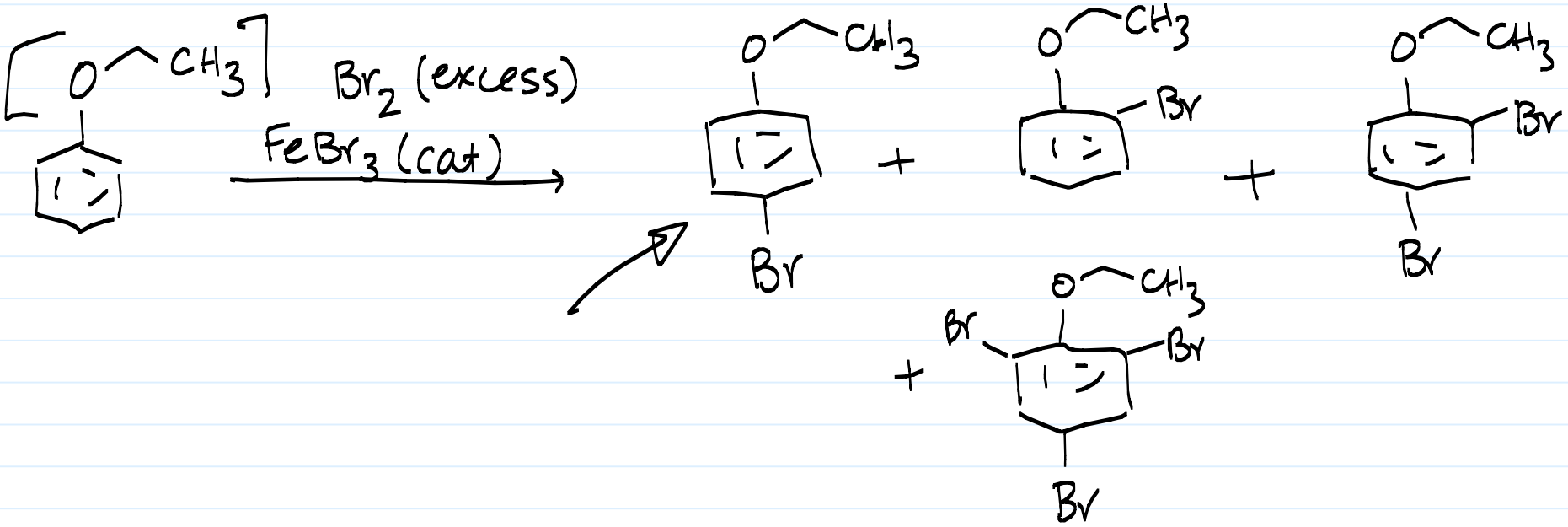
Analytical Chemistry of Organic Compounds (Chapter 15!)

Note Title 3/11/2014

The Reaction is only the beginning...



Step 1: Purification

Methods: 1) Crystallization (if solid)

2) Distillation (if liquid & each component has substantially different boiling points)

★★ 3) Chromatography
- TLC (thin layer chromatography
- Column chromatography
- HPLC (high pressure liquid chromatography)
- GC (gas chromagraphy)

Step 2: What is it?

Options (usually need ≥ 1 of these):

(1) Elemental Analysis (Combustion Analysis) → Burn it

(2) Mass Spectrometry → Blast it

(3) Infrared (IR) Spectroscopy → Shine a light on it.

(4) Nuclear Magnetic Resonance (NMR) Spectroscopy

↓

Play it a song.

Elemental Analysis (or Combustion Analysis)

Derive Empirical Formula by "burning" the compound.

ex: Unknown (C, H, O) + O_2 $\longrightarrow$ CO_2 + H_2O

↑ Carbon content from CO_2

↖ Hydrogen content from H_2O

Oxygen content as the "leftovers."

10 mg unknown $\xrightarrow{\text{burn}}$ 29.31 mg CO_2 + 5.99 mg H_2O

Carbon Content:

$$wt(C) = \frac{MW(C)}{MW(CO_2)} * wt(CO_2) = \frac{12.011\ g/mol}{44.009\ g/mol} \times 29.31\ mg = 8\ mg\ C$$

$$wt(H) = \frac{2\,MW(H)}{MW(H_2O)} * wt(H_2O) = \frac{2(1.008\ g/mol)}{18.015\ g/mol} \times 5.99\ mg = 0.67\ mg\ H$$

$$\%C = \frac{8\ mg\ C}{10\ mg} \times 100 = 80\%\ (\text{by weight})$$

$$\%H = \frac{0.67\ mg\ H}{10\ mg} \times 100 = 6.7\%$$

$$\%O = 100 - 80 - 6.7 = 13.3\%$$

Now what??

