°C (MW = 106.2) C_8H_{10}
IOR = 1.496
MP = 13°C

Ethylbenzene is added to keep it from solidifying @ cooler temperature.

You did superbly but your MW determination has some big errors with it.

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Now that we have succeeded with two different solvents (actually 3) lets go back to the raw lipids and observe carefully.

125° 1st bubbles
130°
$\bar{x} = 136^\circ$ 132 starting
135 steady
140 It is ending 1st cycle
143 all done 1st cycle

153 starts cycle 2 ends quickly

208° still not

It still did not boil @ 208°.

Let's calculate mw.
Use approx eq wt @ 55.85

$$\Delta = \begin{array}{r} 56.26 \\ - 55.85 \\ \hline .41 \end{array}$$

56.33 Let's assume we are .05 high w/ more foil

$$= \begin{array}{r} 56.28 \\ - 55.85 \\ \hline .43 \end{array} \qquad \frac{.43}{137\,ml} = \frac{x}{1000} \qquad x = 3.14\ gms/l$$

$$MW = \frac{3.14(.08206)(225+273)}{.9} = \cancel{142}\ 143$$

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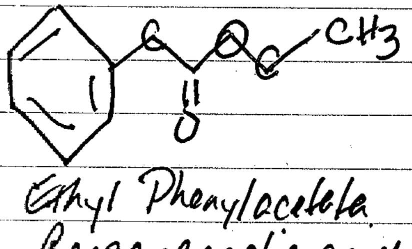
We have an estimated MW of 143
An estimated BP of ~225°C
and IOR
Brix = 81.9 ⇒ 1.494

MW IOR 2 Candidates: BP
164 1.498 1. Ethyl Phenylacetate $C_{10}H_{12}O_2$ MP? 229°
166 1.495 2. Phenyl Acetaldehyde Dimethyl Acetal $C_{10}H_{14}O_2$ 221°C

Use a BP of 220
62 1.495 1. Triethyl benzene MP unknown 215
2. Phenyl Acetaldehyde Dimethyl Acetal MP unknown ~~215~~

Use a BP of 215
1.499 1. Phenyl Propionate MP = 20°C Can't be 211
1.498 2. Butyl Phenyl Ether ??? MP -19° 210
1.497 3. Triethyl benzene MP 20° Can't be no? 215



Ethyl Phenylacetate
aka Benzeneacetic acid,
ethyl ester

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May 31 2016

Last full day in the lab for a week and a half or so.

We need to pack & have a short list this time

Rainwater BP & MP tests today & IOR
Carbonic Acid BP & MP & IOR

Sep 12 2016

Our Priority List:
NONE 1. DNA work
Initial Progress 2 Env. Filament
Generally Good 3. CDB, Protein & lipids work
Additional Findings 4. Write up rainfall studies
Generally Good 5. IR recovery
NONE 6. Electrophoresis
[redacted] Book
Generally Good 8. Hair Analysis & Pyrolysis
Generally Good 9. Gas C. skill development, applications
Very Little 10. Davis, Organic Chemistry
Generally Good 11. Citizen samples

Crutzen book?
Atmospheric Composition book
Exp. Organic Chem Book
ID by Chromatography Book
2 previous lab notebooks
Koji,
Current spectra
Davis Videos
Env. Chem Book
Menthol, Tea Tree & Supplements
Bicycle
Supplement Pill Container
Bring 2 masks
Hot Spot & Computer & Phone
Chemistry Book & Simulation Instead?
Lab Printouts?
Supplement Box
Headset
Ham Radio Books

Equipment
1. Chemistry Set

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~~Distilled~~ water	IOR (Brix)	BP
Distilled	0.0	
Carbonic Acid	0.0 pH 5.6	
Conc. Rainwater	0.0	

Carbonic Acid

92°C small bubble forming.
95° small bubbles.
97°
97.6 brisk bubbles
98.1
98.4
99.0 stable
99.2

It has stabilized @ 100.0 !!! (even though we are at altitude)
So it looks like the CO_2 boils out.
and that Carbonic acid may boil @ ~ 97.6
Let's repeat.

81° bubbles on glass side
86° edge bubbles
92° slow
96° brisk
97.0 steady
98 full boil

98.1. steady & stable
@ full boil

Slows down then temp
rises again.
~~99.3~~ ~~99.2~~ ~~stable~~ stabilizes
99.25

~98.0 98°C Carbonic Acid ~99.2 H_2O

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~~Again~~
Run for rainwater.

83 some bubbles on side of glass
88 slow bubbles ~~rise~~ forming
90° quiet
93 quiet
94 quiet
95 quiet
96 quiet
96.5 quiet
97 slow bubbles forming
97.5 slow " "
98 slow bubbles
98.7 steady bubbles now (large, full size but slow)
99 quiets down
99.3 ~~quiet~~ mostly quiet, some surface activity
99.6 surface activity
99.6 holding steady
99.5 Steady but a very quiet boil, not full or brisk
99.6 same, not a full boil. Oil is @ 135°C
99.2 But not a full boil.
99.4 went quiet again
99.2

Reduce sample tube.

104 Boiling now. in smaller tube.
103°.0 slow and stable.

102.5 slow and stable oil is @ 146°C

We have done well. The boiling temperature of Conc. rainfall clearly shows a problem also @ 102.5C

It is time to start regrouping for departure.

We only have about 3 hrs left of lab time for today. What else can you work on?

What about turning the GC on?

OK, let's revisit it. I have it on.
Gas flow still OK.

We have the gas flow @ 5
TCD Detector is set @ 210 (this should be
slightly above oven)
The detector is continuing to heat
The oven max is set @ 210.
The oven is currently @ 50.
Use .5 cc when you inject gas.
1 ul for liquid
We can start w/air.

There are residuals in the column.
Let's clean & bake it out. @ 180°C
Lots of activity in the column.
Still very active after 20 min.

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GC Area:

.024 CO_2
69.300 O_2
Σ: 69.324

$\frac{.024}{69.324} = .000346^{\%} = \frac{x}{1E6}$ $x = 346$ PPM

$\frac{.000346}{100} = \frac{x}{1E6}$ $x =$

38 times as much CO_2 in balloon breath vs Air CO_2.

Example is
$\frac{61.95}{1441.4} = \frac{x}{1E6}$ $x = 43{,}000$ PPM

Our Case is 13,000 PPM
Their case is 3.3 times higher than mine.

Ok, now how to approach xylene.

It boils on hot plate @ about 68°C. Why?
We also get CO_2.
O_2 & CO_2

We are [illegible] working w/ headspace idea.

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I think the xylene peak is the xylene peak and not O_2!

Notice how high the peak is & how concentrated it was. I could smell it. Notice no CO_2 peak this time.

You can test this by adding something to xylene

Maybe acetone? and then ~~x~~ lowering temperature to 80 [illegible]

Notice Xylene (commercial) has a much lower volatile residual in it that did not boil until ~ 185°C.
What is this? Try IOR first.

You must keep the room ventilated

I do not have enough material for IOR

Compare Air & Xylene

Air: Air Peak @ 0.15 @ 60°C Δ
N_2O_2 @ .476 $\Delta = 0.33$
CO_2 3.17 $\Delta = 3.02$

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Xylene headspace @ 170° @ 60°C

Air Peak 0.06
Main Peak 0.430 Δ= 0.37
CO_2 3.06 Δ= 3.00

We are not sure if they are different.
Set to 50°C.

Air @ 50°C:

		Δ
Air Peak	.05	0.
N_2O_2	.426	0.376
CO_2	3.95	3.90

Xylene @ 50°C Headspace 165°C

Air Peak	.04		
Main Peak	0.413	.409	Δ_2= .033
CO_2	3.97	3.93	Δ_2=.03 ~~= 2secs~~

.03m = 2secs. So they are both w/in the same margin of error. We have no way of showing that the headspace is different from the air. We will need to use fluid.

Now @ 120°C w/ liquid form of xylene
Air Peak .10
Main Peak 0.47 Δ = 0.37.
No CO_2

Now for 80°C
Air .06 Δ = .41 maybe?
Main .47

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50°C
Now for 60°C
Air .06
Main 0.49 $\Delta = 0.43$

N_2O_2 It was 0.38 so it seems to be different
= 3 secs

You still need to test further, however,
it does seem to be different

If that is true it xylene is highly
polar. You would have to try something
less polar. It also helps to get rid
of all air, then you know that it's clean

On cleanout, you had a definite peak
Very intense!.

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There is a very good chance that I can determine C-H ratio from GC & TCD

The small peek up first is H_2!

The second peek is O_2 & N_2 combined

the third peak is CO_2.

When you burn something you get

CO_2 & H_2O

you do not get Nitrogen.

So we should get H_2?, O_2, CO_2, H_2O?

We are start testing these on known hydrocarbons

We also are starting to separate xylene & MEK

Try Fructose?

Jun 17 2016 Page 37

Let's think about how to organize our time.

1. We want to copy Davis onto SD and have it available on the tablet
2. We want to watch Davis.

What are the most immediate needs:
1. To develop the demise paper
[redacted]
3. NHFC preparation
Relationship diagram
4. Organic analysis of rainfall would be very beneficial
You have conflicting & weak results except for acid-base reaction.

5. GC & IR are plain outright fun

What I would like to do is:

1. GC skill development and exploration
2. Try out the Edvotek labs
3. Watch Davis persistently.
4. Clarify the rainwater organics
5. Try new methodologies on the Env. Filanat.
6. Explore headspace and pyrolysis issues
7. Look @ citizen samples
8. Try microwave digestion more. Can you digest tree hair?

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Acetone Headspace.

.03m H_2 (or Air?)   80-120°C
.396 O_2 No additional peaks seen.
1.98 CO_2

With Acetone Solvent We have
0.08 H_2?   50° - 120° - 180°
0.50 O_2?

No additional peaks.
Survey mode will be 60-120-180 Total Time 20min

.01m H_2? (or Air?   Acetone Solvent
Δ.416   0.486m O_2? Acetone?   60°C 10min (5min is enough)

Now air.
.04 Initial Peak (H_2 or Air?)
Δ.366   .406 This is different?   So it is different
2.97 CO_2 60°C 5min

Back to Liquid Acetone. Good flat signal.

0.08 (Notice the small peak @ 0.09m)
Δ40   0.48 min   60°C 5min
No CO_2
Now back to air   60°C 5min

0.42

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60°C 5 min

Acetone Solvent:

① Reduce Volumes by 1/2
Liquid 1/2 ul
Gas 1/4 ml

② Amount of air in solvent injection is minimal, ie ~0.1 ul vs 1/4 ml

	Δ
.07	
.486	.416
.08	
.480	.400
.07	
.493	.423
	$\bar{x}$: .413

Air

		Δ		Δ
H_2?	.04		.040	
O_2N_2	.406	.366	0.410	0.370
CO_2	2.970	2.93	2.910	2.87
	.05			
	.420	.370		
	2.920	2.87		
		$\bar{x}$: .369		

Acetone Headspace. Heated Beaker to ~80°C, Column @ 60°C

.04	
.416	.376
2.910	2.870

So this is clearly air

GC Lessons

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Numerous observations here:

1. Air injection & solvent injection have radically different ratios of air

2. Acetone solvent is different than air in retention time

3. Headspace idea on acetone is not working.

4. No CO_2 peak on acetone (air volume injected is extremely small).

You therefore seem to have success.
What if we add a small amount of Campho phenique to the acetone.
We can see that essential oils are better to add.

5. Use 1/2 the stated volume
   1/4 ml gas
   1/2 ul liquid.

6. Only 1% of solute in solvent! Not full strength!

7. Some good news. I have learned how I can use acetone as a solvent. This is very useful

8. Size of the peak does not correspond to the concentration unless the thermal conductivity is the same.

9. Time in the column is primarily a function of
1. solubility of the solute compared to that of the column.
2. the carbon number, ie, molecular weight is second

60-120-180 Survey
10 ul TeaTree oil added to acetone

@ 60°C
We have 3 peaks"

Mag.
.013 .07 air peak
.162 .10 small secondary peak
1.10 0.400 Δ = 0.41 acetone

~~0.34mV~~
.034mV 1.88 min - unknown? Tea Tree
a small peak

No peak @ 120° ~ 180°C

Again: Poor injection.
.06
0.41 .41 (acetone)
1.93 weak, barely detectable.

Again: .06 Δ
.41 .41 (acetone)
1.95 definite but disrupted peak-
this is our solute (ie tea tree)

Again: Nothing beyond solvent this time.

$\frac{0.5ul}{3ml} = \frac{0.5E\text{-}6l}{3E\text{-}3l} = \frac{x}{1E6l}$ x = 166 PPM

Good, this is about right.

Again The signal is barely detectable.
Double the strength and add benzene (next round, not this)

This one is a little different.

.07
0.40
1.09 very very weak

3.66 broad, but possible new signal.

Double the tea tree oil first.
Still did not show up?

.07
0.41
No peak @ ~1.9. Why?

Seems like noise.

Again: No minor peak. Maybe 2.23?
Increase Concentration farther.

We seem to have a weak but broad signal @ 3.14 m.
This all looks uncertain.

You might try high current

Increase flow rate & current level. & temp

We have a definite peak @ 19+ min!

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Everything has changed completely using a higher flow rate and high current & high temp.

Condition
Flow rate = 7 (vs 5)
Current = High
Temp = 180°C.

We have picked up a strong broad peak w/in a *much* shorter time interval!

Let's set to 8 PSI
We may even have another peak ~ 15.5
as well as ~1.5

We have another huge peak @ ~20m.
It may well be residual and it may be a water or an alcohol peak from the past.
Sharp rise, very gradual descent.
The magnitude and area are huge.

The mean 3 definite peaks in the system in addition to acetone

Increasing the flow rate and temperature are the only way the new large peak was discovered as well as higher current.

We have a huge peak now @ ~17. m.
Now we must resolve if the might be
acetone instead of the tea tree oil.
The size of the peak is huge.

You are only using 1 ul so how could the
peak be that big?

You must now revert to acetone again.

I think that you are also driving out a lot of
water or alcohol also from the paste.

Now acetone alone
You still have to wonder if 0.286 is an O_2 peak?
based upon the size of the large peak found @ ~19.

It does not produce the 20m peak.
This says the 20m peak is Tea Tree
and 0.286 is acetone @ 8 flow rate & high current.

But guess what? Now we have a new peak
showing up @ 25 min! So it still requires
sorting out!

Another peak may have come in @ ~ 32 min
on the second round.

Jun 18 2016

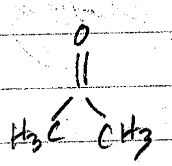
Lets get ourselves organized for today:

1. The rotary tool is charged up.

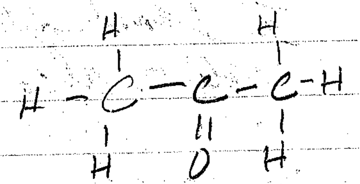
2. The GC column has become very active after I increased the flow rate (probably to a max). We need to learn if the column is cleaning itself out from the past and what is a current reading.

Our factors of investigation are:

Acetone is a Ketone



1. Acetone Liquid
2. Air
3. Acetone Headspace
4. Tea Tree oil solute 10 ml



And we are separating out these influences.

3. BP and IOR (actually melting point of liquid paste is a very interesting topic).

4. KOH & Microwave of rain concentrate is an interesting topic. So is microscopic analysis prior to destruction of the sample.

5. IR ATR of a paste worked flawlessly.

you are not getting a very good IR analysis of the current rainfall concentrate. This is of concern to me.

We already have an index of refraction estimate for the CDB lipids. It is 1.494.

We also have an estimated BP of 225°C.
This is for the liquid form.

So we have some molecular estimates from the organic database already for that.
Methyl propyl benzene is one candidate for that.

But we also know from the IR analysis of the evaporated paste that we are matching perfectly (In this evaporated paste form) that of a

saturated fat w/ esters and phosphate groups.
This satisfies perfectly the bacterial domain.

This is almost as clear as you can get.
This seems to be the preferred final form.

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Notice that Ketones DO NOT fare very well on the GC separation chart.
Acetone may be much harder than you think to separate.

So now we have to wonder about our "air peak" again.

Acetone may not be the best thing to be putting onto the column.
It seems like it would be good since it is so volatile but it is not good on the chart.

—

* I have lost my flash drive.
It will need to be replaced.

—

I have found a way to penetrate the residual materials (solid) from the rainfall concentrate. By microscopic examenation, it obviously contains proteins & filaments characteristic of morgellons.

You also have an acid base heat reaction that you have discovered that will produce these filament materials from the rainfall concentrate itself.

Important discoveries and methods regarding:
1. Production of filament-protein structure from concentrated rainfall samples
2. Penetration of filament structures w/ microwave & KOH

The methods are:

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This is a very important discovery page

1. To produce the filament-protein structure
   1. Add ~ 3-4 drops 1M HCl to the rainwater (~ 3-4 ml).
   2. Shake and let it set for a minute or two.
   3. Add enough KOH to turn the solution basic
   4. Heat @ about 65-70°C.
   5. The filament structure will become apparent under close examination w/ a magnifying glass.

2. To penetrate the solids of the rainfall concentrate
   1. Pipette the solids from the concentrated rainfall sample. (~ 3-4 ml)
   2. Evaporate the solution & solids in a watch glass
   3. Scrape the solids into the center of the watch glass and collect in a central location.
   4. Place the evaporated solids on a KCl IR disk
   5. Add 1 drop 1M KOH
   6. Place in a microwave under the lowest setting possible - no more than 1 minute. Actually energy transfer is ~ 10 secs in the microwave. The sound of the microwave will demonstrate absorption.
   7. This will form a film on the KCl plate.
   8. IR Analysis as usual.

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I have done three good things today

1. The rainfall particulate matter has been penetrated and examined w/ IR. Some interesting results for certain.

2. I have posted the spectrum of the penetration by alkaline microwave.

3. I have cleaned the GC column after two days of running & I am aware of the acetone - ketone question.

4. I have posted the four primary conclusions (4) to the Demise of Rainwater paper.

Now we can go back to acetone & tea tree oil.
I can also review my lectures.

Watch Davis! Lecture 04
A fantastic course.

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Acetone (liquid)
We have a clean run w/ a clean Column.
180°C for 60 min w/ low current.

It is essentially flat.

.06 m.	H_2?	3.76mV	narrow
0.20m	Solvent	2.06mV	broad
0.46 m	Air: N_2, O_2 or CO_2?	.08mV	minute

We suspect H_2, solvent, Air or CO_2?

Now let's add 10 ml Tea Tree.
Turn to high current.
180°C 60m High Current.

The high current seems to be causing a lot of baseline drift that was not there w/ low current. Maybe it needs to stabilize for a long period first.

You could try IR runs while the long term GC run is executing.

We have success with a peak!
It is occurring @ ~ 60 + 3 + 18 min = 81 minutes
@ 180°C w/ high current.
Now we can see that temperature and flow rate were critical to displaying the peak.

Now the question remains. Is it Acetone or Tea Tree Oil?

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We have a peak 81 minutes into the run!

After a 4hr run w/ no additional peaks it is becoming clear that the 83 min peak is acetone, because it is polar.

Our summary should be

1. "Air Peak"
2. $O_2 N_2$ peak

3 concentration of Teasho is too low.
(< 200 ppm)
needs to be higher.
4. 83 min is - acetone w/ a long tailing peak.

Acetone is not advantageous as a solvent.

What is the structure of MEK? a xylene?
or naptha?

MEK has an oxygen also. It is soluble in water!
Xylene ~~C_{10}~~ C_8H_{10} a bit high
Naptha C_8 - C_{20} which is high.
Ethyl benzene is also C_8H_{10}

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Total Success!

Used xylene, which is actually a mix of

methyl benzene 85%
& ethyl benzene 15%

We get 2 peaks, very close together and overlapping @ ~ 48 minutes @ 180°C + 60 ml

The second peak does not finish and we should have left it for 90 min., low current.

Now we can add tea tree oil
and see.
We should drop flow to 6.

and add tea tree.

GC - More Lessons

1. The first peak is the helium peak, the carrier gas
2. The second is O_2 N_2
3. From there it depends upon solubility, molecular weight, and the volume of sample, and the volatility of the sample as a whole.
4. The GC Diagnostic Chart gives you an idea of what you are up against.

Jun 19 2016 GC

Benzene would be the next thing to try but you do not have a reliable supply of that. It is good for testing purposes however.

Ethanol not so good also.

Now Xyl & Tea Tree Oil.

1. Air Peak
2. O_2, N_2

Flow@ 6 ml/m.
T = 180°C
Low Current

I need a non polar column (eg, resin) so that polar solutes do not react w/it and get pushed through.

I have a polar column, polar things interact with it, and they take a long time to get through the colum.

A non polar column would allow things to get through it more easily.

GC is going very well. I have clear separation of Xylene & Tea Tree (Comm Xylene)

1. Ethly-benzene
2. Methy benzene
3. Tea Tree
4. O_2, N_2
5. Air Peak

I just barely made it w/ 90 min. Adjust to 120 min right away, maybe more.

Jun 19 2016

1. Check that acetone does not damage pipette stem. It does not. Good.

IR Study.

ATR worked extremely well for acetone. You are doing good.

Acetone is not damaging the pipette yellow stem @ all. This means that you can collect gas in acetone and then the acetone will evaporate very quickly. This should leave what is left.

It looks like it could work very well.
Do acetone & xylene mix? Yes!.

Evaporation of Acetone mixed w/ Xylene seems to have worked very well. There is a way that you can ~~isolate~~ things.

Let's work on mixing rainwater w/ →
acetone → xylene

Xylene also evaporates after the acetone (you have mild heat @ 42°C). So this is great. You are indeed left w/ a residue that you should be able to use on the ATR. What you will do is watch the evaporation closely and pick up the last drop or two, place it on a KCl disk or on the ATR, slowly warm it up to drive off the remaining volatiles.
This is going to work great.

Rainfall Analyses   Page 57

I am mixing rainfall concentrate
w/ acetone.

Now adding the xylene.

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You are doing some very good work.

1. You now have ~~3~~ 4 different methods of examining the rainfall.

   1. Direct evaporation
   2. Microwave w/ KOH & then evaporation
   3. Extraction w/ Acetone & Xylene
   4. GC Extraction into Acetone or Xylene
      Transfer to IR.

2. You have run a GC-IR Extraction trial w/ Tea Tree Oil - Acetone - Xylene injection into Acetone. You have evaporated the volatiles. I believe you have the tea tree oil left. You have an ATR spectrum of it w/ only 10 ul. The spectrum looks excellent & the two closest matches are Campho phenique & COB lipids @ 107°C. This most certainly makes sense and I think that the process has succeeded incredibly well, on ATR no less.

Let's go use the tea tree directly to see how it matches.

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3. You also have very good GC separation taking place now and you understand how it works better w.r.t.
   1. solubility
   2. Carbon number
   3. The GC diagonal chart.

4. 10 µl looks to be plenty for IR transfer and anal.

5. We have only a partial match, but not too bad, w/ actual teatree oil. Tea tree oil by ATR worked just great.

Let's try the extraction (acetone - xylene) that we have created from rainfall.

It will occur in two stages.
1. The xylene evaporated for residual.
2. The solids @ the acetone - xylene interface layer.

You have a Xylene ATR background now it is performing flawlessly.

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You are now reducing a rainwater concentrate — xylene extraction – separation to a 10 ul sample that goes into ATR IR.

This is superb work.

You are getting a very strong spectrum.

Absolutely superb work here.
Fantastic spectrum achieved.

From GC work of the extraction acetone-xylene medium we know that we have at least 2 additional components that make up the organic structures. Both of them have clearly a broad peaks so that indicates that they are not straight forward hydrocarbons and that they probably have a polar ~~element~~ aspect to them especially the latter one, that is more likely of a higher molecular weight. The latter compound is very broadly tailing. It is out @ 160 minutes.

June 20 2016

Keeping organised.

1. You may have 3 components in the organic extraction. Extended now to 240 min @ 180°C

2. There may be a "fatty acid methyl ester" aspect to either the rainfall concentrate or the COB lipids.

Microbial fatty acid profiles are unique from one species to another. There are libraries.

There are three methods of identification of bacteria:

1. Biochemical ~~tests~~
2. Fatty acid profiling
3. DNA sequencing.

What is your goal here today?
To assess the general complexity of the organic components being found w/in rainfall concentrate.

Let's go, we know how to do it.

Question: Did I evaporate the xylene rainfall extraction before placing it in the ATR IR? I think I did.

GC Xylene sample is running also. 180°C Low Current
We had a problem w/ initialization but since — 6 ml/min
first peak does not come in (ethyl benzene) until
05 min there was no problem in restarting
the instrument and software. We have the
xylene peaks coming in for me @ approx 35 min.
Now we will see what happens afterward.

We are also evaporating rainfall xylene extract
(it must be made fresh) for transfer
to ATR IR for second run.

Running benzene would be of value on the GC also.

Remember that in testing the xylene ATR run
you placed pure xylene on the ATR, so you
did not "evaporate" it. Evaporation of xylene by itself
does not change the concentration of the xylene.

But evaporation of the xylene with a solute will increase
the concentration of the solute w/in the xylene,
which is what we want.

So therefore we wish to and we are evaporating the
solute-xylene mixture under mild heat to
concentrate the sample for the ATR-IR second run.
It is @ 70°C in the gravel bed.

We are also going to try and collect (GC)
the samples this time! in acetone traps!

Watch GC closely for additional peaks

We are seeing the tailing start on the first peak after xylene. If we see a rise we will go for the trap.

Let's start it. The slope is changing from xylene methyl benzene peak.

Chromatograph minor disruption shows the ~~start~~ of the collection point
You picked it just right! It is a bump on the tail of the methyl benzene peak!

We know that peak #1 has a polar element to it. There are two reasons solutes take a long ~~[illegible]~~ time to elute on the column. They are either polar or have a high carbon number, or both. The long tails indicate a polar nature which is what the compound has. Therefore acetone should be a good solvent for the trap. When the tail is done the compound is done for the trap. OK, it is flattening out. Time to stop.

Now we wait until peak #2 or until the slope flattens out more thoroughly.

We have inserted the trap (acetone) for peak #2. (~14 min)

The IR sample is now sufficiently evaporated. It took almost 40 min @ 70°C gravel/bed temp. We get a very weak result. Since we used only the rainwater, we know now that the organics are coming from the solids at the bottom of the rain.

This does not work. You must use the solids @ the bottom of the rainwater.

OK, on GC we are collecting the 2nd peak. It started @ 12 min and it looks superb.

This is why we did not see solids @ the xylene - acetone-water interface form. This will be your clue.

On GC peak #2 we are also getting tailing. This indicates a 2nd polar compound. (Remember this is from the good extraction sample).

Tailing is actually very useful for collecting in traps.

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By IR alone we have now separated the water soluble (rainwater only) component from the total components (solid + rainwater).

From this we can deduce which functional groups derive from the solid material.

GC:

Peak #1 more likely has a greater hydrocarbon content based upon the slope of the tail. So we presume that it is a mildly polar hydrocarbon based molecule.

Peak #2, however, appears to be highly polar. Maybe an ester? a phosphorus compound?

IR:

It was an accident supposedly a mistake, in how you separated water soluble vs total organics with the use of IR.
This was good:

GC:

Peak # 2 is starting to flatten. It is time to remove a vent.
Let Peak # 2 flatten out some more now.

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Trap for peak #3 has been inserted.

There are no additional peaks beyond #1 & #2
that have been detected.

Vacuum distillation would be a great thing to
develop here. You cannot use the main pump
but what about the hand pump?

We have nothing in trap #1.
This does not mean nothing exists, it means
that it is not soluble in acetone.
We will repeat w/ xylene trap.

You will need to try the GC work again
w/ a xylene trap.

The xylene extraction method applied to solids
requires the evaporation & concentrating of the xylene!

The signal is too weak from the solvent alone.
GC:
You have a small peak 5.5 min.
Does this correspond to CO_2?
It does not seem plausible. It is too broad
& too large relative to the air peak.
There is something there.

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Remember to get traps set up in Xylene.

You are working both IR & GC @ the same time

Trap1 Initialized @ 17m (Xylene)
Trap1 done @ 73m

Trap 2 Inserted @ 73m (Xylene)
Trap 2 complete @ 101m

Trap3 Inserted @ 110 min (Xylene)

Ok, we are on the shutdown of the lab

June 27 2016 Back in Lab.

We are working on several fronts:

1. Working w/ the rainfall - acetone - xylene extraction results & GC. We are showing two components that you are trying to learn about.

   Your qualitative study on GC should be helping you. You are also trying to feed the elutes into solvent traps and then feed them into IR.

2. You have a good IR plot of the rainfall extract. It is based upon a xylene extraction w/ evaporation of the solvent (xylene). You have a good solid plot.

3. You have a lot of methods that have now developed for organic analysis of the rainfall.
   1. Direct evaporation (results seemed poor, most likely because of salt emphasis.)
   2. Microwave w/ KOH is quite interesting as a method.
   3. Extraction w/ Xylene-Acetone & evaporation of Xylene solvent under mild heat.
   4. GC Traps into a solvent, dependent upon polarities

4. We have succeeded to a large degree now with vacuum distillation. This is really interesting what has been developed here. We can probably work w/ 5 ml if we need to.

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FB ⇔
Rodent 01 St

So what is your real goal here?

1. Develop GC skill set. Isolate & identify the nature of materials. BP de~~termination~~. Vacuum distillations. Rainfall organic components. Standardization of GC column results. IR library purchased.

2. Get the hotspot renewed.

3. We know that whatever is in the xylene is soluble so it should be safe to use in the GC.

Several projects could be of interest here.

1. Distilling the acetone portion of the rainfall extraction. Noticed that we have 3 layers that have separated so there is more going on than just the xylene layer.

Adjust here Now

Distillation ~ 5ml.

89°C ~~81°C~~ 1st elute 10R = 10.5 = 1.347
Adj = 89 + 12 = 101°C

91°C ~~92°C~~ 2nd elute 10R = 2.6 = 1.336
= 103°C

Water is 1.332
Water is not in the database.

Lets now distill water (distilled) to see how close it comes to 90°C @ 4·2600'

Good news that we can successfully distill 5 ml of solution. This is very workable.

The Japan SDBD database is back online! Very valuable, esp when combined w/ the organic compound database.

The data base is @

www.colby.edu/chemistry/cmp/mole.cgi

Water is boiling @ 86°C. So there is a lot of a problem in terms of calibration.

It looks like the higher temperature is what you want, not the lower.
We get 94° for the high temp.
80° for the low.
Actual should be ~ 90° @ the elevation.
Therefore we need to adjust our low measured value by + 10°C that is a lot of error.
+2° for sea level = 12°C

We are going to boil water again because obviously there is a calibration issue. We are setting the thermometer deeper. You need another one

Distilled Water.

We are measuring 94°C, actually about ~~94.5~~ 94.2
This is about 4° too low.
Plus sea level change = +2°C
So we anticipate our correction is ~ +6°C

This means our first elute has a temp of

89° + 6°C = 95°C w/ IOR 1.347

Our second has
91° + 6 = 97°C w/ IOR = 1.336

So elute 2 is water.
Elute 1 is something else and it is NOT acetone.

Let's repeat the test of rainfall concentrate.

Pure water measure 94°C + 6°C = ~~9~~ 100°C ok.
Hi was 101 (94+101)/2 = 97.5

Elute 1 again 94(Lo) - 99(Hi) $\bar{x} = 97.5$
IOR = 11.1
or 94+6 = 100°C IOR = 1,348

It appears that we do have a compound to analyze.

Acetone has an IOR of 1.359 but the BP is 56°C
So this cannot be acetone

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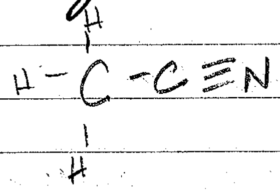
Compounds w/ an IOR of 1.348 include
Methyl Cyanide   BP = 82°C   IR = 1.344
Diethyl ether. BP = 34°C Not possible.

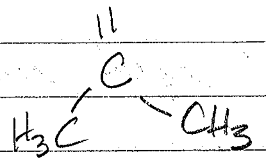


$H_3C-C\equiv N$

Guess what? It is also called Acetonitrile

So clearly there is an acetone component.

Acetone is



We could have had the transformation take place.

Use ATR! Superb. It worked perfectly.

You have indeed isolated the cyanide compound.
clearly acetone in the extraction process.

The compound you have distilled is indeed methyl cyanide, or Acetonnitrile.

You need now get the spectrum of methyl cyanide

* You have done superb work here w/ distillation and IOR analysis (index of refraction), boiling point analysis and infrared analysis w/ ATR

This is very good work.

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For tomorrow:

1. Upload new organic extraction photo
2. Retrieve methyl cyanide spectrum.
3. You need to start writing up the paper.
4. You need to start preparing a relational diagram.
5. You would like to distill the lipids also.
6. Have Analyse a look on.
7. Air Filter Project

[redacted]

  2. Relational Diagram Prep
  3. Infrared searches (not firm).
  4. pH research.

We actually have a minimum of three organic components in the extraction Crangall sample. One from distillation of the acetone layer (acetonitrile), and 2 from the GC work.

We are working on [illegible] of the Xylene layer as we do not have enough material to distill.

COB Distillation of Lipid

Page 74

Let's distill the lipids. No vacuum!

~~B. Vacuum pressure is -9.0 -13.0 -14.0~~

① ~~119°C~~ 122°C -126°C

② 182°C

③ 182°C+

For Elute #2, we had a BP of 187° on earlier trials
This means that our average is (182+187)/2 = 184.5
We also had a IR of 1.493
Now we have 1.491 so $\bar{X} = 1.492$

We have a very close match with

1 Methyl 3-Propylbenzene BP=182°C IR=1.494
Functional Types: Aryl, saturated & unsaturated CH

1 Methyl 4 Propylbenzene BP=183°C IR=1.492
Functional Types Aryl, saturated & unsaturated CH

Diethyl benzene BP=184° IR=1.495

Jun 28 2016 Page 75

What I need to do that is most important is

1. Create NHFC plans
2. Write rain paper
3. Interpret all IR spectra
4. Plan for CI longer term

[redacted]

2. NHFC Relational Diagram
3. pH progress

2. My projects:
   1. The rainfall paper should be worked on slow & steady
   2. The IR plots should be analyzed. radically simplified
   3. The layer should be identified within the rainfall. you have one. now I have both.
   4. The Japan database is back alive
   5. Colby database is also fantastic
   6. Would be good to start working w/ the proteins.
   7. GC skill development & model development.
   8. We also have the Env. Filament Project of course
   9. Pyrolysis & Head space methods GC & IR
   10. Distillation is a very powerful technique
   11. Air Filter Project

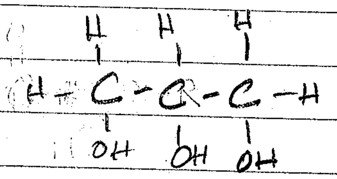
Page 76

Need to
1. Revise Organic Chart (2)
2. Revise Extraction Picture

Hexane means 6 Carbons, not in a ring

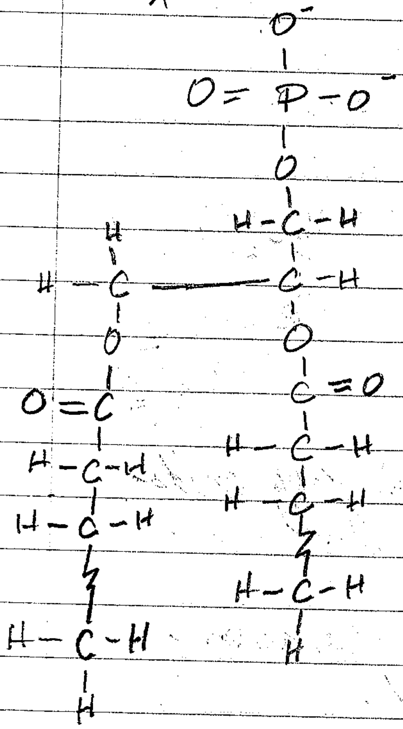
We know the lipids have a general structure of

See next page

Glycerol is 

$H-\overset{H}{\underset{OH}{C}}-\overset{H}{\underset{OH}{C}}-\overset{H}{\underset{OH}{C}}-H$

This is polar

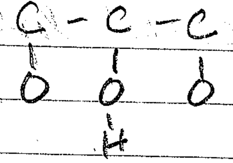


This is lecithin

Phosphates are polar
Glycerols have OH attached
Esters link the fatty acids
Aliphatic chains are hydrophobic

These general comments may be as far as you can go right now.

A glycerol bridge can have only 1 OH



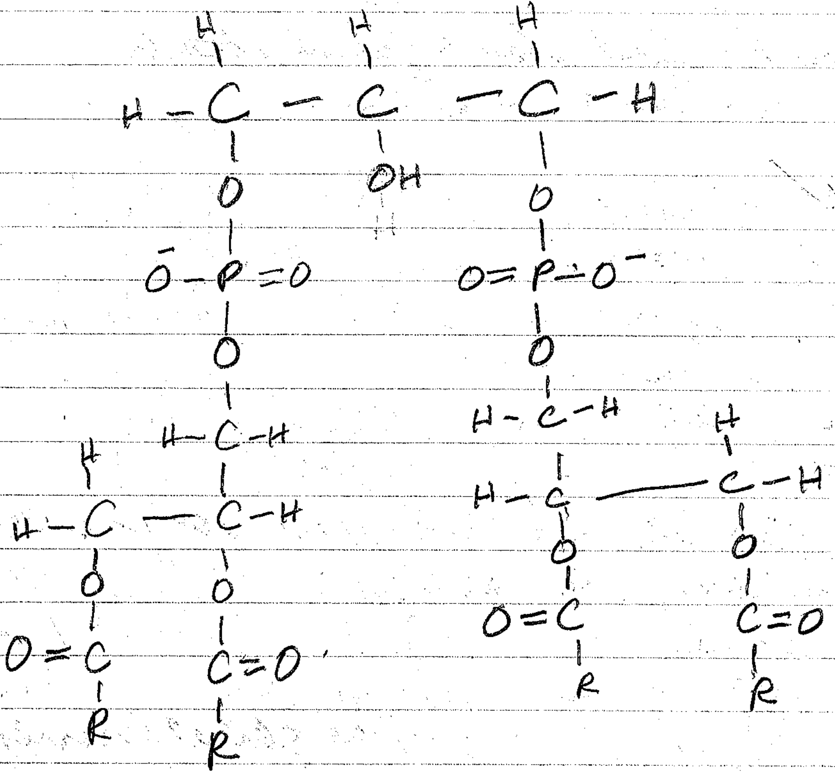
$\underset{O}{C}-\underset{\underset{H}{O}}{C}-\underset{O}{C}$

Page 311 Lehninger gives a prime example.

Page 77

A prime example of bacterial lipid form as shown on p 311 of Lehninger.

It is called Cardiolipin



R means the tails of long chain fatty acids

This is an example of how a common bacterial lipid is constructed.

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I think that we do need to start looking @ the protein

We see that a base (KOH) applied to the protein definitely has an effect upon it. We therefore conclude that the protein is likely acidic in nature.

5 solubility tests performed

Acid - none
Ethanol - none
MEK - none
Xylene - Coagulation
Base - KOH - some solubility here.

Microwaving elemenates the solubility in KOH.
It must therefore denature it.

There are only two acidic amino acids, glutamate and aspartate.
Glutamic Acid

I was testing protein in Sept 2015 (end of Sept)

Page 79

The classification of amino acids

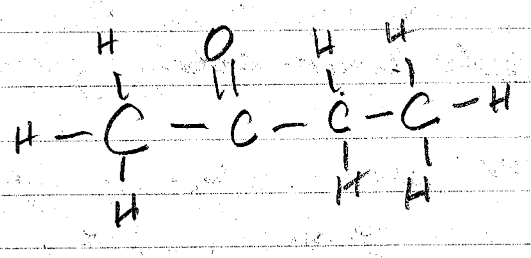
Polar & Non Polar
Acidic & Basic

Seems to me we have one that is non polar and one that is acidic

Milk, however, contains 18 amino acids, so how would you ever know how to begin?

You are however, pretty sure that you have an acidic amino acid because of the base reaction.

You also have some solubility in MEK.



Wait a minute. The amino acid dissolved completely in HCl!

Ok, we have a fairly important discovery.

The protein is soluble in acid. We also know that it dissolves in hot acid water.
This means that it should be polar and basic.