Ch Empirical Formula

Calculate # of moles (assume 100 g)

C: $80\,g \left(\frac{1\,mol}{12.01\,g}\right) = 6.66\,mol$

H: $6.7\,g \left(\frac{1\,mol}{1.01\,g}\right) = 6.63\,mol$

O: $13.3\,g \left(\frac{1\,mol}{16.0\,g}\right) = 0.831\,mol$

$$C_{\frac{6.66}{0.831}} H_{\frac{6.63}{0.831}} O_{\frac{0.831}{0.831}}$$

$$\Downarrow$$

$$C_8H_8O$$

$$C_{16}H_{16}O_2$$

From molecular Formula ⇒ Degrees of Unsaturation

HCF

Step 1: Convert Molecular Formula to "Hydrocarbon Formula"

Rules:

Each atom of	is removed and
F, Cl, Br, I	replace by H
O, S	ignored
N, P, B, Al	one H removed also
Si	replace w/ C.

ex: $C_8H_8O \longrightarrow C_8H_8$

(actual) (HCF)

Step 2: Calculate # of H's for alkane w/ same # of C's:

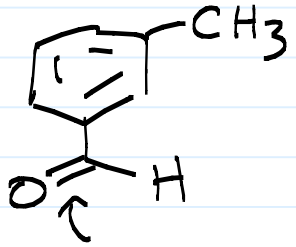
$C_n H_{(2n+2)} \rightarrow C_8 H_{18}$

Step 3: Difference in H's:

Alkane	18
− HCF	− 8
	10

$10/2 = 5$ degrees of unsaturation

$H_3C-CH_3 \rightarrow H_2C=CH_2$



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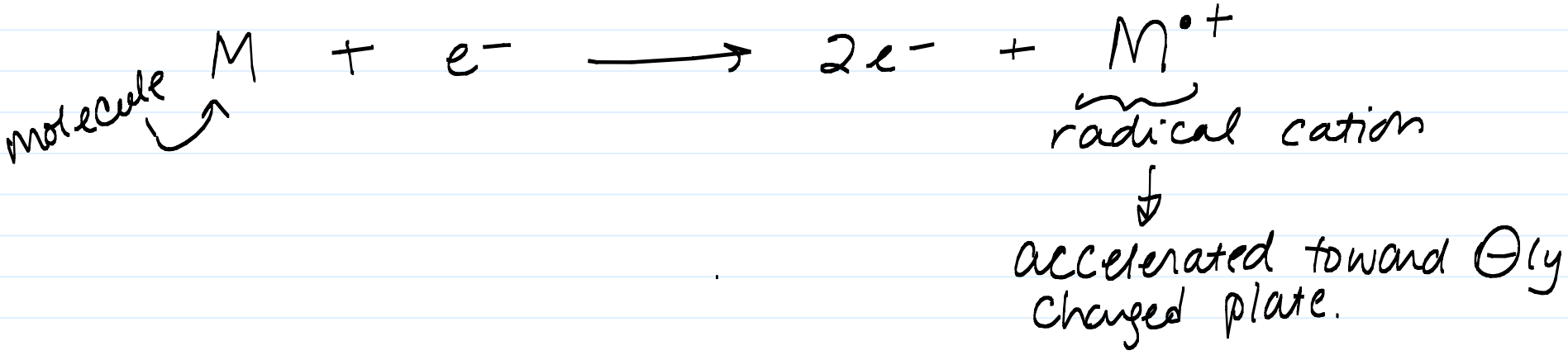
Mass Spectrometry (MS)

- Determination of Molecular Weight → Exact Molecular Formula

1) Bombard molecule w/ e^- (70 eV)