Histidine is staying still on that list.

Reacts to acids — The ATR crystal

CAUTION!

We believe that we have a
1. basic amino acid (histidine a strong candidate?)

and a
2. non polar amino acid (proline a strong candidate?)

I am seeing that we have complete control of precipitation of the protein complex via pH adjustment.

We can avoid the acid on ATR by precipitation of the protein.

Page 81

On Proteins:

You have ~~3~~4 approaches established thus far

1. ATR w/ acid removed?

2. Isoelectric precipitation, ie pH control

3. Electrochemistry Possibilities

4. Edvotek Labs.

I would like to work w/ GC.
There is no actual need w/GC until you get a non polar column. For now it is only GC skill development and modeling.

It starts w/ a homologous series.
But what if you don't have this?

GC Gas Experiment

Propane is C_3H_8

Butane is C_4H_{10}

Methane is CH_4

100°C 6 ml min

Butane Sample					$\bar{X}$
	O_2, N_2	~~0.34 m~~	.33 m	0.33	.33 m
	Propane	~~1.56 m (?)~~	2.53	2.52	2.52 m
	Butane	~~5.25 m. (?)~~	5.20	5.19	5.20 m
	CO_2	~~1.15~~	0.97?	1.02	1.00 m

Propane Sample Much Cleaner Sample		
O_2	0.31	
CO_2	0.96	
Propane	2.25	
Butane	4.71	
Pentane	6.44?	

Breath Sample O_2 .31
(Good Sample) CO_2 1.11
? CO 2.58? why?
Should be ? NH_3 6.56? why?

Human Breath - Sample 2
O_2 0.31 m
CO_2 1.10 m
③? 2.58? Why, what? CO?
NO? ④? 6.56? Why, What? Should be ammonia.

Car Exhaust:
0.31
1.09
2.58
6.55
Same as Human!

What an interesting topic
trace gases of human breath
and diseases

Jun 29 2016

a broad GC peak can mean more than one component.

Eg N_2 & NH_3 are overlapping.

We have learned some important things today already.

1. NH_3, ammonia, is overlapping w/ N_2 as well as O_2 because the thermo conductivity is so similar.

2. This means that I was NOT detecting ammonia in my breath OR in the car. I almost certainly am ~~detecting~~ NO, or nitric oxide. This may be raising some health concerns.

3. We know now that the high temp thermometer is off by -4 degrees. This must be taken into account especially when determining compound structure.

4. When you are examining gases, there is a good chance that water vapor is a part of the story. This means that you must always clean out your column when you are done with the session. The peak of H_2O is rather identifiable.

Page 84

5. Question: Can you examine NH_3 on IR ATR?

6. You have adjusted the flow rate to 5 ml/min. Every temperature setting requires an adjustment but you can use to ratios.

7. The low reading thermometer is right on.

Now @ 100°C 5 ml/min

There is a difference between fresh f breath and old breath!

Today you only have N_2, O_2, & CO_2 in your breath. You are still not sure what the other gases were last night but I believe that you were breathing propane & butane and that it was contaminating the sample.

My sample today is as clean as can be. No explanation really.
We did pick up a very small peak @ ~9 min
And now we know that this is CO. Almost imperceptible.
Human breath CEC Ratio factor = .430/.310 = 1.39

~~N_2, O_2 .430m~~ 1.10
~~CO_2 ~1.7m (1.39 · 2.58)~~
~~CO ~9.0m (1.39 · 6.56)~~

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$_2O_2$	.43	(1.39 · .31) = 0.43	$O_2 N_2$
O_2	~1.7	(1.39 · 1.10) = 1.53	CO_2
	~3.3	(1.39 · 2.58) = 3.59	CO, NO?, NH_3
	~9.0	(1.39 · 6.56) = 9.12	CO, NO?, NH_3

—

Fractional Distillation is performing flawlessly. I had a couple of leaks to begin with but all is sealed now & delicate.

You are separating 2 isopropanol @ 78.6°C

+2° elevation correction = 80.6°C vs theoretical 82°C. It is perfect what you are doing. A question is can you separate rainwater contaminants between 90° & 102°C?

$$\text{Density} \cong \frac{49.78\text{gms} - 33.45\text{gms}}{\approx 20\text{ml}} = \frac{16.33\text{gms}}{20\text{ml}} = \frac{.82\text{gms}}{1\text{ml}}$$

$$\text{Density} \cong 0.82\text{gms/ml vs } .\frac{79\text{gms}}{\text{ml}}$$

Not bad.

26.4 Brix = 1.373

Residual solution measures @ 97.9° + 2° = 99.7°C vs 100°C for water.

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Now look @ organic data base

BP ≅ 80.6°C = 81°C

Density ≅ 0.82 gms/ml

IOR = 1.313

1st on list: 2-propanol!

Exactly what it is.

IOR = 1.377

BP = 82°C

Density = 0.78 gm/ml

Perfect job.

Then a good work

Jun 30 2016 Page 87

Let's get organized for today :

1. We are learning about Gases in GC and how they behave. Transition to solutions.

We have the prospect of creating a model as well - this could be very interesting.

2. We have a question whether rainfall can be separated further by distillation.

3. We want to get into the protein in more than one way. We must protect the IR crystal. We already know that we have isoelectric precipitation as a mechanism of control.

4. You have the Edvotek Labs

5. You have DNA electrophoresis

6. You have the Demise paper

7. You have the relational chart.

Page 88

W.r.t. breath analyses, you have learned that all appear to be in range @ this point. Apparently you were actually breathing the very gases that you were testing.

We have data on:

100°C 6 ml/min

Factor		t	$\bar{X}$		5 ml/min
1.32	O_2, N_2, NH_3	.31	.41	Air, Ammonia	.410 .403, .410
1.57	CO_2	.96	1.51	Air, Breath	1.50 ~~1.014~~ 1.51, 1.52
Est 2.07	Propane	2.25	$\hat{4}.65$	Canister Propane	
Est 3.01	Butane	4.71	14.19	Canister Butane	
Est 3.60	? Pentane ?	6.44	23.70	Canister Propane	

Car Exhaust:

.31	N_2, O_2
1.09	CO_2
2.58	NO, CO?
6.55	NO, CO?

Expected Relationship: $\text{Factor} = 0.385\, t_{old} + 1.20$

Lets boost to 120°C. It will take too long otherwise.

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120°C Propane Entered   ACE Hardware Canister
Very clean signal coming in.

				Peak Ht	%
	0.44		O_2, N_2, (NH_3)	114.2	37.4%
	1.05		CO_2	1.68	0.5
C_3H_8	2.07	alkane	Propane	184.1	60.3
C_3H_6	3.95	alkene	Propylene	5.05	1.6
C_4H_8	5.29	alkane	~~Butane~~ NO! Butylene	.13	.04%
				Σ = 305.16	

C_4H_{10} Butane: (Ronson Brand)

0.44	O_2, N_2 (NH_3)	57.65	27.5
1.15	CO_2	1.07	0.6
2.33	Propane (?)	6.32	3.37
4.38	Butane yes	128.6	68.5
		Σ = 187.64	

Car Exhaust:

			%
0.43		583.8	96.1
1.17	38,000 PPM ~~38 K PPM~~	23.02	3.79
2.44	420 PPM	.254	.042%
5.44	629 PPM	.382	.063%
		Σ = 607.46	

X		Y
C_3H_8	Propane	~2.28
C_4H_{10}	Butane	~4.38

$Y = -5.74 + 7.30 \ln(n)$

for n = 5, Y = 6.01

C_5H_{12} Pentane estimate = 6.01 m

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We have a gas @ ~5.36 m that's occurring @ ~500 PPM in both Propane Canister and Car exhaust.

Our measured gas seems a bit too low for pentane ~~estimate~~.

Lets find out what gases are in propane.
Lets create CO and measure it.

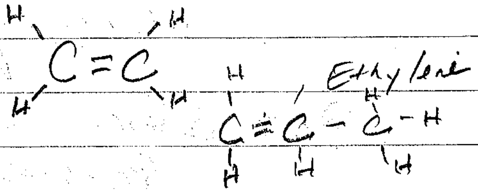
Propane commonly has in it:

Ethene C_2H_4

Propene Propylene C_3H_6

Butane

Ethyl Mercaptan (odorant)



We see from the ACE Hardware MSDS that ~~the~~ ~~main~~ a 2nd ingredient is propylene (propene)!

NOT PROPANE! Even though it has that title.
It also has butane in it.

Composition of ACE Propane:

Propane > 90%

Propylene < 5%

Butane < 2.5%

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Benzomatic Butane. We have:

0.42	O_2, N_2 (NH_3)	143.2	50.6%
1.09	CO_2	2.14	0.75%
2.34	Propane	13.23	4.7%
4.56	Butane	99.97	35.3%
5.65	double peak (strong tailing)	24.46	8.64%
		Σ = 283.0	

I would suspect this should be C_4H_8

which should be butene, or butylene.

Now for Ace Propane again. It has 6 gases in the sample w/ two double overlapping peaks.

0.41	O_2, N_2, (NH_3)	265.1	59.0%
1.16	CO_2	9.37	2.09%
1.63	(Could be ethene). Double trailing peak ?	~~9.34~~ ~~167.4~~ 1.11	2.5%
2.10	(Some trailing) Propane	167.4	37.27
~~3.03~~ 3.92	Propylene	4.71 | 3.93	0.87
5.36	Butylene ~~Butane~~ NO!	1.74	0.39%
6.16	Double trailing peak Possible Pentane.	0.51	0.11%
		Σ = 449.16	

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Our gases for our model are therefore

O$_2$
N$_2$
CO$_2$
Propane
Butane
Propylene

w/ possible addition of

ethene
propylene
pentane
] all highly questionable

Our data should be
1. BP
2. Thermal Conductivity @ 120°C
3. Carbon No.

So collect the data

ln(tr)		~~BP~~ VP(1kPa)	choose 300°K TC	Carbon No
67 (.42)	O$_2$	-211.9	26.3	0
67 (.42)	N$_2$	-236	26.0	0
3 ~~1.09~~ (1.12)	CO$_2$	-159.1	16.8	1
50 (2.34)	Propane	-156.9	18.0	3
679 (5.36)	Butane	-134.3	16.4	4
369 3.93	Propylene	-160.6	14.67	3

TC table is on p 6-188

VP table is on p 6-66

Page 94

Vapor pressure instead of boiling point

$\log(t_r) = C_1 \cdot VP + C_2 \cdot TC + C_3 \cdot CN$

$\ln(t_r) \cong -2.816E{-3} \cdot VP - 7.536E{-2} \cdot TC + 0.499\,CN + .452$

$R^2 \cong \underline{0.991}$!!!

or

$t_r \cong e^{(-.0028 \cdot VP - .0754 \cdot TC + .499 \cdot CN + 0.452)}$

eg CO_2 results in $t_r = 1.14$ minutes vs 1.13 meas.

This is therefore a superb first level model.

Let's try pentane VP = -115.5 TC = 14.4 CN = 5

$t_r \cong 8.\underline{89}$ min this actually looks reasonable.

4.89
5.94

You can solve each variable linearly for first ~~estimates: eg $\ln(t_r) = a(TC) + b$~~

~~$\ln(T_r) \cong -4.18 \cdot TC + 21.3$~~

~~or~~

~~$t_r \cong e^{-4.18 \cdot TC + 21.3}$ $r^2 = .81$~~

~~or $TC \cong \frac{\ln(t_r) - 21.3}{-4.18}$~~ ~~$VP \cong \frac{\ln(t_r) + 188.6}{31.88}$ $r^2 = .82$~~

~~$CN = \frac{\ln(t_r) - 1.25}{1.53}$~~ $r^2 = .96$

Page 95

Each variable solved for independently.

Carbon Number.

$\ln(t_r) = 1.53 \cdot CN + 1.25$ $r^2 = .96$

$\exists t_r \cong 1.76$ if $CN = 3$

Example:

$CN \cong \left(\frac{\ln(t_r) - 1.25}{1.53}\right)$

~~$\overset{x}{\ln(t_r)}$ $\overset{y}{CN}$ $CN \cong 0.63 \cdot \ln(t_r) - .77$~~

Why are we having an issue here —

Perform individual regression.

$\ln(t_r) = -4.18\,TC + 21.3$ $r^2 = .81$

$\ln(t_r) = .026\,VP + 4.92$ $r^2 = .82$

$\ln(t_r) = 0.63\,CN - .77$ $r^2 = .96$

Page 96

We need the inverse of these functions.

$$t_r = e^{-4.18TC + 21.3} = e^{-4.18TC} \cdot e^{21.3} \qquad 3^{2+3} = 3^2 \cdot 3^3. \qquad 243 = 20$$

$$t_r = 1780215035\, e^{-4.18TC}$$

$$e^{-4.18TC} = \frac{tr}{1780215035}$$

$$-4.18TC = \ln(tr) - \ln(1780215035)$$

TC $$\boxed{TC \cong \cancel{\ln(tr)}\ \frac{21.3 - \ln(tr)}{4.18}}$$

eg $tr = 2.34 \Rightarrow TC = 4.89$ vs 2.34

then

VP $$\boxed{VP \cong \frac{4.92 - \ln(tr)}{-.026} \quad \boxed{= \frac{\ln(tr) - 4.92}{.026}}}$$

eg $tr = 2.34 \Rightarrow VP = -156.5$ vs -156.9 excellent

CN ~~tr~~ $$CN \cong \frac{-.77 - \ln(tr)}{-.63} = \frac{.77 + \ln(tr)}{.63}$$

eg $tr = 2.34$ $CN = 2.6$ vs $C = 3$

These are excellent first estimates. With multiple regression

$$\tilde{t_r} = e^{(-.0028(-156.9) - .0154(4.89) + 0.499(2.6) + .452)}$$

$tr \cong 6.17m$ vs 2.34 not that good.

Page 97

You could weight the function differently

you could weight by ~~r^3~~ $(r^2)^3$

eg $r^2 = .81$ then $w = .53$

to do this we have

$$tr \cong e^{\left[\frac{\left(-r_1^3(.0028)(-156.9) - r_2^3(.0754)(4.89) + r_3^3(.499)(2.6)\right)}{r_1^3 + r_2^3 + r_3^3} + .452\right]}$$

So

$$r = e^{\left[\frac{\left(-.53(.0028)(-156.9) - .55(.0754)(4.89) + .88(.499)(2.6)\right)}{1.96} + .452\right]}$$

$$tr = e^{.598 + .452} = 2.86 \text{ vs } 2.34$$

This is excellent.

We will use this method.

Page 98

Charcoal Briquets Pyrolysis GC@120°C 5ml/m
~200°C ~~220~~°C

			Peak Ht	%			%
O_2, N_2	0.41		536.7	93.4%	0.41	518.7	91.%
CO_2	1.15		36.9	6.4	1.16	30.4	8.8
	2.40	Mystery	.33	.06	2.41	.15	.03
C_3H_6	4.10	Propylene	.32	.06	4.12	.24	.04
C_4H_{10}	5.48	Butane	.11	,02	5.44	.09	.02
C_5H_{12}?	11.31	possible Pentane?	.08	.01% (140PPM)	11.0	.04	.01
			Σ = 574.4			Σ = 569.62	

Carbon Monoxide - Predict it
Notice 11.31 is a very broad shallow peak

0.43
1.13
2.41
4.08
5.45
10.4 Possible
17.8 Possible
22.5 Possible

a filter applied dont help.
eg
Savitzky Order 3
Filter width 30
Iterations 20

Pese 99

We have a fresh sample now.
It was heated cleanly to red hot on the tube.

O_2, N_2, NH_3 0.42 O_2, N_2
CO_2 1.12 CO_2
C_3H_8 2.09 dominant Propane

2.38 secondary peak
This was one of the mysteries.
There are two Components

C_3H_6 4.06 Propylene
(4.56 Butane)
(C_4H_{10})
C_4H_8 5.50 This has a long trail which is suspect for an additional attached Component. Butylene?

9.05 possible

11.6 possible

We believe we have the case of

Ratio to O_2				Car Exhaust
	.42	O_2, N_2, NH_3		0.43
2.67	1.12	CO_2		1.17
4.90	2.09	Propane C_3H_8		
		unknown	2.38	2.44
[illegible]	4.06	Propylene C_3H_6		
	4.56	Butane C_4H_{10}		
	5.50	Butylene C_4H_8		5.44
		unknown?	9.05?	
		unknown?	11.6?	

Atmosphere pressure 15.~100 KPA not ~~100~~ 1KPA

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GC Model Work

6-66
6-188

Compute: Unknowns are

2.38

NO 9.05(?)

CO 11.6(?)

Pentane

Redevelop the model

Candidate	tr	ln(tr)	(1KPa) VP	mW/mK TC	CN
		Y	X	X	X
O_2	.42	-.867	-183.1	26.3	0
CO_2	1.12	.113	-78.6	16.8	1
C_3H_8	2.09 (propane)	.737	-42.3	18.0	3
C_3H_6	4.06 (propylene)	1.401	-47.9	14.67	3
C_4H_{10}	4.56 (butane)	1.517	-0.8	16.4	4
C_4H_8	5.50 (butylene)	1.705	-6.6	14.50	4
N_2	.42	-.867	-195.9	260	0
NH_3	.42	-.867	-33.6	24.4	0

$R^2 = 0.990$ superb design.

$\ln(t_r) \cong -.0011\,VP - .082\,TC + .446\,CN + 1.054$

Now individual regressions $VP = \frac{\ln(t_r) - 1.18}{.011}$ $VP = \left(= \frac{1.18 - \ln(t_r)}{-.011} \right)$

$.011 \cdot VP + 1.18$

$\ln(t_r) = \cancel{50.21 \cdot VP + 91.6}$ $r^2 = .56$ $r^3 = .18$ or $VP = \cancel{\frac{91.6 - \ln(t_r)}{50.21}}$

$-0.21\,TC + 4.46$ $TC = \frac{4.46 - \ln(t_r)}{0.21}$

$\ln(t_r) = \cancel{4.20\,TC + 21.1}$ $r^2 = .88$ $r^3 = .68$ or $TC = \cancel{\frac{21.1 - \ln(t_r)}{4.20}}$

$0.61\,CN - 0.79$

$\ln(t_r) = \cancel{1.57\,CN + 1.31}$ $r^2 = .96$ $r^3 = .88$ or $CN = \cancel{\frac{1.31 - \ln(t_r)}{1.57}}$

$CN = \frac{-.79 - \ln(t_r)}{-.61} = \left(\frac{\ln(t_r) + .79}{.61}\right)$

$CN = \frac{\ln(t_r) + 0.79}{0.61}$

GC Model work.

Using appropriate weights, our model is therefore:

$$tr \simeq e^{\left[\left(\frac{(-.0011)(.18)VP - (.082)(.68)TC + (.446)(.88)CN}{1.74}\right) + 1.054\right]}$$

and the individual regression estimates are:

$VP \simeq$ ~~$1.18 - \ln(tr)$~~ $\frac{\ln(tr) - 1.18}{.011}$

$$TC \simeq \frac{4.46 - \ln(tr)}{0.21}$$

$$CN \simeq \frac{\ln(tr) + 0.79}{0.61}$$

Page 102

Lets analyze ~~NH_3 &~~ CO & NO

	VP	TC	CN	estimated tr
CO	-191.7	25.0	1	1.64m
NO	-151.9	25.9	0	1.27m

CO is estimated to fall in between CO_2 & Propane

NO also, but closer to CO_2

Our Car peak could easily be Co therefore @ 2.38min
(our secondary peak)
also in briquets.

Isopropanol does not work well @ all.

Possibly 120 min detected w/ 180°D
Very poor broad peak. Do not do this again.

July 03 2014   Page 103

Another very important discovery today.

Recently it has been an awareness of the importance of the phosphorous issue. If the 'hackers' have it, then it comes to them @ our expense.

P - [illegible] Ca - Mg balance required. Bns, numbness, fatigue, etc

But for today, we have a dominant signature within the lipids of

DEHP

Bis(2-ethylhexyl) phthalate
"thă late"

or close to it

and all "that this entails", which is a lot.
Plasticizers, Biological Transformation,
Glucuronidation, etc

Page 104

We actually seem to have a respite here.

1. You have a handle on the lipids now
2. You have some information on the proteins
3. You have information on the rainfall agencies
4. You see the overlap between rainfall & microbiology

5. Env Element data remains upfront

6. NHFC remains paramount now Relational Diagram next.

7. Demise of rainwater is waiting for lesser contributions now.

8. You have developed some good distillation skills

9. You might fire up Column Chromatography again but you would prefer to get a non polar Column & work w/GC.

10. You have made an interesting GC model for gases & non polar injections

11. You have established isoelectric precipitation as a means of control over the proteins.

12. You might be able to apply electrochemistry to the proteins. You also have Edvotek labs.

Page 105

13. You can start working w/ DNA & gel electrophoresis @ the right point.

14. You can now analyze IR plots much more effectively w/ the NIST IR database.

15. Pyrolysis & head space methods developed.

16. HEPA air filter analysis

17. Public samples

18. Davis & Organic Chemistry

Dipole Moments are in 9-44 molecules

15-14 to 15-10 solvent

Page 106

On GC skills, you would like to find a volatile.

We learned that isopropanol does not work well @ all.

Next we proceed to acetone.
Then acetone w/ tea tree oil?

Let's predict

isopropanol & acetone w/ our model

C_3H_8O C_3H_6O

VP = 96.9 55.7
NC = 3 3
TC = .154 = 154 mW .161 = 161 mW
Dipole Moment 1.58 2.88 u/D
to estimate: 1.71m 1.72

and obviously this did not work. So you must be missing something big and I would ~~take~~ it to be polarity. What's the polarity of a gas?

Many gases are non-polar, such as hydrogen helium, Oxygen, CO_2 & nitrogen.

log P is lipophilicity.

VP ~~T~~°C 6-66 9-44 Dipole Moment
6-180-190 15-14 to 18 Solvents

Page 107

Dipole Moment

Water	~~1.86~~ 1.85
Oxygen (O_2)	0
N_2	0
CO_2	0
C_3H_8	.084
C_3H_6	.366
C_4H_{10}	.132
C_4H_8	.438
NH_3	1.49
Acetone	2.88
Isopropanol	1.58

~~$r^2 = .995$ still suspect.~~
$r^2 = 0.909$

tr=

C_4H_8O

For kick, lets predict MEK: Methyl Ethyl Ketone

are actually
measuring temp

			use r^2	r^3
VP =	79.6 °C	$\ln(tr) = .021\,VP + 2.27$	$r^2 = .72$	.39
TC =	~~.145~~ 145.	$\ln(tr) = .034\,TC - 0.27$	$r^2 = .75$	.42
CN =	4	$\ln(tr) = 0.92\,CN - 0.59$	$r^2 = .44$	.08
DP =	~~4.385~~ 1.96	$\ln(tr) = 1.70\,DP + .16$	$r^2 = .51$	

$\Sigma = 2.42$

Pese 108

You can not weight the way that you are trying to

Our current GC model is therefore.

$$\hat{tr} \approx e^{\left[\left(\overset{0.11}{(.0020)\cancel{(.72)}}VP^{oc} + \underset{\cancel{2.42}}{\underline{\overset{3.26}{(.030)\cancel{(.75)}}TC + \overset{1.09}{(0.62)}}}\cancel{(.44)}(CN) - \overset{.13}{(.13)}\cancel{(.51)}DP\right) - 1.20\right]}$$

and the individual regression estimates are (~~need to~~ invert all regression on previous page)

Check these

$$VP \approx \frac{\ln(tr) - 2.27}{.021}$$

$$TC \approx \frac{\ln(tr) + 0.27}{.034}$$

$$CN \approx \underline{\ln(tr) + .59}$$

$$DP \approx \frac{\ln(tr) - .16}{1.70}$$

These now need to be revised.

MEK = 253 m OK!

MEK

~~tr = 0.47~~ I do not believe this.

1.81m

So the problem was in your weighting.

You are now on the right track, this is essentially expected time on the column. You had a problem you can not weight it that way, the coefficients are already weighted.

VP 6-66
TC 6-188-190
15-14 to 18
DP 9-44

Page 109

C_8H_{10}

Now let's try to predict xylene

$$\hat{t_r} = e^{((.002)VP + (.03)TC + (0.62)CN - (.13)DP - 1.20)}$$

Xylene

VP: = 144
TC: = 130 mW
CN: = 8
DP: = .60

$\hat{t_r} \approx 26$ Min

The Carbon number is dominating the results. You need to get xylene into the regression.

We can choose about 50 min @ 180°
so @ 120 it will only be longer.

Choose about 90 minutes & try get into the regression $\ln(90 min) = 4.50$

Ok, now the regression model is changing

$$\hat{t_r} \approx e^{(.0036VP + .021TC + .32CN + .31DP - .51)}$$

$r^2 = 0.93$

Now for xylene estimate

$t_r \approx 240$ min, not too bad, it is looking better. This would be about right @ 120°C ~~as~~ since it takes about 60 min @ 180°C

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This page allows performing multiple linear regressions (multilinear regressions, multiple linear least squares fittings). For the relation between several variables, it finds the linear function that best fits a given set of data points. The result can have a small -usually insignificant- deviation from optimality, but usually it is very good and further improvement possibilities are very small. In the case that the number of variables is equal to the number of data points an hyperplane containing all the points results.

- **Copy & Paste:** You can copy and paste data directly from a spreadsheet or a tabulated data file in the box below. Any character that cannot be part of a number -space, comma, tabulation...- is considered a column separator. By default commas are considered column separators; in the case you are using them as decimal separators check the option below. The exponent can be indicated by preceding it by the character E or e, as you can see in the example. Data must consist of $n+1$ columns, $x_1 \ldots x_n$ and y, to get the multiple linear regression $y=a_1x_1+a_2x_2+\ldots+a_{n-1}x_{n-1}+a_nx_n+b$.

Example:

```
1.794638 15.15426 5.10998918E-
1
3.220726 229.6516 105.6583692
5.780040 3.480201e+3 1.77699E3
```

```
-183.1 26.3 0 0 -.867
-78.6 16.8 1 0 .113
-42.3 18.0 3 0.084 .737
-47.9 14.67 3 .366 1.401
-0.8 16.4 4 0.132 1.517
-6.6 14.50 4 0.438 1.705
-195.9 26.0 0 0 -.867
-33.6 24.4 0 1.49 -.867
96.9 154 3 1.58 5.29
55.7 161 3 2.88 5.29
```

-Now Available-

RegressionTools: The Program

☐ Allow comma as decimal separator

Result: $y = 3.656496377\cdot10^{-3}\,x_1 + 2.13280581\cdot10^{-2}\,x_2 + 3.248503644\cdot10^{-1}\,x_3 + 3.091450898\cdot10^{-1}\,x_4 - 5.074040541\cdot10^{-1}$

Residual Sum of Squares: **rss = 4.187340123**
Coefficient of Determination: $R^2 = 9.264122201\cdot10^{-1}$

- **Insert manually & See details:** If you prefer you can insert all the points manually, for which you first have to specify the number of data points. You also can see details of the calculation -as the calculated value of *y* and the error at each point- in this area.

Enter the number of data points: 11

Select the number of variables: 5

	VP x_1	TC x_2	CN x_3	DP x_4	ln(tr) y	Calculated y	Error
1.	-183.1	26.3	0	0	-.867	$-6.159806127\cdot10^{-1}$	$2.510193873\cdot10^{-1}$
2.	-78.6	16.8	1	0	.113	$-1.116429289\cdot10^{-1}$	$2.246429289\cdot10^{-1}$
3.	-42.3	18.0	3	0.084	.737	$7.223504756\cdot10^{-1}$	$1.464952437\cdot10^{-2}$
4.	-47.9	14.67	3	.366	1.401	$7.180305778\cdot10^{-1}$	$6.829694222\cdot10^{-1}$
5.	-0.8	16.4	4	0.132	1.517	1.179659511	0.337340489
6.	-6.6	14.50	4	0.438	1.705	1.212526919	$4.924730809\cdot10^{-1}$
7.	-195.9	26.0	0	0	-.867	$-6.691821838\cdot10^{-1}$	$1.978178162\cdot10^{-1}$
8.	-33.6	24.4	0	1.49	-.867	0.350768469	1.217768469
9.	96.9	154	3	1.58	5.29	4.594431727	$6.955682729\cdot10^{-1}$
10.	55.7	161	3	2.88	5.29	~~4.9949691~~	~~$2.950309002\cdot10^{-1}$~~
11.	144	130	8	0.6	4.50	5.576068946	1.076068946

Result: $y = 3.656496377\cdot10^{-3}\,x_1 + 2.13280581\cdot10^{-2}\,x_2 + 3.248503644\cdot10^{-1}\,x_3 + 3.091450898\cdot10^{-1}\,x_4 - 5.074040541\cdot10^{-1}$

Residual Sum of Squares: **rss = 4.187340123**
Coefficient of Determination: $R^2 = 9.264122201\cdot10^{-1}$

-Now Available-

RegressionTools: The Program

NHFC Prep   Page 111

Some very good work today.

The relational diagram is in place now.

The next step is to create the outline

& then pick the exploratory papers
that support that outline.
You are on the right track. Get them
in place before you leave town.

What is next for shortime tonight?

Your model is attached to their work.

If the time is projected to be too long,
you raise the temperature

Δ30°C is apparently roughly a factor
of 2 w.r.t. time.

Page 1/2

Topics:

1. Match the spectra of interest w/ ~~NITS~~ NIST
   database

2. For NHFC, bring 2.5 meter
   email of body donation
   flow chart or outline

   Static web site

July 05 2016

1. Some things going on today.

I am working on getting a non polar column to be able to separate polar compounds. There is some confusion w/ SRI so I am trying to work that through.

2. You are learning that nothing is dissolving the Env. filament easily.

Your assessment is that
1. The exterior coating is dissolved fairly easily w/ KOH & microwave.
This might mean similar w/ a keratin shell.
Try the same on hair.

You get some readout on IR with this solution after evaporation and reduction to a powder, but it is a very weak spectrum.

3. Now, working w/ the evaporated powder (after scraping from the evaporation dish) ~~that~~ looks to be a difficult matter.

(Ethanol) Alcohol
Acetone
MEK
Xylene
KOH
] all seem to fail miserably.

There does however, seem to be a very limited degree of success w/ heated & concentrated HCl. There is some color introduced into the solution. My solvent also contains ethanol (denatured alcohol)

So acidic ethanol heated seems to be our strongest option thus far.

The analysis indicates that the exterior of the filament has an acid aspect to it and that the interior has a basic component to it.

4. The problem w/ getting something into an acid is that you cannot put it on the ATR. Also the water is not good for the KCl IR crystals. Acids & water create problems in IR.

So somehow we need to remove the water and neutralize the acid.

5. We have learned that the TCD in the GC should not be run above 150°C. We have been baking out @ 100°C. We need to bake out @ a max of 150.

6. Acid base extraction is the name of the game today along w/ proceeding to ATR! There is an acid base precipitation factor also involved in the env. filament analysis underway.

Important methods here.

Env Filament "Internal" Spectrum

We appear to have ONE & ONLY ONE internal spectrum of the env filaments.

It was very hard to achieve. The steps to accomplish it, best as I can remember are;

1. The external portion of the filament is handled in the following way

   1. Filament was placed in a watch glass with several drops of 1M KOH. Microwave @ absolutely lowest heat possible (~~magneto~~ magneto is active for 5 secs about every 30 secs) for ~ 2 min.

   2. The resulting solution is evaporated under mild heat so the majority till just about 30 - 50 ul is left. Transfer 20 ul to ATR IR w/ a background set of ATR water. You will get a very weak spectrum but some signal and information will be extractable.

3. The internal portion of the filament is handled in the following manner (there is the remaining ~~insolub~~ insoluble material within the KOH evaporative process. You scrape that material into a powder form for further processing.

4. For now we take the powder and ~~[illegible]~~ place it in ethanol (denatured alcohol) overnight. This may not be required (ie the time) as it does not seem to have much effect.

5. Next we add Conc (8.7M) HCl to this ethanol solution and heat to about 60°C. Sustain the heat for up to an hour. There will be some color change. We used ~5 drops Conc HCl for in 1 ml of ethanol.

6. Now the material (Conc acid) is highly damaging to the ATR so it must be neutralized. pH meter upside down a 1M HCl can be used. Get the pH in mid range. You will see some precipitation take place when the pH gets too high (past the isoelectric point). Maintain the pH just below the point. We estimate the pH here will be about 4.5, ~~[illegible]~~ much higher than it was for the Conc acid.

7. Evaporate this to ~ 40 ul & transfer 20 ul to ATR w/ slide. Watch the signal that you can. There is one, but it is weak and difficult. The background is ATR - water.
We will call this the "~~external~~ internal" env filament spectrum.

8. The internal spectrum does not match anything well, Amanda's sample is the closest.

Back to GC regression model.

Individual case:

~~$\ln(tr) = 1.66VP + 0.49$~~ $r^2 = 0.4$

X_1 List 5 = Polarity

X_2 List 4 CN

X_3 List 3 TC

X_4 List 2 VP

(y) List 1 $\ln(tr)$

$\ln(tr) = 0.02VP + 2.16$ $r^2 = .76$

$\ln(tr) = .035TC - 0.20$ $r^2 = .79$

$\ln(tr) = .10\,CN - .22$ $r^2 = .49$

$\ln(tr) = 1.66\,\text{Polarity} + 0.49$ $r^2 = .41$

~~$tr = e^{(.02VP + 2.16)}$~~

$VP \cong \frac{\ln(tr) - 2.16}{.02}$ $TC \cong \frac{\ln(tr) + 0.20}{.035}$

$CN \cong \frac{\ln(tr) + \overset{.22}{\cancel{.28}}}{\cancel{.035}\,.10}$ DP (dipolemoment) $\cong \frac{\ln(tr) - .49}{1.66}$

Jul 06 2016 Page 119

Iron Content of CDB

Stainless Steel Crucible: 11.06 gms

w/ CDB 11.23

Diff 0.17 gms

after Torch. 11.18

$\Delta =$ $\begin{array}{r} 11.23 \\ -11.18 \\ \hline = .05 \end{array}$

gms
thru organic combustion

$\frac{.05}{.17} =$ 29.4% Organic ~30% Organics

$\frac{.12}{.17} =$ 70.6% Iron ~70% Inorganics
or Organics

CDB Pyrolysis

We also have several peaks.

Partial matches include

Cyanoacetylene $H-C\equiv C\equiv N$

NIST 1300 cm^{-1}] Strong match
665 cm^{-1}]

2260 cm^{-1} proximity match

Tribromo methane

NIST 670 cm^{-1} Strong match

3046 proximity ~~match~~ match

Page 121

Jul 07 2016 QTip & Stem Trial.

Pyrolysis testing in progress.
It is going superbly well. Using a Q Tip & Stem.

If I can get a QTip into both IR & GC
(and I can!)
I am doing something right. Maybe even have
potential down the road.

Height	I see:	
253	0.42	
176	0.48	We have a double peak here. (weak)
19 mV	1.04	a double peak (weak)
52.2	1.19	primary peak
1.1	1.93	
.09	2.28	
1.32	3.66	
.10	9.70	
.82	11.79	

This shows 9 different compounds in the Q tip after pyrolysis minus O_2, N_2 & CO_2

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We have on second trial:

Peak Height mV			$\bar{X}$
400	0.42	(w/tail indicates 2nd Component)	0.42
25	1.09	secondary peak (weak)	1.06
31.1	1.21		1.20
.09	1.95		1.94
.38	2.31		2.29
9.0	3.71		3.68
.28	9.93		9.82
1.23	11.92		11.85

The set under quick pyrolysis gave even better results. Results are repeatable.

Let's form 1st estimate of VP, TC, CN, DP

$VP \cong \frac{\ln(tr)}{.02} - 2.16$

$\overset{TC}{\cancel{CN}} \cong \frac{\ln(tr)}{.035} + 0.28$

$\overset{CN}{\cancel{TC}} \cong \frac{\ln(tr)}{\cancel{.035}\ .70} + \overset{.22}{\cancel{0.28}}$

$DP = \frac{\ln(tr)}{1.66} - .49$

Page 123

Now for Proteins Pyrolysis : IR & GC

Peak Height		
597.6	0.41	N_2O_2
56.3	1.00	CO_2
0.26	1.94	Propane (?) C_3H_8
0.38	3.75	Propylene (?) C_3H_6
.06	11.09	Pentane (?) C_5H_{12}
.03	25.1 possible	?

QTip : 1st Estimates of $\overset{VP,}{TC}$, CN & DP

meas t_r	$\hat{VP}$	$\hat{TC}$	$\hat{CN}$	$\hat{DP}$
.42	-151	-17	-1.0 (0)	-.83 (0)
1.06	-105	+10	+0.4 (0)	-.25 (0)
1.20	-99	+13	+0.6 (1)	-.18 (0)
1.94	-75	+27	+1.3 (1)	+.10
2.29	-67	+32	+1.5 (1,2)	+.20
3.68	-43	+45	+2.2 (2)	+.49
9.82	+6	+73	+3.6 (4)	+1.08
11.85	+16	+79	+3.8 (4)	+1.19

VP 6-66
TC 6-188, 190
15-14, 18
DP 9-44

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Protein Pyrolysis Estimates for VP, TC, CN & DP

$\hat{tr}$		$\hat{VP}$	$\hat{TC}$	$\hat{CN}$	$\hat{DP}$
	r^2	.76	.79	.49	.41
		100KPA	300°K		
	meas. tr				
	0.41	-153	-17	-1.0 (0)	-.83 (0
	1.08	-104	+10	0.4 (0)	-.25 (0)
	1.94	-75	27	+1.3 (1)	0.10
	3.75	-42	46	+2.2 (2)	0.50
17.4m	11.89	+16	79	+3.0 (4)	+1.20
49m	25.1(?)	+53	100	+4.9 (5)	+1.65

We can now form estimates for $\hat{tr}$ to compare against meas tr

$$\hat{t}_r \approx e^{(.0036VP+.021TC+.32CN+.31DP-.51)}$$

Not bad, somehow you seek the closest match.
There is no combined data base of course.
But you do have general properties that have been predicted.

Page 125

What we are doing here is making the information more manageable so that you can visualize the relationships.

We are breaking the problem into 2 separate regressions:

1. tr = f (VP & TC mg)

2. tr = f (CN & DP mg)

VP = vapor pressure (°C @ 100 kPA)

TC = Thermal Conductivity in mW/mK

CN = Carbon number

DP = Dipole Moment (Polarity) in U/D

We can also form individual regression for each V variable and then use the 4 variable regression to predict the associated tr.

We can also use tr graphically with 2 separate regression to arrive @ a first estimate of the four variable.

This is all a bit crude it is fair to say but it is much better than nothing.

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CN & DP only as variables

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Example:

```
1.794638 15.15426 5.10998918E-
1
3.220726 229.6516 105.6583692
5.780040 3.480201e+3 1.77699E3
```

```
0 0 -.867
1 0 .113
3 0.084 .737
3 .366 1.401
4 0.132 1.517
4 0.438 1.705
0 0 -.867
0 1.49 -.867
3 1.58 5.29
3 2.88 5.29
```

-Now Available-

RegressionTools: The Program

☐ Allow comma as decimal separator

Result: $\mathbf{y = 6.544604159\cdot10^{-1}\,x_1 + 1.522209093\,x_2 - 1.140952263}$

Residual Sum of Squares: **rss = 9.269222535**

Coefficient of Determination: $\mathbf{R^2 = 8.371038685\cdot10^{-1}}$

- **Insert manually & See details:** If you prefer you can insert all the points manually, for which you first have to specify the number of data points. You also can see details of the calculation -as the calculated value of *y* and the error at each point- in this area.