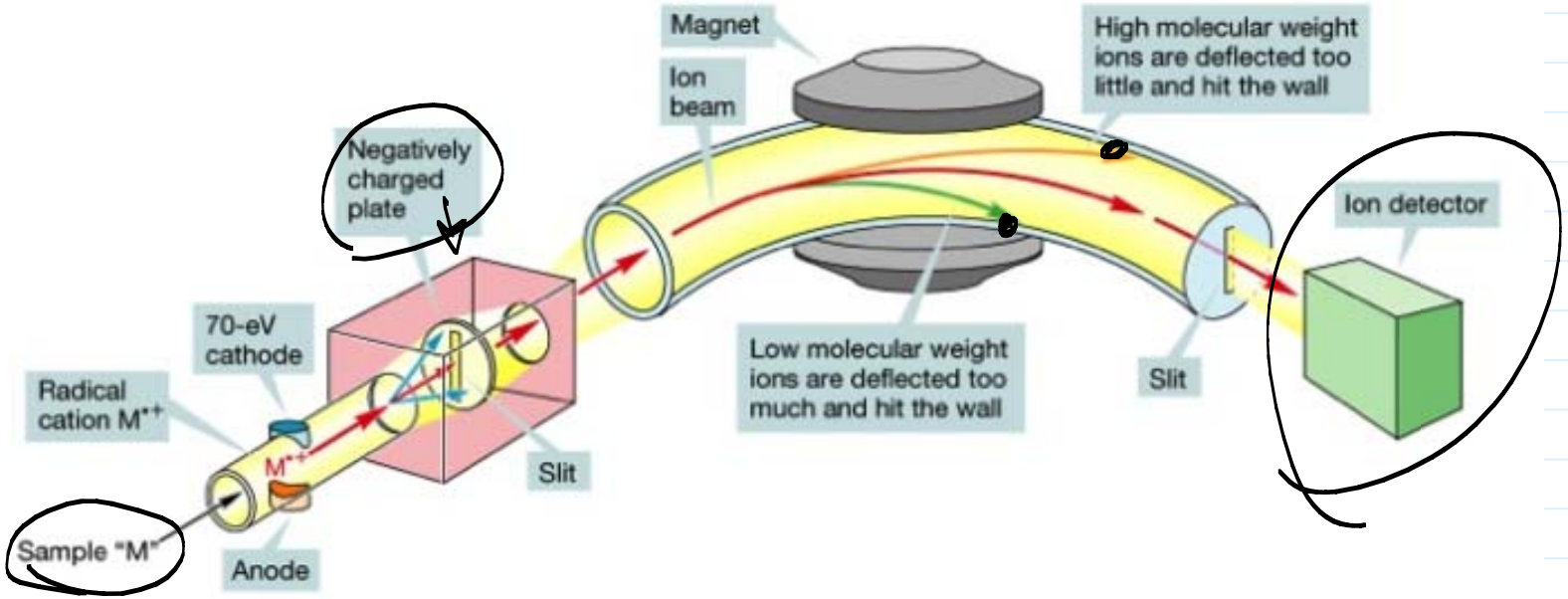


Figure 15.04

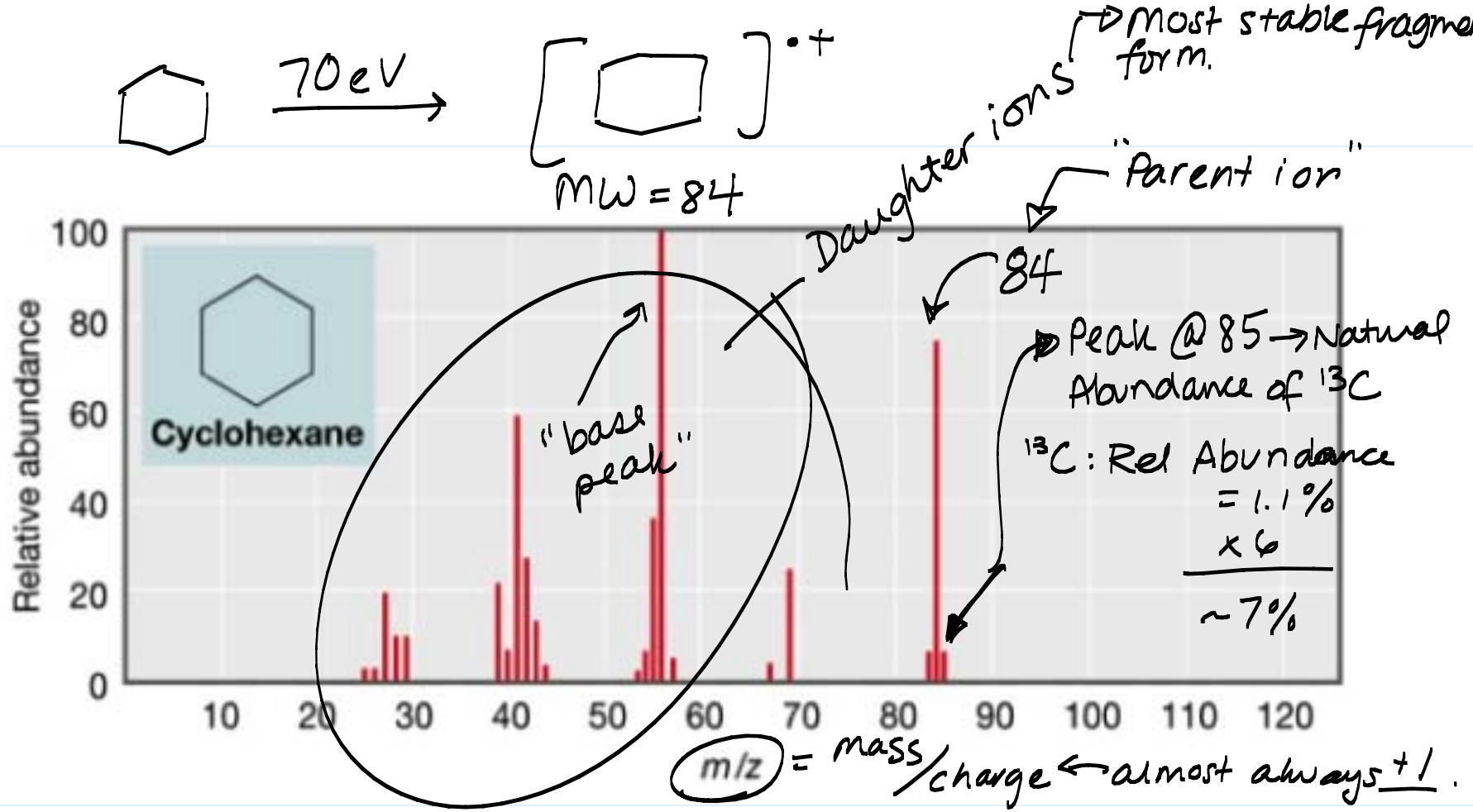


Figure 15.07

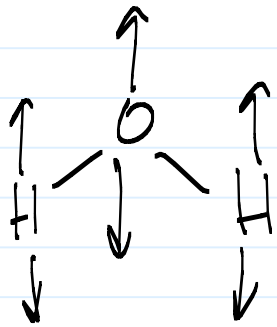
Infrared Spectroscopy (IR)

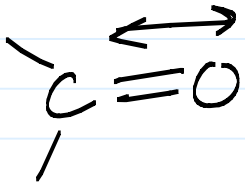
Molecules are always moving ... molecular vibrations

↳ frequency depends on

1) relative masses of atoms
2) force constant (strength) of bond.

ex:





IR Source → IR Radiation, $\bar{\nu}$ = 100–100,000 cm^{-1} → Sample A–B → All other λ's pass through → Detect

Fxnal Groups

Molecular vibrations have energies in IR region.