Enter the number of data points: 11

Select the number of variables: 3

	CN x_1	DP x_2	ln (tr) y	**Calculated y**	**Error**
1.	0	0	-.867	-1.140952263	$2.739522631\cdot10^{-1}$
2.	1	0	.113	$-4.864918472\cdot10^{-1}$	$5.994918472\cdot10^{-1}$
3.	3	0.084	.737	$9.502945484\cdot10^{-1}$	$2.132945484\cdot10^{-1}$
4.	3	.366	1.401	1.379557513	$2.144248741\cdot10^{-2}$
5.	4	0.132	1.517	1.677821001	$1.608210008\cdot10^{-1}$
6.	4	0.438	1.705	2.143616983	$4.386169832\cdot10^{-1}$
7.	0	0	-.867	-1.140952263	$2.739522631\cdot10^{-1}$
8.	0	1.49	-.867	1.127139285	1.994139285
9.	3	1.58	5.29	3.227519351	2.062480649
10.	3	2.88	5.29	5.206391172	$8.360882794\cdot10^{-2}$
11.	8	0.6	4.50	5.00805652	$5.080565198\cdot10^{-1}$

CN DP

Result: $\mathbf{y = 6.544604159\cdot10^{-1}\,x_1 + 1.522209093\,x_2 - 1.140952263}$

Residual Sum of Squares: **rss = 9.269222535**

Coefficient of Determination: $\mathbf{R^2 = 8.371038685\cdot10^{-1}}$

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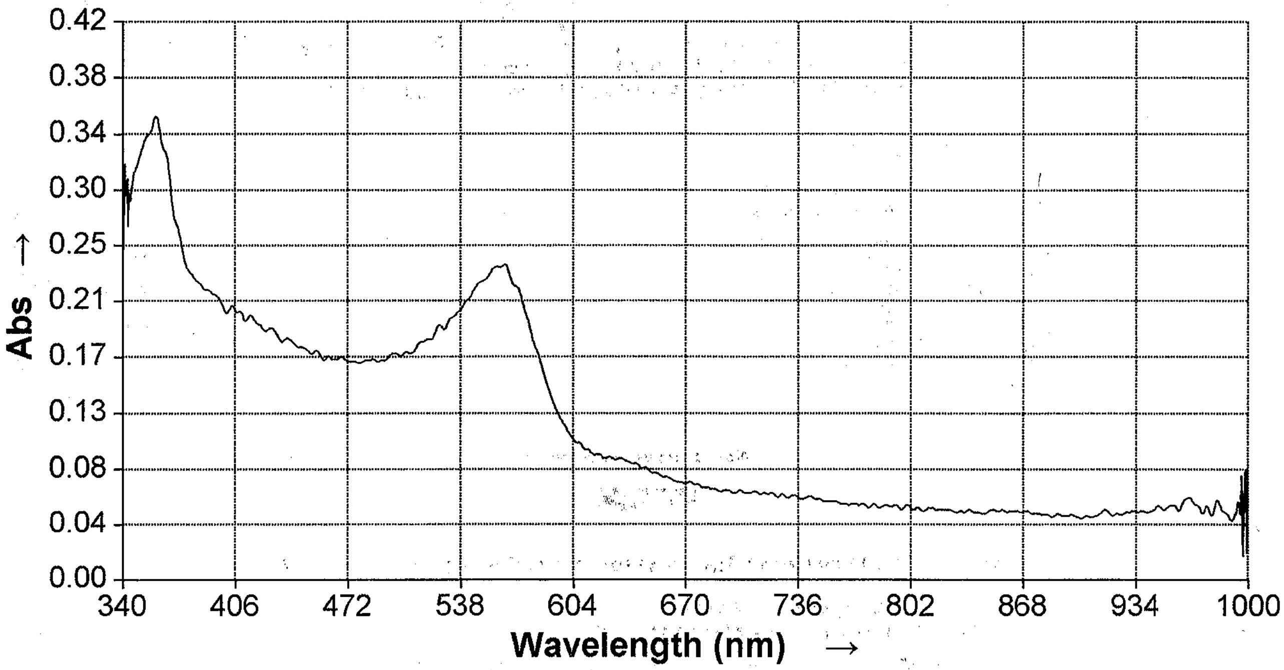
SPECTRONIC 200

Scan report

Spectrum of :	Rain Heated Alkaline3	Analysis date :	22 - May - 2016
Analyzed by :	CI	Analysis time :	6:42:14 PM
Channel # :	3	Print date :	22 - May - 2016
		Print time :	6:43:08 PM



VP & TC only as variables.

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Online Multiple Linear Regression

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This page allows performing multiple linear regressions (multilinear regressions, multiple linear least squares fittings). For the relation between several variables, it finds the linear function that best fits a given set of data points. The result can have a small -usually insignificant- deviation from optimality, but usually it is very good and further improvement possibilities are very small. In the case that the number of variables is equal to the number of data points an hyperplane containing all the points results.

• **Copy & Paste:** You can copy and paste data directly from a spreadsheet or a tabulated data file in the box below. Any character that cannot be part of a number -space, comma, tabulation...- is considered a column separator. By default commas are considered column separators; in the case you are using them as decimal separators check the option below. The exponent can be indicated by preceding it by the character E or e, as you can see in the example. Data must consist of $n+1$ columns, $x_1 \ldots x_n$ and y, to get the multiple linear regression $y=a_1x_1+a_2x_2+\ldots+a_{n-1}x_{n-1}+a_nx_n+b$.

Example:

```
1.794638 15.15426 5.10998918E-
1
3.220726 229.6516 105.6583692
5.780040 3.480201e+3 1.77699E3
```

```
-183.1 26.3  -.867
-78.6 16.8  .113
-42.3 18.0  .737
-47.9 14.67  1.401
-0.8 16.4  1.517
-6.6 14.50  1.705
-195.9 26.0 -.867
-33.6 24.4 -.867
96.9 154  5.29
55.7 161  5.29
```

-Now Available-

RegressionTools: The Program

☐ Allow comma as decimal separator

Result: $\mathbf{y = 1.110338026 \cdot 10^{-2}\, x_1 + 2.138957862 \cdot 10^{-2}\, x_2 + 7.562167378 \cdot 10^{-1}}$

Residual Sum of Squares: **rss = 5.264875336**

Coefficient of Determination: $\mathbf{R^2 = 9.074757541 \cdot 10^{-1}}$

This is tr = f(TC & VP only)

• **Insert manually & See details:** If you prefer you can insert all the points manually, for which you first have to specify the number of data points. You also can see details of the calculation -as the calculated value of *y* and the error at each point- in this area.

Enter the number of data points: 11

Select the number of variables: 3

	VP x_1	TC x_2	ln(tr) y	**Calculated y**	**Error**
1.	-183.1	26.3	-.867	$-7.142662707 \cdot 10^{-1}$	$1.527337293 \cdot 10^{-1}$
2.	-78.6	16.8	.113	$2.428359699 \cdot 10^{-1}$	$1.298359699 \cdot 10^{-1}$
3.	-42.3	18.0	.737	$6.715561677 \cdot 10^{-1}$	$6.544383226 \cdot 10^{-2}$
4.	-47.9	14.67	1.401	$5.381499415 \cdot 10^{-1}$	$8.628500585 \cdot 10^{-1}$
5.	-0.8	16.4	1.517	1.098123123	$4.188768771 \cdot 10^{-1}$
6.	-6.6	14.50	1.705	0.993083318	0.711916682
7.	-195.9	26.0	-.867	$-8.628064117 \cdot 10^{-1}$	$4.193588304 \cdot 10^{-3}$
8.	-33.6	24.4	-.867	$9.050488792 \cdot 10^{-1}$	1.772048879
9.	96.9	154	5.29	5.126129392	$1.638706079 \cdot 10^{-1}$
10.	55.7	161	5.29	4.818397176	$4.716028244 \cdot 10^{-1}$
11.	144	130	4.50	5.135748716	$6.357487157 \cdot 10^{-1}$

VP TC

Result: $\mathbf{y = 1.110338026 \cdot 10^{-2}\, x_1 + 2.138957862 \cdot 10^{-2}\, x_2 + 7.562167378 \cdot 10^{-1}}$

Residual Sum of Squares: **rss = 5.264875336**

Coefficient of Determination: $\mathbf{R^2 = 9.074757541 \cdot 10^{-1}}$

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TABLE 3

Reduction Reactions Having $E°$ Values More Negative than that of the Standard Hydrogen Electrode (continued)

Reaction	$E°$/V	Reaction	$E°$/V
$Am^{2+} + 2e \rightleftharpoons Am$	–1.9	$ZrO(OH)_2 + H_2O + 4e \rightleftharpoons Zr + 4OH^-$	–2.36
$Pa^{4+} + e \rightleftharpoons Pa^{3+}$	–1.9	$Mg^{2+} + 2e \rightleftharpoons Mg$	–2.372
$Es^{3+} + 3e \rightleftharpoons Es$	–1.91	$Y^{3+} + 3e \rightleftharpoons Y$	–2.372
$Cf^{3+} + 3e \rightleftharpoons Cf$	–1.94	$La^{3+} + 3e \rightleftharpoons La$	–2.379
$Lr^{3+} + 3e \rightleftharpoons Lr$	–1.96	$Tm^{2+} + 2e \rightleftharpoons Tm$	–2.4
$Eu^{3+} + 3e \rightleftharpoons Eu$	–1.991	$Md^{2+} + 2e \rightleftharpoons Md$	–2.40
$Er^{2+} + 2e \rightleftharpoons Er$	–2.0	$Th(OH)_4 + 4e \rightleftharpoons Th + 4OH^-$	–2.48
$Pr^{2+} + 2e \rightleftharpoons Pr$	–2.0	$HfO(OH)_2 + H_2O + 4e \rightleftharpoons Hf + 4OH^-$	–2.50
$Pu^{3+} + 3e \rightleftharpoons Pu$	–2.031	$No^{2+} + 2e \rightleftharpoons No$	–2.50
$Cm^{3+} + 3e \rightleftharpoons Cm$	–2.04	$Dy^{3+} + e \rightleftharpoons Dy^{2+}$	–2.6
$Am^{3+} + 3e \rightleftharpoons Am$	–2.048	$Pm^{3+} + e \rightleftharpoons Pm^{2+}$	–2.6
$AlF_6^{3-} + 3e \rightleftharpoons Al + 6F^-$	–2.069	$Be_2O_3^{2-} + 3H_2O + 4e \rightleftharpoons 2Be + 6OH^-$	–2.63
$Sc^{3+} + 3e \rightleftharpoons Sc$	–2.077	$Sm^{2+} + 2e \rightleftharpoons Sm$	–2.68
$Ho^{2+} + 2e \rightleftharpoons Ho$	–2.1	$Mg(OH)_2 + 2e \rightleftharpoons Mg + 2OH^-$	–2.690
$Nd^{2+} + 2e \rightleftharpoons Nd$	–2.1	$Nd^{3+} + e \rightleftharpoons Nd^{2+}$	–2.7
$Cf^{2+} + 2e \rightleftharpoons Cf$	–2.12	$Mg^+ + e \rightleftharpoons Mg$	–2.70
$Yb^{3+} + 3e \rightleftharpoons Yb$	–2.19	$Na^+ + e \rightleftharpoons Na$	–2.71
$Ac^{3+} + 3e \rightleftharpoons Ac$	–2.20	$Yb^{2+} + 2e \rightleftharpoons Yb$	–2.76
$Dy^{2+} + 2e \rightleftharpoons Dy$	–2.2	$Bk^{3+} + e \rightleftharpoons Bk^{2+}$	–2.8
$Tm^{3+} + e \rightleftharpoons Tm^{2+}$	–2.2	$Ho^{3+} + e \rightleftharpoons Ho^{2+}$	–2.8
$Pm^{2+} + 2e \rightleftharpoons Pm$	–2.2	$Ra^{2+} + 2e \rightleftharpoons Ra$	–2.8
$Es^{2+} + 2e \rightleftharpoons Es$	–2.23	$Eu^{2+} + 2e \rightleftharpoons Eu$	–2.812
$H_2 + 2e \rightleftharpoons 2H^-$	–2.23	$Ca^{2+} + 2e \rightleftharpoons Ca$	–2.868
$Gd^{3+} + 3e \rightleftharpoons Gd$	–2.279	$Sr(OH)_2 + 2e \rightleftharpoons Sr + 2OH^-$	–2.88
$Tb^{3+} + 3e \rightleftharpoons Tb$	–2.28	? $Sr^{2+} + 2e \rightleftharpoons Sr$	–2.89
$Lu^{3+} + 3e \rightleftharpoons Lu$	–2.28	$Fr^+ + e \rightleftharpoons Fr$	–2.9
$Dy^{3+} + 3e \rightleftharpoons Dy$	–2.295	$La(OH)_3 + 3e \rightleftharpoons La + 3OH^-$	–2.90
$Am^{3+} + e \rightleftharpoons Am^{2+}$	–2.3	$Ba^{2+} + 2e \rightleftharpoons Ba$	–2.912
$Fm^{2+} + 2e \rightleftharpoons Fm$	–2.30	$K^+ + e \rightleftharpoons K$	–2.931
$Pm^{3+} + 3e \rightleftharpoons Pm$	–2.30	$Rb^+ + e \rightleftharpoons Rb$	–2.98
$Sm^{3+} + 3e \rightleftharpoons Sm$	–2.304	$Ba(OH)_2 + 2e \rightleftharpoons Ba + 2OH^-$	–2.99
$Al(OH)_3 + 3e \rightleftharpoons Al + 3OH^-$	–2.31	$Er^{3+} + e \rightleftharpoons Er^{2+}$	–3.0
$Tm^{3+} + 3e \rightleftharpoons Tm$	–2.319	$Ca(OH)_2 + 2e \rightleftharpoons Ca + 2OH^-$	–3.02
$Nd^{3+} + 3e \rightleftharpoons Nd$	–2.323	$Cs^+ + e \rightleftharpoons Cs$	–3.026
$Al(OH)_4^- + 3e \rightleftharpoons Al + 4OH^-$	–2.328	? $Li^+ + e \rightleftharpoons Li$	–3.0401
$H_2AlO_3^- + H_2O + 3e \rightleftharpoons Al + 4OH^-$	–2.33	$3N_2 + 2H^+ + 2e \rightleftharpoons 2HN_3$	–3.09
$Ho^{3+} + 3e \rightleftharpoons Ho$	–2.33	$Pr^{3+} + e \rightleftharpoons Pr^{2+}$	–3.1
$Er^{3+} + 3e \rightleftharpoons Er$	–2.331	$Ca^+ + e \rightleftharpoons Ca$	–3.80
$Ce^{3+} + 3e \rightleftharpoons Ce$	–2.336	$Sr^+ + e \rightleftharpoons Sr$	–4.10
$Pr^{3+} + 3e \rightleftharpoons Pr$	–2.353		

from Sigma Aldrich

Hayesep D should be useful for:

290°C max

gases
acids NO ON TCD!
amines
low carbon number organics
water
volatile organic compounds
inert & hydrophobic
polar, low molecular weight compounds, such as
methanol
ethanol
non polar volatile organic compounds, such as
benzene
toluene

This sounds perfect.

But ACIDS & halogens attack
& damage a TCD. So NO ACIDS!

Working w/ an acetone - denatured alcohol mix.

This should have
acetone
methanol
ethanol

We now have a 1st peak @ ~ 9 min
Water still seem to be a problem.
But we could never do this before.

Now we are getting a second peak @ ~ 11 min

Even though it has major tailing, there are two definite peaks between 8-12 min.
This is good.
Peaks look good over a long time interval.

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July 07 2016

We have the new column in and it is working after a couple of trials w/ the gas flow.

We are getting some results but we also continually see the advantage of pyrolysis over liquids.

MEK injected has not produced a definite result yet @ 180°C for almost 40 min.

We have overloaded a pyrolysis injection of a rubber eraser w/ relatively modest heat and we have many gaseous components immediately @ 180°C. Flow rate = 6 min.

7 already @ 19 min.
Therefore gases are working very well.

The 14 min + (30 min) peak could be MEK since it is so much larger than #5 & #6

So now we are @ 180° vs 120°C
6 ml/min vs 5 ml/min

Let's record and repeat.

Peak Ht	tr 180°C 5ml/min
294	.356 (O_2, N_2)
18.3	.636
2.14	1.19
0.55	2.57
.06	6.12
.05	7.83
.21	15.22

No results on the second trial. Why? Incorrect injection? The needle is plugged!

Something happened here that is odd but then I got ~~room~~ air ~~into~~ balloon balloon air working again fine.

Balloon breath air has 3 peaks

Peak Ht			adj	
661	O_2, N_2	.346	.40	98.9%
7.06	CO_2	0.93	1.08	1.0%
0.13	?	3.12	3.62	.02% ≅ 200PPM
Σ=668.19			(Each ×1.16)	

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Now Ace Propane. 120' 6ml

Very clean peak Coming in.

I have 5 Clean peaks. Very clean signal

Peak Ht	
6 ~~0.34~~	.35
1.52	.82
226	1.55
6.5	2.99
.38	4.09

Jul 8 2016   80°-140°-200°   60m

We saw something very strange taking place.

1. Human breath is working fine w/ O_2 & CO_2
2. ACE Propane I believe is working fine
3. Pyrolysis of charcoal & rubber does not seem to be working @ all – why?

The pyrolysis gases appear to be too heavy to be getting through the column @ lower temperatures and are displacing O_2 & CO_2? This does not seem possible.

With propane from ACE we are also getting very good separation.

The question is why is pyrolysis failing? Vaccuum created?

Propane is C_3H_8

Peak Ht   Ratioed

0.37 O_2 ~10.4 ?

~1.3 CO_2 vs ~1.0   29m ? very broad peak

3.46   Propane?   Huge broad multiple peak.

8.63   ?

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You have a very broad multiple peak that is taking place.

It looks to be a minimum of 3 components. Is this fallout from MEK, ethanol, acetone work?

We will keep cleaning house until we figure it out. In the meantime do not keep adding liquids until this clears out.

We have @ least 3 components in this broad peak.

This means we have @ least 8 components that are passing through the column now. We are set now for a 4 hr additional run.

You could keep cleaning this out gradually and continue to overlay propane until you see repeatability. This would avoid an excessive run.

O_2	.37	
CO_2	~~1.3~~ 1.29	
Propane	~~3.46~~ 3.44	
Unknown	~~8.85~~ 8.82 Ramping	Might need to change to 10 mins
Unknown	10.5 definite	

You keep getting what appears to be broad peak formation of the 10.5 minutes.

Is the broad peak part of the ramping effect or is it a real component(s)?

If it was a ramp effect it would not rise and decline, which it has.

Baking the filter has dropped the peak out of the system almost immediately and seems to have established a new reference point that is considerably lower. Negative Territory —

After baking it has risen up immediately

Bake it again to see the effect

Baking is definitely having an impact.

The column is all over the place.

—

Good news, the column is showing itself to be cleaning up.

Repeat Program again.

We have set up a better temperature program

12 min @ 80°
12 min @ 140
remainder @ 200° to 60 min total.

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We need a dry run w/out anything added.

then
Butane by itself

then Propane & Butane mixed.

Your program can be reset @ 25 min

We can see what is happening now.
Ramping is causing broad peak distortion.

Small peaks can (and are) superimposed
on the ramp peaks.

In this case they are of higher molecular weight.

80 - 140 - 200
(12) (12) (15)

We have

0.30	O_2, N_2
1.29	CO_2
3.47	Propane C_3H_8
9.30	
37.01	

Now Ronson Butane

The easy way to get butane into the syringe is to stick the syringe needle into the valve and let the propane - butane fill up the syringe under pressure.

$\ln(tr) = C_1 \cdot CN + C_2$

X Y Y X

$\ln(tr)$ $CN_i \; H_{2n+2}$ based upon Propane C_3, C_4 Butane

$\ln(tr) = 0.986 \cdot CN - 1.71$

if $CN = 5$ $\ln(tr) = 3.22$ $tr = 25.03$ Pentane Estimate
Our point is 37.01
This suggest pentene vs pentane
But you are ramped. This compresses the graph.

Merge together both gases when you are done.

I have learned now how to create a gas mixture with a balloon.

1. Fill small balloon partial w/ propane, leave slack stem
2. Pressurize syringe w/ butane
3. Inject back into balloon

Now it is getting very interesting. We mix:

1. Two different brands of propane
2. Two different brands of butane
3. Fairly light charcoal pyrolysis

We have

Peak Height						T°
35.1	20.9%	0.37-.38		O_2, N_2	O_2, N_2	
18.8	11.1%	1.32-1.30		CO_2	CO_2	80
	1.0%	1.66 CO?		CO?	CO?	80
107.5	63.9%	3.50 -3.6	w/ tail indicates second compound in region of 4.5	C_3H_8 C_3H_6	Propane Propene	80
1.34	0.8%	9.46-9.64			Unknown	80
4.49	2.7%	11.92-11.96		C_4H_{10} –	Butane	80
1.0	0.6%	13.96-13.93		C_4H_8	Butene?	140
168.25						

Rank is

64%	Propane
21%	O_2; N_2
11%	CO_2
3%	Butane
1%	CO(?)
1%	Unknown
1%	Butene

8 different gases in the balloon

Briquet project does not look so good.

Let's try water.

First, we have a very small, almost none existent air peak.

Then we have a very small CO_2 peak.

Very good baseline is active.

Baseline is perfect & flat @ 80°C

I have ramp up @ 12 min in.
I have a very clean ramp up.
Goes to ~ + 3mV. No water peak of any kind present thus far.
It is possible that some peaks have been masked by the ramp up process.

Very Very Cool. We have picked up the water peak
SHARP & SWEET!
@ ~ 30m @ beginning of ramp up from 140°C to 200°C
A very sharp entry.

This opens up many possibilities now.
The peak ht is about 65 mV. Good & solid.
There is some trailing but it is manageable.
The ramp effect could be creating the tail on the downward slope.

Next run @ 200°C isotherm.

2nd run @ 200°C isotherm.

No air peak to speak of.
No CO_2 peak.
There was no air bubble in the syringe this time.

We get a very strong peak @ a convenient time of approx 5 min.
It does have a fairly broad tail but it is descending.

We raise column to 220°C and add rainwater.

The peak has risen @ ~5 min and then diminished to the baseline by 20 min. Not bad!

No contaminant detected thus far.

Since the expansion volume is so high reduce the sample size even further.

You can run the blank filter to clean out also in addition to high temp in oven (220°)

We appear to have a peak in the sample @ ~41 min but this wasn't after a bakeout.

July 09 2016

Working w/ Rainwater again. 220°C 150
Reduced volume of injection to < 1/2 ul.
Peak came in sharp & strong @ 3.5 min @ 220°C

[redacted]

The broad tail might mean that components could be masked, but these can be determined by comparison w/ distilled water.

We can see that the column is very good @ detecting water. This is different than using a solvent of water.

1. What types of liquids or gases can be detected w/ Hayes SepD?

2. What types of solvents can be used w/ HepD?

66 min.
We see we have one very minor component @ ~ ~~68~~ min.
It has to be on the order of a few PPM.
We would assume that it is a higher C carbon such as C6.

We are learning some good things about the Hayes Column.

Hayes Sep D should be reasonably similar to Hayes Q. It is made of the same thing w/ similar polarity!

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TABLE 3
Reduction Reactions Having E° Values More Negative than that of the Standard Hydrogen Electrode (continued)

Reaction	E°/V
$Am^{2+} + 2e \rightleftharpoons Am$	–1.9
$Pa^{4+} + e \rightleftharpoons Pa^{3+}$	–1.9
$Es^{3+} + 3e \rightleftharpoons Es$	–1.91
$Cf^{3+} + 3e \rightleftharpoons Cf$	–1.94
$Lr^{3+} + 3e \rightleftharpoons Lr$	–1.96
$Eu^{3+} + 3e \rightleftharpoons Eu$	–1.991
$Er^{2+} + 2e \rightleftharpoons Er$	–2.0
$Pr^{2+} + 2e \rightleftharpoons Pr$	–2.0
$Pu^{3+} + 3e \rightleftharpoons Pu$	–2.031
$Cm^{3+} + 3e \rightleftharpoons Cm$	–2.04
$Am^{3+} + 3e \rightleftharpoons Am$	–2.048
$AlF_6^{3-} + 3e \rightleftharpoons Al + 6F^-$	–2.069
$Sc^{3+} + 3e \rightleftharpoons Sc$	–2.077
$Ho^{2+} + 2e \rightleftharpoons Ho$	–2.1
$Nd^{2+} + 2e \rightleftharpoons Nd$	–2.1
$Cf^{2+} + 2e \rightleftharpoons Cf$	–2.12
$Yb^{3+} + 3e \rightleftharpoons Yb$	–2.19
$Ac^{3+} + 3e \rightleftharpoons Ac$	–2.20
$Dy^{2+} + 2e \rightleftharpoons Dy$	–2.2
$Tm^{3+} + e \rightleftharpoons Tm^{2+}$	–2.2
$Pm^{2+} + 2e \rightleftharpoons Pm$	–2.2
$Es^{2+} + 2e \rightleftharpoons Es$	–2.23
$H_2 + 2e \rightleftharpoons 2H^-$	–2.23
$Gd^{3+} + 3e \rightleftharpoons Gd$	–2.279
$Tb^{3+} + 3e \rightleftharpoons Tb$	–2.28
$Lu^{3+} + 3e \rightleftharpoons Lu$	–2.28
$Dy^{3+} + 3e \rightleftharpoons Dy$	–2.295
$Am^{3+} + e \rightleftharpoons Am^{2+}$	–2.3
$Fm^{2+} + 2e \rightleftharpoons Fm$	–2.30
$Pm^{3+} + 3e \rightleftharpoons Pm$	–2.30
$Sm^{3+} + 3e \rightleftharpoons Sm$	–2.304
$Al(OH)_3 + 3e \rightleftharpoons Al + 3OH^-$	–2.31
$Tm^{3+} + 3e \rightleftharpoons Tm$	–2.319
$Nd^{3+} + 3e \rightleftharpoons Nd$	–2.323
$Al(OH)^- + 3e \rightleftharpoons Al + 4OH^-$	–2.328
$H_2AlO_3^- + H_2O + 3e \rightleftharpoons Al + 4OH^-$	–2.33
$Ho^{3+} + 3e \rightleftharpoons Ho$	–2.33
$Er^{3+} + 3e \rightleftharpoons Er$	–2.331
$Ce^{3+} + 3e \rightleftharpoons Ce$	–2.336
$Pr^{3+} + 3e \rightleftharpoons Pr$	–2.353
$ZrO(OH)_2 + H_2O + 4e \rightleftharpoons Zr + 4OH^-$	–2.36
$Mg^{2+} + 2e \rightleftharpoons Mg$	–2.372
$Y^{3+} + 3e \rightleftharpoons Y$	–2.372
$La^{3+} + 3e \rightleftharpoons La$	–2.379
$Tm^{2+} + 2e \rightleftharpoons Tm$	–2.4
$Md^{2+} + 2e \rightleftharpoons Md$	–2.40
$Th(OH)_4 + 4e \rightleftharpoons Th + 4OH^-$	–2.48
$HfO(OH)_2 + H_2O + 4e \rightleftharpoons Hf + 4OH^-$	–2.50
$No^{2+} + 2e \rightleftharpoons No$	–2.50
$Dy^{3+} + e \rightleftharpoons Dy^{2+}$	–2.6
$Pm^{3+} + e \rightleftharpoons Pm^{2+}$	–2.6
$Be_2O_3^{2-} + 3H_2O + 4e \rightleftharpoons 2Be + 6OH^-$	–2.63
$Sm^{2+} + 2e \rightleftharpoons Sm$	–2.68
$Mg(OH)_2 + 2e \rightleftharpoons Mg + 2OH^-$	–2.690
$Nd^{3+} + e \rightleftharpoons Nd^{2+}$	–2.7
$Mg^{+} + e \rightleftharpoons Mg$	–2.70
$Na^+ + e \rightleftharpoons Na$	–2.71
$Yb^{2+} + 2e \rightleftharpoons Yb$	–2.76
$Bk^{3+} + e \rightleftharpoons Bk^{2+}$	–2.8
$Ho^{3+} + e \rightleftharpoons Ho^{2+}$	–2.8
$Ra^{2+} + 2e \rightleftharpoons Ra$	–2.8
$Eu^{2+} + 2e \rightleftharpoons Eu$	–2.812
$Ca^{2+} + 2e \rightleftharpoons Ca$	–2.868
$Sr(OH)_2 + 2e \rightleftharpoons Sr + 2OH^-$	–2.88
? $Sr^{2+} + 2e \rightleftharpoons Sr$	–2.89
$Fr^+ + e \rightleftharpoons Fr$	–2.9
$La(OH)_3 + 3e \rightleftharpoons La + 3OH^-$	–2.90
$Ba^{2+} + 2e \rightleftharpoons Ba$	–2.912
$K^+ + e \rightleftharpoons K$	–2.931
$Rb^+ + e \rightleftharpoons Rb$	–2.98
$Ba(OH)_2 + 2e \rightleftharpoons Ba + 2OH^-$	–2.99
$Er^{3+} + e \rightleftharpoons Er^{2+}$	–3.0
$Ca(OH)_2 + 2e \rightleftharpoons Ca + 2OH^-$	–3.02
$Cs^+ + e \rightleftharpoons Cs$	–3.026
? $Li^+ + e \rightleftharpoons Li$	–3.0401
$3N_2 + 2H^+ + 2e \rightleftharpoons 2HN_3$	–3.09
$Pr^{3+} + e \rightleftharpoons Pr^{2+}$	–3.1
$Ca^+ + e \rightleftharpoons Ca$	–3.80
$Sr^+ + e \rightleftharpoons Sr$	–4.10

The first project is that I think that we should be able to separate water & alcohol
We see that it will still take a long time to clean the column and we think that other solvents should be easier to use.

It is better for detection than it is trying to use it as a solvent.

We are now testing 91% Isopropanol @ 220°C

We are getting lots of interesting activity.

We have:

1. a detectable air peak] no air bubble
2. a very small CO_2 peak] in syringe
3. A very broad immediate peak, which is the alcohol. This peak is masking the water but a minor slope break can be detected @ 3.5 min. This is the water.
4. We would now best be served by reducing the temp to 150°C and see if we can start to separate the water.
5. We now have another peak coming in @ 10 min. This could be where the water dominates the

column since it takes 20 min for the water to elute. 220°C

This way the water eventually comes in and allows a degree of separation.

We see now how the overlap of peaks affects the development of retention times.

You could drop the temp to 150° and get better separation but then it will lead to a longer bake out time, which is not what we want.

Now pure isopropanol should give a better peak.

This is a case when the response factor is hardly the same. Isopronal is much more responsive than water is. You must take this into account when determing concentrations.

This is actually very cool to see how this is working. It is clean in 30 min @ 220°C

Let's try pure isopropanol.
Remember to always use very little water in injections

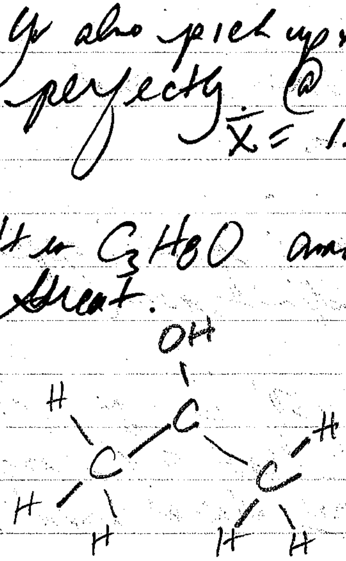
You have the peak and have ~~determent~~ ron just about right now.

220°C

You pick up the air peak perfectly.

You also pick up the isopropanol peak perfectly @ 1.84 min vs 1.80 min
$\bar{X}$ = 1.82 m @ 220°C.

It is C_3H_8O and you pick it up under 2 min.
Great.



Very very cool.

Now we are not confined to hydrocarbons non-polar.

Interesting how even the "pure" isopropanol also has water in it.
Is it possible that I filled the bottle w/ my 91% supply. I don't think so because it is yellow.

We are now running 90% Isoprop again but this time w/ a few drops of acetone added.

Isoprop peak occurs @ 1.91 ok
Slope break for water occurs @ 3.5 noticed before

The tail is not decending as quickly because the detector was not fully stabilized.

Page 144

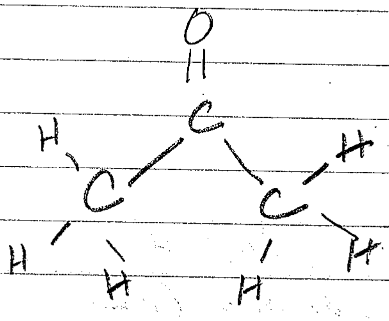
Ok, what we really want to do is test acetone by itself. 220°C

Acetone gives a weak ~~response~~ but it is detectable and it ~~finishes w/in~~ 3 min @ 220°C

Not true.

The small peak looks like it was CO_2 instead!

Guess What? I am seeing two major peaks.



is acetone.

Sunnyside says the solution is pure acetone.

Then how do we get two major peaks?

2.906

60 m = .1

10 min = .5

-.01 + .5

$.1 = 60 \cdot x$ $x = \frac{.1}{60}$

y = ~~-125x + 725~~

$\boxed{-.008 + .6}$ Print Formula

Inches/Min = -.008 * #min + 0.6

On 2nd pass, it appears that acetone failed. But it did not. 30 min in @ 220°C the peak appears.

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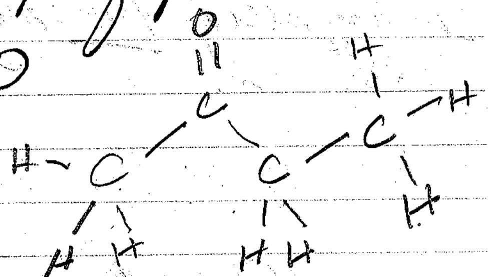
We now have some basic experience shaping up with Water, Isopropanol, and Acetone.

Also some of the nuances of mixing water and isopropanol.

Now, how do you start using this information?

What if you flipped into MEK?

C_4H_8O



Notice similarity to acetone but it has a higher boiling point, as you can imagine.

MEK dissolves polystyrene and many other plastics. It is a VOC

Now we are trying a practical problem: Separation using an extract of banana w/ 90% Isoprop

Here is a Case where you are working w/ a banana.
You make an extraction into acetone.
But in the process, water comes into the picture.
You don't want the water.

You also have the banana in 90% isoprop.
It actually looks quite good and clear.

You may have picked up two components in small quantity.

July 17 2016 @ Bear Creek.

Just maybe you have your peaks misidentified.

Look @ water by itself 220°C
Water came out @ ± 4.25 min

Look @ acetone by itself 220°C
Acetone came out @ 24-31 min

Now w/ 90% isopropanol, what makes you think water came out so late, especially w/ what you see w/ acetone. With isopropanol, maybe the first peak is water and the second is isopropanol!

you need to try a run like w/ distilled water and just a little bit of isopropanol added.

We are now thinking you will get a rapid large peak and then a subsequent smaller peak for isopropanol, maybe @ ~ 10 min.

[redacted]

July 24 2016

MRP Review [redacted]

1. Consent
2. Demographic
3. General Health
4. Skin & Nails
5. Head & Hair
6. Eyes & Vision
7. Ears & Hearing
8. Nose & Sinuses
9. Mouth & Throat
10. Cardiovascular
11. Respiratory
12. Musculoskeletal
13. Digestive System
14. Endocrine System
15. Neurological System
16. Cognitive & Psychological
17. Immune System
18. Reproductive System
19. "Morgellons" & Ass. Symptoms
20. Environmental
21. Free Response
22. Submit

pages		Questions	Full	Partial	Total	% Completion Ratio
2500 (5 reams)	Short	151	215	728	943	4.4
	Long (I)	814	204	706	910	4.5
30,000 (60 reams)	Long (II)	1031	34	76	110	3.2
	Σ	1996	453	1510	1963	

Page 149

—— 90%

#4 Hrs Skin & Nails

$\bar{x}$ 12.6

9.8 1.6449

9.77(16.1)

12.6 + 1.6449 (9.8) = 28.7

80% Skin

Page 150

Parametric stat tests w/Casio:
Determine Pr as a function of the z score

Use Distributions $\mu = 0$
$\sigma = 1$

put in $X = 1.6449$ you will get prob = .103

$1 - .103 = 0.90 = 90\%$ OK

Put in $z = 1$ $pr = 0.24$? I do not understand this

The ~~it~~ inverse method is working
prob = 0.9 $z = \pm 1.6449$
.68 $z = \pm 1.0$ OK
.80 = 80% $z = \pm 1.28$

OK, we have it. You need to use the Normal cumulative distribution.

So the upper 20% is $z = 1.28$

Now we can proceed.

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NO.	Topic	$\bar{X}$	σ_s	Count Pr(80%) (z=1.28)
1				
2				
3				
4	Skin & Nails	12.6	9.8	$12.6 + 1.28(9.8) = 25.1$
5	Hair	16.9	12.7	33.2
6	Eyes	26.8	15.4	46.5
7	Ears	22.3	16.0	42.8
8	Nose	23.9	14.5	42.5
9	Mouth	12	8.2	22.5
10	Cardio-Vascular	14.2	14.0	32.1
11	Respiratory	9.9	7.5	19.5
12	Muscle	11	13.4	28.2
13	Digestive	6.3	5.7	13.6
14	Endocrine	7.5	13.5	24.8
15	Neurological	6.8	7.6	16.5
16	Cognitive	5.8	7.3	15.1
17	Immune	7.6	7.7	17.4
18	Female	5.3	4.1	10.5
19	Associated Conditions	10.9	11.2	25.2
20	Urban, Rural			
21	Personal Comments			
22				

Page 152

Selecting Set	Top 20%
	4 Materials or substances emerging from the skin
	Open and/or slow healing lesions
	Rashes or other skin conditions
	5 Itchy Scalp
	6 Change in the quality of vision (eg blurry or fatigued)
	7 Unusual & Chronic Ringing in the ears
1, 3, 5	8 None
1	9 Unusual Dental Conditions
3	10 Fatigue
5	11 Shortness of breath, Persistent or excess mucus or sputum
None	12 Stiffness in Joints, Fatigue (2)
1	13 Constipation, Bloating, Unusual Wgt Gain, Extreme Fatigue (3)
4	14 Fatigue (4) Anxiety, Nervousness & Irritability
6, 12	15 Headaches, Dry Eyes & Mouth, Fatigued (5)
1, 4,	16 Forget Events
2, 5, 6, 21	Reliance on ext. memory aids (Calendars notes)
1, 2,	Loss of train of thought or flow of thread
1, 3, 9,	of Conversations
1, 2, 3,	17 Difficulty diagnosing, identifying or
2, 6, 9	explaining the illness
♀ 3, 4, 7	Skin Problems
1, 14, 18	Fatigue (6)
	~~18 ♀ Depression, Moodiness, Irritability, Anxiety~~
	19 Lyme, Chronic Fatigue (7), Herpes

MN Trip Planning – Preparation

Need graphics and outline
Need symptom list
Need to work through the flow –
List of Symptoms

July 21 2016

A little bit of time in the lab left.
Very much enjoy the GC work w/ the non polar column.

We have isopropanol and water figured out.
Obviously the response factor of water is much different than isopropanol.

Lets try a test w/ response factor
and compare to a known standard.

	Weight Factor
Water:	.55
Acetone	.68
Isopropanol	.71

Lets measure.

3 ml H_2O
.1 ~~.5 ml~~ ~~91~~90% Isopropanol   0.1 ml = x microliters
.1 ~~.5 ml~~ Acetone   0.1ml = 100 ~~ml~~ ul

This was too much Isop. for a good
separation. It should have been reduced to 50ul.

We can however, still estimate.

Pesc 156

By wgt we have

3ml(1gm) = 3gms
(100E-6)(.8gms/ml) = ~~.00008 gms~~ .08 gms

= 2.7%

3(.55) = 1.65 gms
.08(.71) = .057 gms   = 3.3%

.47% (.71) = .33%   Normalize   .60%
99.53 (.55) = 54.74%   100%

Actual is · 3ml H_2O = 3gms H_2O
.08 gms Isoprop   = 2.7% vs 0.60%

Not terrible
The actual area of water peak might be less
and that of isopropanol more.
We expect acetone near 27 min

Isopropanol could be a useful solvent since it has a
clean and relatively linear response.

Remember that 91% isoprop will give a strong
response by itself because of the water.

You should really distill pure alcohol.
You have this to some degree.

Page 157

Work on image for website

460 x 143

July 29 2017.

Last run on GC before MN.

How about just an air?

Room Air

~~Room~~
Breath w/ Tea Tree Oil?

Vapor of Tea Tree would be interesting.

Page 158

July 30 2016 On the train to MN!
Now east of Havre MT.

Want to study vectors, curl, gradient, dot prod & Maxwells equation.

Casio does handle vectors in the interactive mode.

Angle between 2 vectors. $[1,2],[3,4] = 10.30^\circ$
Yes, this is correct.