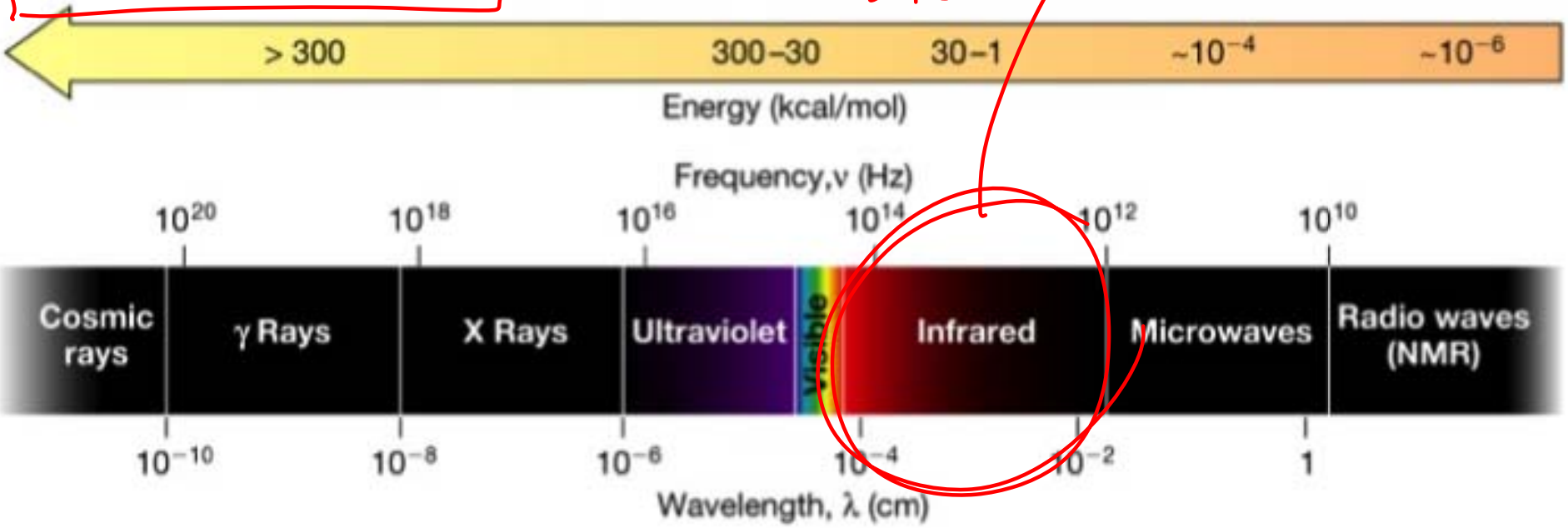


Figure 15.16

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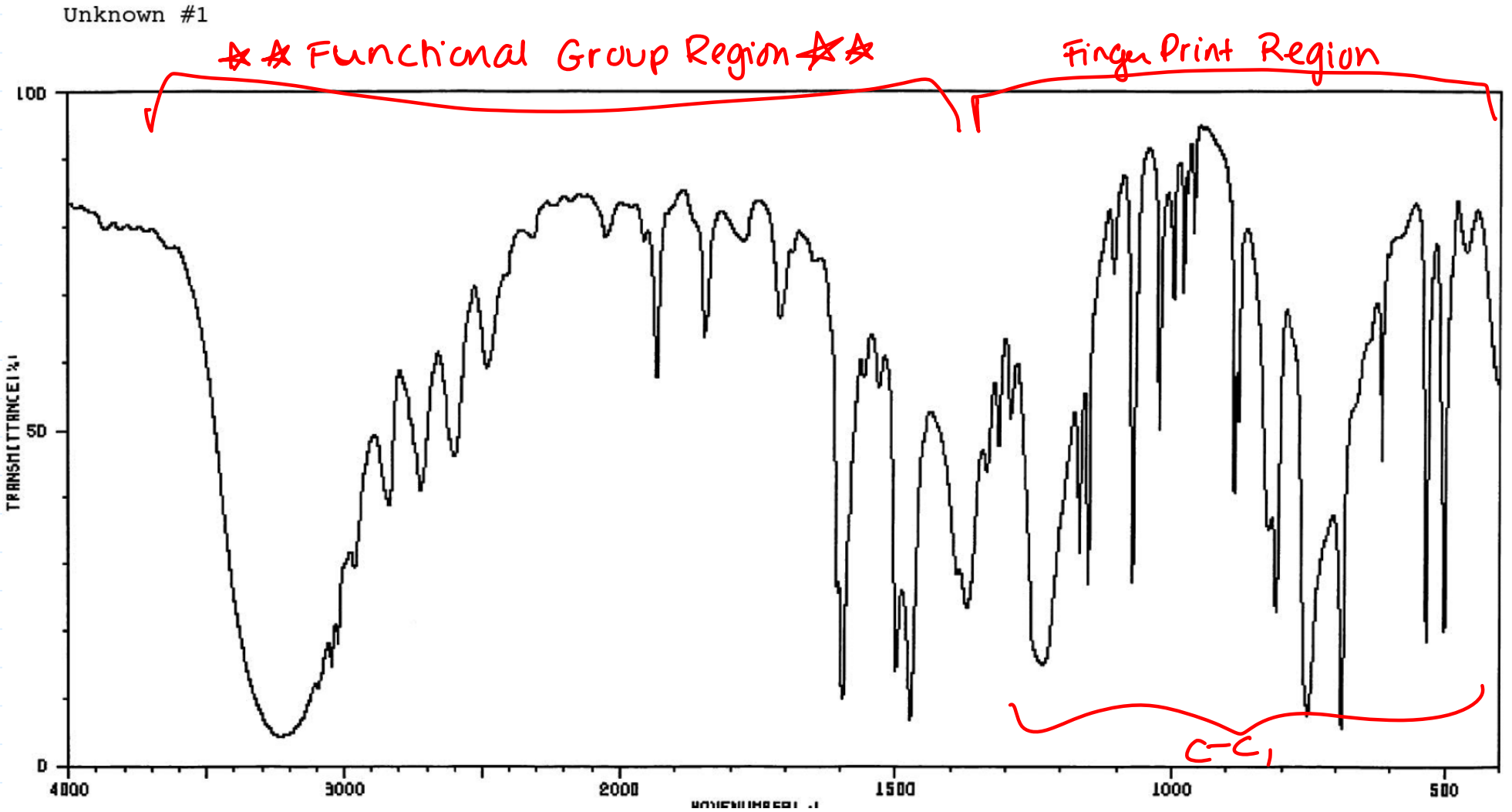