Vectors are entered as $([1,2],[3,4])$
or separately as $[1,2]$ in the interactive menu.
$[3,4]$

Action menu version is p 77
Interactive menu version is p 89

since $\vec{A}\cdot B = |A||B|\cos\theta$

$$\theta = \cos^{-1}\frac{\vec{A}\cdot\vec{B}}{|A||B|}$$

mag of A = 2.24
mag of B = 5

$$\vec{A}\cdot\vec{B} = 11 = \cancel{(1\cdot 2)}\ (1\cdot 3) + (2\cdot 4)$$

$$\theta = \cos^{-1}\left(\frac{11}{2.24\cdot 5}\right) = 10.8^\circ$$ so this is correct.

Now, what does the dot product mean?

$\vec{A}\cdot\vec{B}$ means a projection of A on the x axis. This is what it means. I don't think B has to be on the x axis. It can be anywhere.

Why do you want it?

Dot Product.

① So the reason for the dot product is that it gives you the angle between 2 vectors. This is geometric information of value.

This is really interesting.

This is a scalar

Dot Product $\vec{A} \cdot \vec{B} = |A||B| \cdot \cos\theta$

a $\vec{A} \cdot \vec{B} = |AB| \cdot \cos\theta$ No orientation is required!

and $\vec{A} \times \vec{B} = |AB| \cdot \sin\theta$ No orientation is required!

This is a VECTOR!

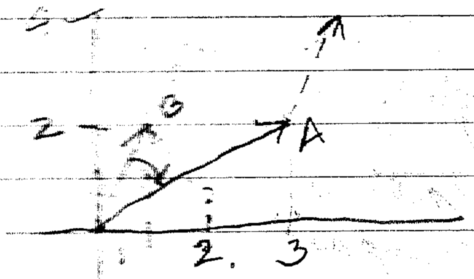
This is great, dot product & cross product have a direct geometric interpretation.

NO THEY ARE NOT EQUIVALENT!

Dot Product is a Scalar!
This is why it is also called a scalar product.

Cross Product is a Vector.
This is why it is also called a vector product

Page 160



θ = 29.74°

θ = 29.74

The length of B does not matter as long as the vector has the same orientation.

The dot product, therefore, is all about the angle between the two vectors.

The length of either vector, $\vec{A}$ or $\vec{B}$ does not matter. It is the orientation of the vectors that matter.

The cross product must form a ~~unit~~ vector ⊥ to both A & B plane.

$[3\ 2\ 0] \times [1\ 2\ 0] = [0, 0, 4]$

$[6, 4, 0] \times [1\ 2\ 0] = [0\ 0\ 8]$

$|A| = 3.60$
$|B| = 2.24$
$|A| = 7.21$
$|B| = 2.24$

and $(3.60)(2.24) \cdot \sin(29.74) = 4.00$!!
$(7.21)(2.24) \cdot \sin(29.74) = 8.00$

The magnitudes do matter.

Now go back to dot product.
Magnitudes matter here also, but the angle remains the same.

The dot product is a number. (2 dimensional space)
The cross product is a perpendicular vector (3 dimensional space)

Page 161

The seem to be a critical factor that is not really brought forth.

The Dot product is about an angle between two vectors in space.

It can exist in 2 dimensional space but it does not have to.
It can also exist in 3 dimensional space.

The Cross product is about a perpendicular vector. It requires 3 dimensional space and 3 dimensional vectors to all make any sense.

Notice that your cross product is $\perp$ to the xy axis.

Cross product is a determinate situation.

Now let's go on to the more interesting aspect of vectors

1. Gradient

2. Divergence

3. Curl

Page 162

Now what are these about?

1. The gradient is pretty straightforward.

$$\text{grad}\,\phi = \nabla\phi = \frac{\partial\phi}{\partial x} i + \frac{\partial\phi}{\partial y} j + \frac{\partial\phi}{\partial z} z$$

(del operator)

Now, how do you apply and visualize this? What's the difference between this and any regular function?

ϕ is a scalar function
grad ϕ or $\nabla\phi$ is a vector quantity.

Parametric equations are really very interesting
See prob 5.26 on p 136 Schaum, advanced.

—

We now know what the dot product and the cross products & how they visualize. Now let's visualize and demonstrate the

1. gradient
2. divergence
3. curl

Page 163

[redacted]

$\phi = f(x, y, z)$

$$\text{grad}\,\phi = \nabla\phi = \frac{\partial f}{\partial x} i + \frac{\partial f}{\partial y} j + \frac{\partial f}{\partial z} k$$

ϕ is not a vector function. It is just a function, ie, a surface.

I am not sure exactly how the grad differs from the partial derivative ~~stuff~~.

OK, the answer is that $\nabla\phi$ represents a normal to the surface. Not a unit vector normal, but a normal. This is if

$\phi(x,y,z) = c$, a constant.

It does not represent a normal otherwise.

So that seems to be the grad, or $\nabla\phi$

[redacted]

Ok, now the divergence.
This is certainly less obvious
We are talking now about a dot product.
But it is a dot product between
the del operator and a vector.
What is the visualization of this?

Notice on p121 the del operator, or $\nabla \phi$ specifically
make reference to the unit vector.

$$\text{So } \nabla = \frac{\partial \phi}{\partial x}\vec{i} + \frac{\partial \phi}{\partial y}\vec{j} + \frac{\partial \phi}{\partial z}\vec{k}$$

Where $\vec{i}, \vec{j}, \vec{k}$ are unit vectors. So this is
actually really important.

Now, the next most important statement is that
"the gradient of a scalar is a vector".
So you need to think about what the operator
actually means.

It means that the operator is creating a unit vector.
This is the purpose of it.
It is essentially a unit vector that is normal
to the surface.

Page 165

Divergence has finally been conceptualized and visualized And it took Menzel to do it on p 142.

Nobody else would do this, so no wonder that it has been lost over the years.

It all came from hydrodynamics, which was used to explain electromagnetic dynamics. It is basically force field mathematics, whether it be

water
air
gravity
electromagnetics] the same math can be used for all.

Menzel tries to give us the flow of thought.

1. First we created a "unit vector" normal to the surface. This is called the grad or del of Ø, an x,y,z space function.

Now remember this is a vector.

We can explore, with any two vectors
the dot product
and the cross product

since we are certainly "allowed" to do so:

Page 166

One case involve the Cosine of the angle between 2 vectors (ie, the dot product) and the other case involve the sin of the angle between the two vectors, and also the perpendicular vector!

Now, the next thing that happened was to get involved with hydrodynamic problems, as in p 141 of Menzel and guess what? We are going to be led into the divergence and the curl. The divergence itself is a unit gradient vector.

The $\cos\theta$ of the angle shows up in the fluid problem!
Fluid passing a cross section @ an angle leads to the dot product and a change in a vector field is involved so this led to a dot product involving the gradient.

The integral of the divergence across the cross section of a volume element represents the net flow of fluid through a volume element. This then shows you first practical application and visualization of the value of the divergence.

With electromagnetism it will be current instead of fluid, but it is the same idea!

Page 167

Now lets work toward the curl.

the Gradient is a vector
(a normal vector to a surface)
the Divergence is a number
(Integrate a gradient over a volume)
the Curl is a Vector
It is the determinant places.

Menzel did not tell exactly what the curl means, other than it has t "something" to do w/ rotation.

This was weak but it is better than nothing. We also know that it is a "rotation vector" of some type.

Now for kicks, lets look @

Intensity	vs	Volts/meter
Watts/m^2		V/m
$\frac{M^{1/2}}{L^{1/2} \cdot T \cdot k^{1/2}}$		$\frac{M^{1/2}}{L^{1/2} \cdot T \cdot k^{1/2}}$

So it is true, the units are the same. But there is a conversion ratio, not sure why yet

Page 168

OK, you have done much better now.

This last progress is not too bad!

Aug 08 2016 Back from MN NHFC Congress

Time to get organized again and reset the priorities for the final push of Aug-Sept.

1. Three paper are immediate need & maybe 4.

   OK ✓ 1. The GeoEngineering System View

   2. The Demise of Rainfall

   3. The Supplement paper

   Progress – Good 4. MRP Opening & Commenting Platform? Preliminary Stats

2. We have a lot of projects on tap.

   working it. 1. GC skill development, applications, trap & trap transfer to IR, Headspace, Pyrolysis

   2. DNA Project, no progress thus far

   be later 3. A new column for GC

   Study 4. UV ~~instrument~~ from Ocean Optic.?

   data available 5. IR overall assessment

   6. New pH meter

   7. Society registrations

   [illegible] 8. Distill Ethanol

Page 170

Here was our previous list from Meg:

1. DNA work
2. Env. Filament
3. COB, Proteins & Lipids work
4. Rainfall Writeup
5. IR Recovery, Consolidation, Database this is OK.
6. Electrophoresis

[redacted] Book

8. Hair Analysis & Pyrolysis
9. Davis, Org Chemistry
10. Citizen samples

Now we need to regroup on these entries and determine what we can reasonably accomplish.

We also want to develop the winter outfitting

Page 171

Winter Outfit.

1. Electrochemistry Setup w/ Palmsens
2. Oscilloscope
3. Impedance Meter
4. "Rife" Machine
5. ECA Meter
6. New pH meter
7. Refractometer
8. Ocean Optics UV Spec
9. Ionization meter
10. PM 2.5 meter
11. Chemistry Virtual Lab
12. IR Database & Viewing Software
13. UV-Vis Instrument?

Page 172

Distillation of Denatured Alcohol:

1. 1st discovery: It has acetone in it.

BP ≃ ~~62°C~~ 58°C

Actually a fair amount
A surprising amount, at least ≥ 5%

Actually you had just a little bit of acetone up front. Probably an impurity.

Now you are very steady. It is most certainly Methanol which has a BP of 65°C.
And we measure 65°C on the dot.
Ethanol will come later. Which one is poisonous?
Your temperature is perfect.

Methanol is the more dangerous of the two.
Usually den alcohol is about 10% methanol.
We are starting to pass that already. IOR = 63
= 1.341

Temp is now starting to rise. 66°C
Mine has a little bit of rubber dissolved in it.

IOR of Methanol actually = 1.329
This is less than water.

So you did not pick it up w/ IOR but it is close to water. You did pick it up right away with BP.

Page 173

You are now clearly running @ 67°C

and IOR is closer to 1.341

There is no longer methanol. What might it be?

Nothing is found w/ these attributes. So it must be a modified compound.

Rubber + Alcohol (Methanol) has probably created a new compound.

Notice also that it is clear.
The residue is falling out of solution.

—

As we get to the bottom of our flask,
the temp of distillation is indeed rising.
We are now up to 69°C from a reference of
65C. So something is certainly changing.

In GC, here comes the peak of distillate @ 65°C.
@ ~8.5 min.

Now it is up to 70°C

So far, we are seeing that there is a little
bit of acetone up front.

Acetone 58°C lives @ beginning

Then the bulk is methanol @ 65C

Then we start rising gradually
for 66 - 70°C

which tells me that we have some additional
compounds added a portion of what might be
ethanol but they might be more corrosive
than ethanol.

The container does not tell what chemicals
are added but it does say that chemicals are
added. This is what we are seeing.

It is not ethanol that is added. It is toxic
chemicals added to METHANOL.
The can has no significant level of ethanol
added to it. It is poisoned methanol.

We have very clean second generation
distillation taking place.

We picked up 2-3 ml of acetone @ the
beginning 58°C

We now have very pure methanol coming out @ 65°C

Let's look @ IOR for acetone solution. Brix = 13.6
BP = 58°C ⇒ IOR = 1.352

NOT TRUE

Actual BP = 56°C.

IT IS THERE

We learn that acetone is NOT in the
organic compound database from Colby!
This is a big surprise.

Page 175

Not true. Use the database properly.
Acetone shows up @ the top of the list.
w/ BP only.

Actual IOR is 1.359

We get 1.352

Error is .007 vs .005 tolerated.

The database should be adjustable.
Flex the IOR ± .005 from measured
to isolate candidates
you zero in right on acetone

	BP Meas	BP Actual	IOR Meas	IOR Actual
Acetone	58°C	56°C	1.352	1.359

This is superb. you need UV spectroscope.
It has a peak @ 270 nm

You easily can peg acetone here.

After Double Distillation, we clearly have four different levels of separation.

1. Acetone ~0.5% Relatively pure and easily identifiable w/ the organic database.
   1. Boiling point
   2. IOR (±.007 vs .005)
   3. UV spec is coming up.

2. Clean " Double distilled methanol.
   ~50% of the total volume.
   BP = 65°C
   also easily identified.
   Might check IOR again.

3. "Dirty Methanol". Various Contaminants.
   But may be added to the methanol.
   BP = 66-71°C

4. Rubber (most likely) Contamination.
   Restricted to approx 10ml, ie < 10% of total volume.
   Most likely will analyze material.
   Basically the components of a rubber stopper are to be identified. Anticipate hydro carbon and also a that component that gives rubber its flexibility.

In addition we have a very clean chromatogram of methanol.

The double distilled run is much cleaner than the second run.

There is a secondary component showing up. We anticipate that this could be accentuated within the "dirty" methanol.
Now to be called the "Cleaning Methanol".

I think that centrifuging & IR of the 4th material would be of interest.

Very interesting. Centrifugation does not cause separation. This means that the material is in solution.

There is no water present. This could be a candidate for both IR & GC.

This is potentially very interesting and a good demonstration of lab separation methods.

IR would start with the clean & double distilled as well as the cleaning methanol.

Watch out for acid on ATR.

We could obviously evaporate the
solution on an IR cyrstal

Pure Methanol Retention w@ 8.0 min.
Double Distilled

Single distillation was @ 10.2 min.

Aug 07 2016

This is getting very interesting. We are involved w/ a real analysis and separation of "denatured" alcohol.

We are learning what this actually means. Many toxic components are added to methanol to make it undrinkable.

We see that some acetone is added up front. 2nd generation distillation is doing a great job of purifying.

We have some contaminants from the rubber stopper that we are analyzing using VIS & NIR.

Color means conjugation.
Yellow means less conjugation but still significant.

970 peak indicates Ar-OH.
This makes perfect sense.
This suggests a phenol compound.

We have therefore learned some important attributes from VIS & NIR alone.

This is before GC & IR.

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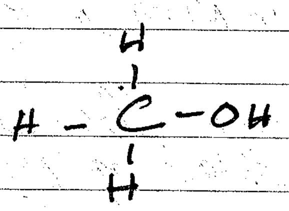
We see now that we blanked it incorrectly. We should have used the double distilled methanol as the blank as we are trying to learn what is in the methanol.

We should not have used methanol as the blank. Having done so, incorrectly, w/ H_2O as the reference we see the NIR existence of

880-920 Suggests CH_3 756 CH
CH_2
ArCH
ROH ← This gets eliminated

Now we know that there is Methanol and a hydrocarbon so the above is quite feasible.

Now let's blank w/ methanol



$H-\overset{H}{\underset{H}{C}}-OH$

The idea did not work.
It does not seem to show much.
Now zero in on the scale.

We see that we should operate between 700 & 910.
But here is a case where we see what we do not have. This is also helpful

This tells that we do not have ArOH
No alcohol - benzene - phenol.

We have learned a lot here today.

We have a very clean separation of denatured alcohol.

1. Acetone
2. Methanol
3. Contaminated Methanol (Slight)
4. Rubber Contamination in methanol.

We have used NIR to analyze part of the rubber - methanol mixture.
We have used methanol as the blank.
We have learned what is not being absorbed (eg in NIR) is of value in eliminating ambiguities.

We have learned to really hone in the detail of NIR info.

Another major finding

Well, of all the unusual findings.

We have already determined that one of the attributes of the CDB lipids appears to be of a plastic nature (flexible plastics).

Now by accident from 2-3 days of investigation and GC development, I find again by an entirely independent method that the CDB aligns very closely w/ IR analysis of artificial rubber.

The method:

1. Contaminate denatured alcohol w/ finely ground artificial test tube stopper rubber.
2. Distill the denatured alcohol into 4 layers
   1. acetone BP 58°C
   2. methanol BP 65°C
   3. Slightly contaminated methanol BP 68°C
   4. Residual (rubber contamination) BP might be unknown here.
3. Evaporate the residual on ATR crystal & run the IR.
4. Search database:
   Two strongest matches
   1. CDB Lipids beyond 182°C
   2. 1,2 Benzenedicarboxylic acid, dicyclohexyl ester.

It is also clear that the contaminated methanol created a film layer on the ATR.

The GC is now set to
150° 20m
ramp to 220°C
220°C 96.5m
$\Sigma = 120$m

The column may simply not be able to elemenate a higher molecular weight carbon compound (eg flexible plastic, artificial rubber).

We see now that we should be able to distill the rainwater.
We have ΔT of 4°C.

We can separate ΔT of 2°C.

There is another lesson to be learned here.

Grinding the artificial stopper w/ the drill allowed us to create a very fine powder. The fine powder was much more easily assimilated by the methanol.

Lesson: Physical pulverization of organic material (probably any material) allows for an easier assimilation within a said solvent.

Another lesson.

The volume of liquids can change w/ heat, ie various liquids.

We noticed this earlier w/ wax.

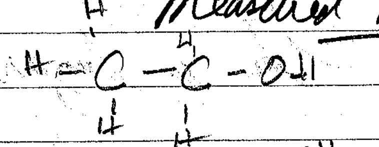
Now we are noticing it with isopropanol. It is significant. and is now borderline for the lively flash

I am distilling isopropanol. It has settled in @ 72°C. Why and how?

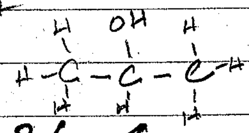
This is really strange. I get a BP of 74-75°. The closest to this is ethanol not isopropanol.

	BP
Ethanol	78° C
Isopropanol (1)	97°
(2)	82°

Measured 75° C



ethanol



2 propanol

What does this mean? Use IOR.

Read Brix 26.4

IOR = 1.373

Ethanol IOR = 1.361 Δ=.012

Isopropanol IOR = 1.384 (1) Δ=.011

1.377 (2)

We seem to have this But the boiling point is off. Why?

H

BP is different because it is an azeotrope !!!

IR is spot on

BP theoretically is 82°C

but I measure 75°C

fascinating. An azeotrope cannot be separated by distillation...

You are not really separating the water from it!

You need to salt it out.

This is bizarre!

Distillation of Concentrated Rainwater

Fraction (1)

1. We start @ 84°C
85°C
86°C
87°C
88°C Steadier
90°C

The water sample seems to be pretty very clear.

Brix = 0.6   IOR = 1.333

The water has an unpleasant odor to it. It is not clear.

It seems to be slowing down. Temp is dropping to 87°C and the nature of the liquid may be changing.

No match @ BP = 88° & IOR = 1.333 w/ Colby.

Nothing yet in IR w/ distilled water as background.
Continue w/ distillation and concentration.

Aug 09 2016

Headed back to basics w/ GC w/ room air.

Had what appeared to be a faulty run with no air detection so decided to investigate for any problem.

All looks OK.

Am looking for more detection of CO_2

Wgt fact We now find 120° OK with

N.67/0.80 N_2O_2 @ 0.36 94.5% $\bar{X}$=.74

.915 CO_2 @ 0.94 5.5%

We have a great table of response factors. Do not lose it!

for CO_2 $.915(.74) = 0.68$

and N_2,O_2 $\bar{X} = 0.74$

so $\frac{.68}{.74} = 0.92$ and $0.92(5.5\%) = 5.0\%$

This is our estimate of CO_2 concentration.

Now let's drop to 100°C.
Very clear also.

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OK, we see where our gas.
It's clean.

The rainwater final concentration did not go so well.

GC picks up nothing of significance and leaves a long tail.

IR is also very difficult and reveals little.

NIR reveals nothing also.

So we learn from all of this that the water is not a strong source of organic compounds but that it appears to be a source of significant inorganic compounds. The solid materials, in contrast, appear to be a very strong source of organic compounds and it is where the filaments were generated from.

So none of the above should be too surprising when all is said and done.

Rain Water → Strength seems to be in Inorganics

Rain Solids → Strength appears to be in organics

This is an important distinction & discovery in its own right.

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A Comment & observation:

Proteins are NOT amino acids.

You do not use a ninhydrin test for proteins. You use it for amino acids,

It is not the same thing.

You would need to break down a protein into the amino acids before you can use a ninhydrin test. You could perform a protein test, however.