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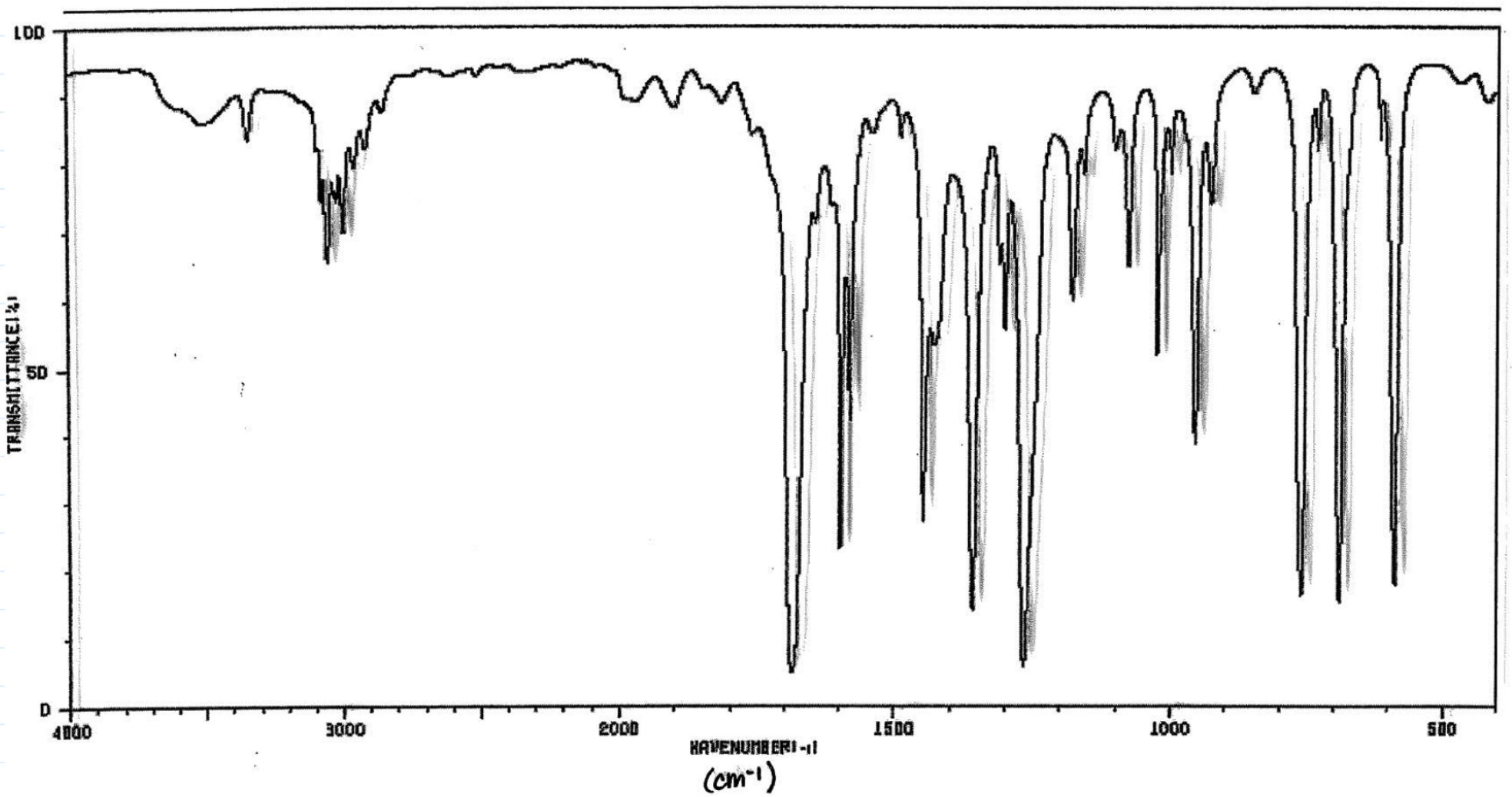