The ninhydrin test works fine when you know that you are dealing w/ an amino acid.

Now, the 1/4 million dollar question is,

What exactly do you want to separate w/ GC?
You know that you can detect gases well.
You know that you can detect water and alcohols easily but they have strong tails and you don't know what to do with them.
You have experimented some w/ headspace and you have made some figures there.
But you are still not seeing what to do with it.
Heavier hydrocarbons also seem to be a problem as they will stay in the column.

Page 190

So the question remains, what exactly can you do with GC?

Permeate did not exactly work either as the solution appears to involve mostly inorganics & not volatile gases.

So how do you use GC exactly?
It seems really good for gases.

Urine & blood could seem like fun.
The problem is that you really do not know what you want to analyze. You always know w/ IR.

Maybe the best approach is pyrolysis & then the capture of the various gases?

But then the question is, why not just directly put the gas into IR? One reason is because it is then complex. GC can tell you how many parts to the whole that you can have.

You really need to think about what are you trying to accomplish here?

Now a very interesting application is
a culture dish. Gases produced? Capture
the gas. How many parts?
What are the gases?
That is an application.

Page 191

Question:

What gases do yeast produce? CO_2

~~Notice that the yeast appears~~ No, the in the balloon.

Aug 10 2016

We have great success in the production of CO_2 by yeast & sugar and captured with a balloon on a test tube.

CDB

We now have several culture in place w/ various mediums to see if any gas is produced.

In the meantime I would like to continue w/ headspace & pyrolysis work.

What type of material would you like to work with a a sample & why are you doing it?
It seems like it would be a lot more valuable to keep up the controls.
You have butane, propane.
CO_2, N_2, H_2

but there seem to be a lot of unknowns.

GC applied to a mothball heated.
(Melting point is fairly low...)

We see O_2, N_2, CO_2 & something is attached to water (long tail)? ~ 55 min or so.
Mothballs are dichlorobenzene.

The culture mediums are indeed showing some bubble forming. The idea might work.

GC is not so straightforward. Most substance do not seem volatile (or low energy Carbon no) even when I think they should be.

Mothballs are a good example.

Distillation was successful and it has taught me a great deal.

You have rainfall examined, now you need to write it up.

Your main goal is actually administrative right now: MRP - Phase II

The laboratory is in a regrouping stage.
You are highly interested in GC.

You did some very good regression investigation w/GC.

Can you make ice water w/ salt colder?

I really really like the idea of identifying
Complete unknowns. I could do this
on a regular and repeated basis.
The Checkom to citizen samples.

Aug 11 2016

Gas production does not appear to be a strong suite from the CDB culture process.

No identifiable pattern or volume of gas is detectable thus far under the initial GC trials.

Remember our glucose runs were very productive as a metabolic indicator.

A yeast run as a trial was also very productive after 24 hrs for CO_2 production. This shows that the method is a viable one.

We also know that the culture suffered in a partial vacuum so we know that it is at least partially aerobic.

I will make another trial w/ the cultures shaken up.

You also learned that baking out the GC was overdue, especially w/ the new column. The column acts like it is still baking out to some degree.

How can we get known gases?

Gas Investigations.
We now have the MSDS for product
Cleaner than before.

We all that propane is commonly a mix of

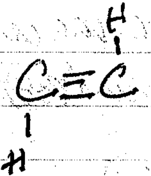
C_3H_8	propane	(saturated)	80-100%
C_2H_6	ethane	(~~vas~~ (saturated)	0-7%
C_3H_6	propylene	(unsaturated)	0-20%
C_4H_{10}	butane	(saturated, 2n+2)	0-2.5%

& ethyl mercaptan if odorized 0-50PPM

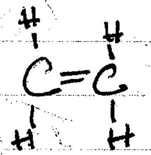
C_5H_{12} It may also have pentane (saturated) 0 - 2.5%

So there can be an entire array.

unsat. What is C_2H_2? Acetylene



unsat. C_2H_4? Ethylene



Now we are looking @ Coleman propane fuel.

Ok, we are getting better.
We definitely have ethane, propane now.
Also N_2, O_2, CO_2.

We have 5 gases to work with now.
We also know how to get hydrogen.

We can also vary the temperature.

It would be good to get butane in here
as well as a ~~satur~~ unsaturated gas
and even pentane.

We see another one coming in.
We now have 2 more gases showing up.

~~11.94 should be~~ butene
~~9.0~~
12.8 is likely butane
10.0 is likely propylene (is unsaturated)

We have a pretty good run on Coleman propane
now we head to Ace Propane.

We can already see that we have a little bit of
CO_2 in the balloon this time.

ACE propane has very little ethane in it!
Very interesting. A very big difference between the
brands.

Notice we get a trailing peak on propane. not in temperature
ACE has a very large propylene. NO.
Which delivers more heat? I think the higher C no.
Then says ACE should burn slower?
Think about acetylene – low carbon no., high temp.
ACE also very low in presumed butane.

Page 197

You are getting really good separation w/ ACE Propane.

You have a total of up to 10 components visible.

N_2O_2
Ethane
CO_2
Propane
Propane?
Butene?
Unknown
Unknown
Unknown.
Unknown
~~Unknown~~.

This is superb work here.

Butane will help to isolate.

Now for the Chevy Blazer Exhaust.

Notice the very minor ethane (likely) Large CO_2 peak.

Notice CO is close to propane but it is not propane.
Yes Cars do put out hydrocarbons.

$$5\% = \frac{\cancel{.005}}{100} = \frac{X}{1E6} = \underline{\underline{\cancel{50\text{ PPM}}}}\ 5000\text{ PPM}$$

You are picking up the hydrocarbon emission on the order of ~~50 ppm~~. 5000 PPM.
This is exactly correct.

Page 198

The GC is working perfectly. You are seeing the sensitivity of the instrument is actually quite high if you know what you are looking for.

There is something very important going on here in understanding how Combustion affects GC ~~interpretation~~ and application.

Gasoline has a C no of what? 4-12
BP = 30° - 210°C

Deisel Carbon no. is 12-20 w/ BP of 170-360°C.

Both are approx 86% Carbon
14% Hydrogen.

—

One of the big tricks in GC is to get the reference points set. These lines will change for each temperature. Maybe you can save them into a Control file.

—

Now working w/ butane. We see that we do not have ethane but we do have CO_2.

A definite propane peak in Ronson butane.
I do not see they disclose them.
Shape of peak at indicates it may be ethane.

It really helped to bake out the Column today.

Page 199

We get an immediate tailing peak of butane.

This indicates the isomer! w/per the MSDS

If you want CO, the easiest way to get it is w/ car exhaust.

I think it is ethane, not CO_2. Hard to say.

Tailing peaks cause complication and they reduce the separation capability.

This butane is pure as it is stated to be w/ two isomers.

Trace hydrocarbons after the isomer.

You are now starting to have very good luck w/ gases. Your next level of understanding is w/ solvents.

Then it is contaminants.

Aug 12 2016 Page 200

You have potentially a very important finding.

The CDB standard culture, @ least w/ H_2O_2 added appear to produce

CO_2
CO
Propane? (Substantial Amount, a definite hydrocarbon gas)

Determined by GC @ 80°C.

It appears that there is a significant time factor involved in the CDB production of gases.

CO_2 is produced immediately, but CO & Propane (?) are coming into the picture later, maybe 24 hrs.

$.36\% = \frac{\cancel{.00}36}{100} = \frac{x}{1E6}$ $x = 3600$ Should be 3600

A problem is that you must number the peaks to print out properly.

~~100 = 1E6 PPM~~

Pase 201

We are in position to start develop a series relationship.

80°C 2n+2
n+r

					n
estimated	Methane	1	CH_4	~0.3 min	1
	Ethane		C_2H_6	~1.32 min	2
	Propane		C_3H_8	~~4.02~~ 3.1	~~6~~ 3
	Butane		C_4H_{10}	~10	4

0.502 n − .962
log(tr) = ~~0.505n − .96~~ $r^2 = 0.99$

.502n − .962
~~.505n − .962~~
or tr ≅ 10

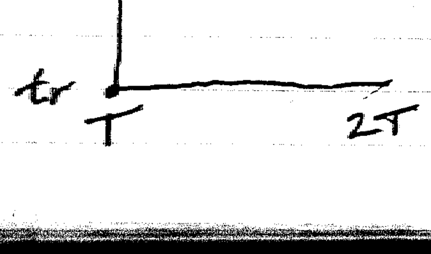
Therefore Pentane prediction is: 35.3 m ~~36.7~~ m @ 80°C

Now how does time affect?
We have learned that doubly the temperature tripled the time. Therefore

$3 \cdot T_r^* = T \cdot 2$

eg T = 80°
Tr = 3 min
2·80 = 3

~~B~~ tr = k·T



~~(n+1)~~(2) (n-1)3

~~(n+1)(2)~~ ~~(n+1)·3~~

~~n-2~~ ~~n·3~~

$T = k \cdot t_r$

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g T = 80, tr = 5 min ~~n=0~~ 1

$2n \cdot 80 = \overset{3}{\cancel{k}} \cdot n \cdot t_r$

~~Method~~

We need propane @ 150°C
And butane @ 150°C (use Benzonetic)
We can now prepare a C_nH_{2n+2} for 150°C

	n	tr
Ethane	2	0.67
Propane	3	1.19
Butane	4	~~1.88~~ 2.09

$\ln(t_r)_{150}$ = ~~0.224n − 0.613~~ ~~r² = .996~~

0.241n − .661

~~0.224n − 0.613~~ r² = .99999

$t_{r_{150}} \cong 10$

0.241n − .661

~~0.224(5) − .613~~

= ~~3.21~~ 3.69

So pentane estimate is 10

hexane estimate is ~~5.38~~ 6.53 matches perfectly.

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We can still combine all data points.

Aug 13 2016

Two immediate goals:

1. Regression formula for C_nH_{2n+2} for GC

$t_r = f(\text{Temp}, C\ \text{number})$ eventually vapor pressure

2. Verify carbon monoxide and any HC's
& Post the paper

~~[illegible]~~ $= C_1 \cdot$

$\log_{10}(t_r) = C_1 \cdot n + C_0$

but we would like

$$\log_{10}(t_r) = C_1 \cdot T + C_2 \cdot n_{alkane} + C_0$$

$$\xcancel{\begin{bmatrix} C_1 & C_2 & C_0 \\ & & \end{bmatrix} \begin{bmatrix} T \\ n_{alkane} \\ 1 \end{bmatrix} = \begin{bmatrix} \log_{10} t_r \\ \vdots \end{bmatrix}}$$

$$\begin{bmatrix} T_i & \overset{(MW?)}{n_{alkane_i}} & 1 \\ & & \end{bmatrix} \begin{bmatrix} C_1 \\ C_2 \\ C_0 \end{bmatrix} = \begin{bmatrix} \log_{10}(t_{r_1}) \\ \vdots \end{bmatrix}$$

It might be that we want to use a molecular weight instead of N

Vapor Pressure
Thermal Conductivity)

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$V+B\Delta=\Delta$

$V=\Delta-B\Delta$

		T	MW				ln
N_2	1	60	28.01	1		0.38	-.968
CO_2	2	60	44.01	1		2.60	.955
H_2	3	60	2.02	1		0.30	-1.204
CO_2	4	40	44.01	1		3.93	1.369
CO_2	5	60	44.01	1		2.57	.944
Propane	6	80	44.10	1		4.25	1.447
O_2	7	60	32. ~~16.00~~	1		0.38	-.968
N_2	8	80	28.01	1		0.33	-1.109
O_2	9	80	16.00	1		0.33	-1.109
Ethane	10	80	30.07	1		1.303	.265
Propane	11	80	44.10	1		3.79	1.332
Ethane	12	80	30.07	1		1.33	.285
Propane	13	80	44.10	1		3.56	1.270
CO_2	14	80	44.01	1		1.63	0.488
CO	15	80	28.01	1		4.27	1.452
Butane	16	80	58.12	1		11.5	2.442
Ethane	17	80	30.07	1		1.2	.182
Propane	18	80	44.10	1		4.1	1.411
Butane	19	80	58.12	1		10	2.302
~~Butane?~~	~~20~~	~~80~~ ok	~~58.11~~ ok	~~1~~	ok	~~13.03~~	~~2.567~~
Propane	21	150	44.10	1		1.1	.095
Ethane	22	150	30.07	1		0.64	-.446
Propene	23	150 ?	42.08	Strike	? (1.36)	1.783 ?	0.578
Butane	24	150 ?	58.12	Strike	? (4.42)	2.343	0.851
Ethane	25	220	30.07	1		.483	-.728
[illegible]	26	220	28.01	1		.84	-.174
Propane	27	220	44.10	1		1.04	.039
Propene	28	220	42.08	1		1.373	.317
Butane	29	220	58.12	1		2.186	.782
Butene	30	220	56.1	1		2.793	1.027
[illegible]	[illegible]	[illegible]	30.1	1		.666	-.406
[illegible]	[illegible]	[illegible]	44.0	1		1.186	.171
[illegible]	[illegible]	[illegible]	44.1	1		1.88	.631
[illegible]	[illegible]	[illegible]	56.6	1		4.416	1.485
[illegible]	[illegible]	[illegible]	56	1		5.38	1.683

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$C_1 \cdot T + C_2 \cdot MW + C_0 = \ln(tr)$

or

$C_2\,MW = \ln(tr) - C_1 T - C_0$

or

$MW = \dfrac{\ln(tr) - C_1 T - C_0}{C_2}$

assume $T = 60°C$
$tr = 3$ min

$C_1 = -.014$
$C_2 = +.065$
$C_0 = -1.082$

$MW = \underline{28.8}$ This is exactly
very interesting

assume $T = 60°$
$tr = 10$ min
$MW \cong \underline{61.9}$

0.24
0.39
0.31 $C_1 = 3.37E{-3}$ $C_2 = .069$ $C_0 = -2.156$

Quite good!
We indeed got Butene from Colby.

Assume $T = 60°$ $tr = 10$ m
$MW \cong 60.49$

$\sigma = 0.30$ $C_1 = 4.818E{-3}$ $C_2 = .072$ $C_0 = -2.342$
$\sigma = 0.32$
$\sigma = 0.30$ $C_1 = -8.192E{-3}$ $C_2 = .074$ $C_0 = -1.462$

$\sigma = .30$ $C_1 = -9.569E{-3}$ $C_2 = .071$ $C_0 = -1.282$

$\sigma = 0.27$ $C_1 = -6.23E{-3}$ $C_2 = .067$ $C_0 = -1.375$
$\sigma = 0.24$ $C_1 = -6.357E{-3}$ $C_2 = .067$ $C_0 = -1.396$

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We can now use our regression to start predicting molecular weights.

n=22 $$MW \cong \frac{\ln(t_r) + 8.192E3T + 1.462}{.074}$$

Now we have a real life case of uncertainty w/ Coleman Fuel.

Coleman Fuel: $t_r = 1.783$ $MW \cong 44.18$
$T = 150°$

This estimates propane but the peak is one above propane.

But propene is 42.08
and butane has a MW of 58 so we know that it is not butane. It must be propene.

Next: $t_r = 2.343$
$T = 150°C$ $MW \cong 47.9$
This must therefore be butane
actual is 58.1

Now we wish to predict item 20:

$$MW \cong \frac{\ln(t_r) + 9.569E-3T + 1.282}{.071}$$

Now, item 20: $t_r = 13.03$
$T = 80°C$
$MW \cong 65.0$

Now we know that it is not butane because it is a small peak.
The MW of butene is 56.1

And we know therefore that it is not propene.

Now what is the MW of pentane? 72.15?

It is possible that it is pentane
But since it did not separate completely even @ 80°C the proper candidate is indeed butene

We do have some predictive capability here.
Now let's look @ CDB activation

Nice clean peak $t_r = 12.32$
$T = 80°C$
$MW \cong 64.2$

And this compares against

		Predicted	
Propene	Butane	Butene	Pentane
42.08	58.12	56.1	72.15

↑
We have a
control here
This is too low
w/ the retention time.

It could be
either one.
Capture the gas
and place into
IR!

Pesc 209

Then is getting very interesting w/ the CDO gas.

It appears to be either Butene

or Pentane

It could be either one but it should help in treating it.

Let's also predict the CO peak in the CDO plot

$t_r = 4.206$

$T = 80°C$

$\hat{MW}^{*} \simeq 49.1$

Actual is 28.0

CO_2 is 44.

So it definitely predicts a higher molecular weight than actual. What is the structure of CO?

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Important findings

CDB Culture gas analyses indicates consistent production of Carbon monoxide. (GC)
Very low levels (regular approx 24 hrs)
Also acetone via (IR)

Lets go back to prediction. Write up paper tomorrow
after another batch of tests.

Predict Charcoal 220 Set

N_2, O_2 $t_r = 0.36$ T=220 $\hat{MW} \approx 30.3$ $N_2 = 28.01$ $O_2 = 16.00$

Not bad.

ethane $t_r =$ ~~2.048~~ 0.483 T=220 $MW^* =$ ~~53.0~~ 34.3 Ethane = 30.1

Good

$t_r = 0.84$ T=220 $MW^* = 41.8$ larger peak $CO_2 = 44.0$ Volatile
Propane = 44.1

Propene = 42.1
$t_r = 1.04$ T=220 $MW^* =$ ~~22.5~~ 44.6 Volatile here Propane = 44.1
$CO_2 = 44.01$
Smaller peak.

so Concentration estimates depending upon Circumstances are an important ~~tool~~ for Abstraction. Burning Charcoal means more CO_2 than propane.

$t_r = 1.313$ T=220 $MW^* = 48.4$ Volatile here Propene 42.1
Butane 58.1

$t_r = 2.186$ T=220 $MW^* = 54.1$ Butane = 58.1

$t_r = 2.793$ T=220 $MW^* = 58.0$ ~~Speed~~ Butene 58.1
Pentane ? MW = 72.1

Our latest regression equation is

$$MW^* \cong \frac{\ln(tr) + 6.23E{-}3 \cdot T + 1.375}{.067}$$

This is therefore a self-improving formula.

Now predict and then integrate charcoal
@ 150°C

tr = .666 T = 150 $MW^* = 28.4$

$\boxed{\text{Ethane} = 30.1}$
$CO_2 = 44.0$

tr = 1.186 T = 150 $MW^* = 37.0$
large peak $CO_2 = 44.0$

tr = 1.88 T = 150 $MW^* = 43.9$ Propane = 44.1
Massive Peak 95%

tr = ~~4.30~~ 4.416 T = 150 MW = 56.2 Butane = ~~58.1~~ 56.6

tr = 5.38 T = 150 $MW^* = 59.6$ Butene 56.1

Therefore our regression now is:

$$MW^* = \ln(tr) + \underset{.067}{\underline{6.357E-3}} \cdot T + 1.396$$

$\sigma_s = 0.24$ Not bad!

also

$$\ln(tr) + 6.357E-3 \cdot T + 1.396 = MW^* \cdot (.067)$$

$$\ln(tr) = (.067 \cdot MW^*) - 6.357E-3 \cdot T - 1.396$$

or

$$tr \simeq e^{.067 \cdot MW^* - 6.357E-3 \cdot T - 1.396}$$

Both forms are useful.

eg MW of acetic acid is: 60.05

Therefore $tr^* = \underline{\underline{5.33\ min}}$ @ 150°C   This is a useful predictive formula.

Bring water, acetone, & acetic acid into picture now.

I suspect that you need to separate out the regression between polar and non polar substances. This should help a lot

Add O_2 & N_2 @ 220°C @ $tr = 0.356$
$\ln(.356) = -1.033$

$\sigma^2 = 0.29$

$C_1 = -6.265E-3$ $C_2 = .069$ $C_3 = -1.506$

So

$$MW^* = \ln(tr) + \frac{6.265E-3 \cdot T}{.069} + 1.506$$

and

$$tr \cong e^{(.069\,MW^* - 6.265E-3 \cdot T - 1.506)}$$

Next consider separation of polar vs non polar compounds.

[redacted]

CDB Gas Analysis:

We are noticing that gas production by the CDB test tube culture is varying.

There are tubes that are not producing higher volume gas but still seem to be producing substantial CDB. I am beginning to wonder if the CDB can become a single large continued feeding culture.
Clarity of the culture spectrum maybe a measure of completeness.

Remember CO is heavier than air.

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