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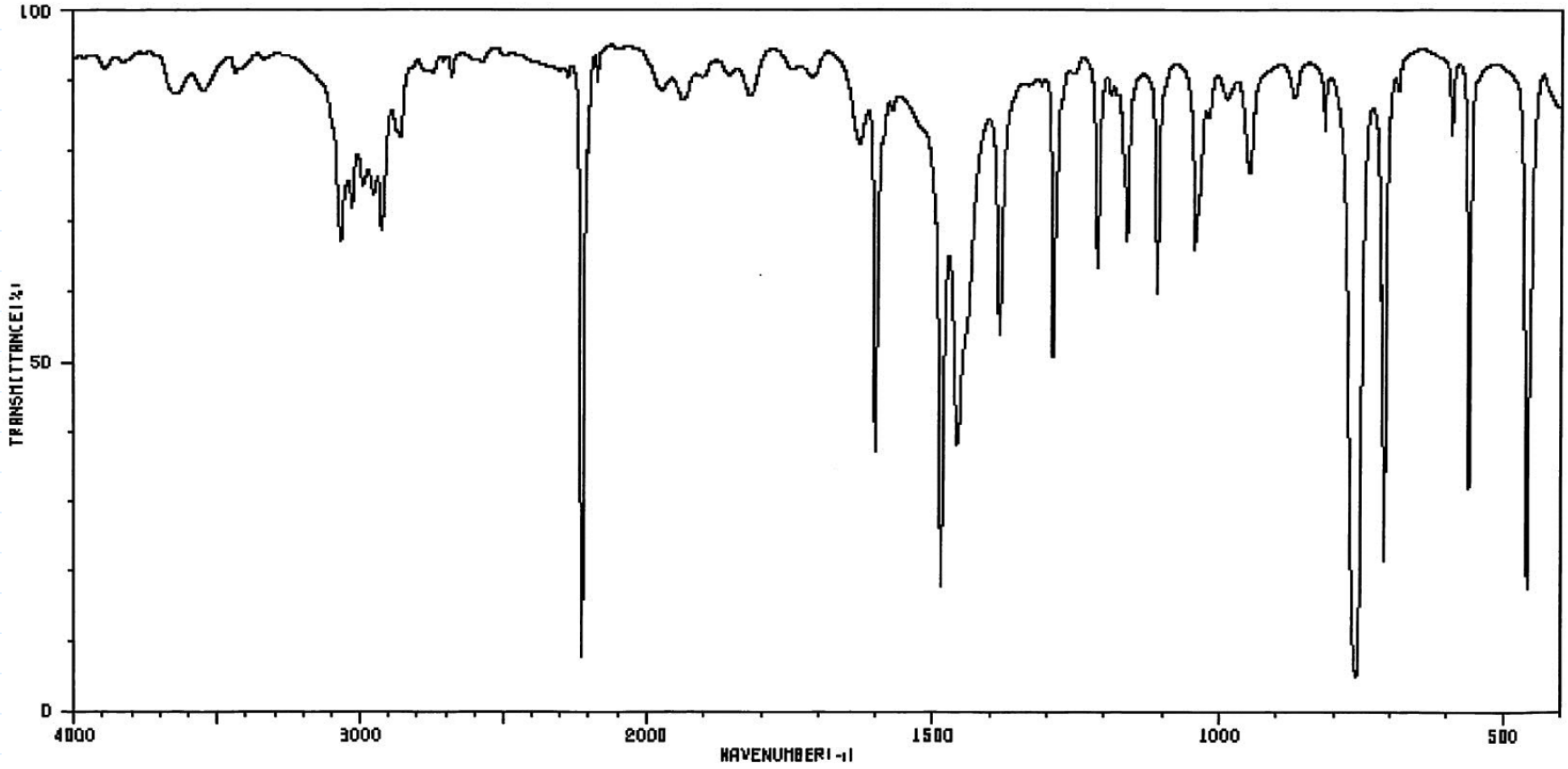
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