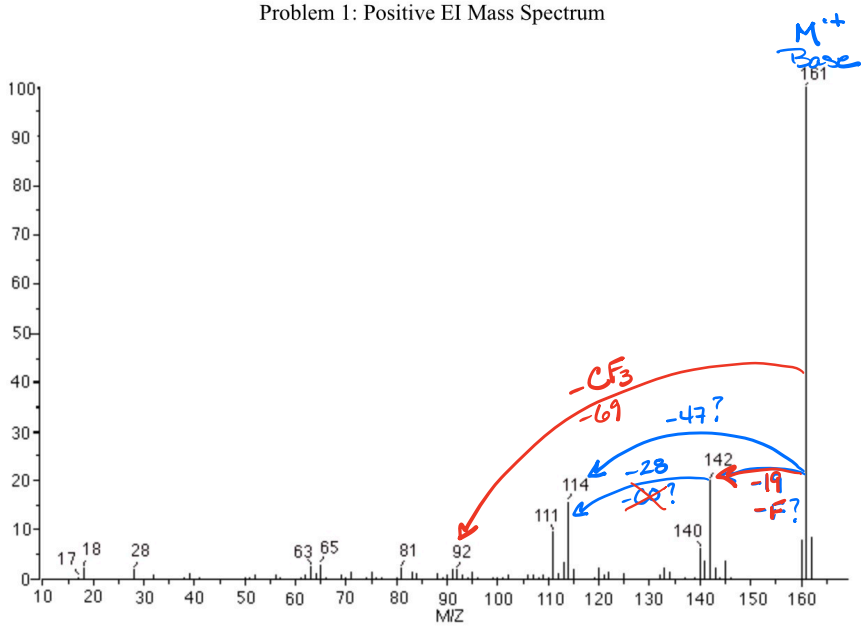
Spectroscopy Rubric

		Beginning (0)	Developing (2.6)	Adequate (3)	Accomplished (3.4)	Mastery (4)
Mass Spec	**Rule 13/13C isotope**	Not used	Rule 13 or 13C used, however error resulting in >±2 carbons obtained	Rule 13 or 13C used, however error resulting in ±2 carbons obtained	Rule 13 or 13C used, however error resulting in ±1 carbon obtained	Number of carbons calculated correctly
	MW	Not Found	Incorrect number of carbons identified, molecular formula does not match molecular ion mass	Incorrect number of carbons identified, however molecular formula matches molecular ion mass	Correct number of carbons found, and formula matches the molecular ion mass, however molecular formula incorrect for other reasons	Correct Molecular Formula Found
	Isotope N,X	Not Found				Significant isotope effects correctly interpreted
	Decomposition Products	Not Found	Few decomposition products calculated, no structural significance noted	Few decomposition products calculated, however significant misinterpretations present, leading to significant errors in corresponding substructures	Some decomposition signals correctly identified, however minor misinterpretations present, leading to minor errors in corresponding substructures	Significant decomposition signals correctly identified with corresponding substructure
IR	**Functional Groups**	No determinations made or all determinations incorrect	Some functional groups determined, however significant misinterpretations present, leading to significant errors	Most functional groups determined, however significant misinterpretations present, leading to significant errors	Most functional groups determined, however minor misinterpretations present, leading to minor errors	All significant functional groups correctly identified. Functional groups not present identified as such.
HNMR	**# Chemical Environ.**	No determination made	Number of chemical environments incorrectly identified, ±2 units	Minor error in determination of chemical environments, ±1 unit	Number of chemical environments determined, however determination is incorrect due to overlapping signals that are difficult to interpret	Number of chemical environments correctly identified
	Spin-Spin Coupling	No determinations made or all determinations incorrect	Some indication that spin-spin coupling determinations were used, however no systematic approach used, leading to significant errors	Systematic determination of spin-spin coupling used, however significant misinterpretations present, leading to significant errors	Systematic determination of spin-spin coupling used, however minor misinterpretations present, leading to minor errors	Splitting patterns correctly identified and number of neighbors correctly determined
	Integration	No determinations made or all determinations incorrect	Some indication that integration values were used, however no systematic approach used, leading to significant errors	Systematic determination of integration values used, however significant misinterpretations present, leading to significant errors	Systematic determination of integration values used, however minor misinterpretations present, leading to minor errors	Integration Values used to correctly determine the number of hydrogens giving rise to each signal
	Assignment	No determinations made or all determinations incorrect	Some substructure identification performed, no systematic approach used, significant major errors made in assignments	Systematic approach used in substructure identification, however significant misinterpretations present, leading to significant errors in assignments	Systematic approach used in substructure identification, however minor misinterpretations present, leading to minor errors in assignments	All chemical signals correctly interpreted into structural subunits
CNMR	**# Chemical Environ.**	No determination made	Number of chemical environments incorrectly identified, ±2 units	Minor error in determination of chemical environments, ±1 unit	Number of chemical environments determined, however determination is incorrect due to overlapping signals that are difficult to interpret	Number of chemical environments correctly identified
	Assignment	No determinations made or all determinations incorrect	Some substructure identification performed, no systematic approach used, significant major errors made in assignments	Systematic approach used in substructure identification, however significant misinterpretations present, leading to significant errors in assignments	Systematic approach used in substructure identification, however minor misinterpretations present, leading to minor errors in assignments	All chemical signals correctly interpreted into structural subunits
Analysis	**Units of Unsaturation**	Not found	Major error in calculation of units of unsaturation, ±2 units	Minor error in calculation of units of unsaturation, ±1 unit	Units of unsaturation determined, however a mathematical error results in an incorrect determination	Units of unsaturation correctly determined
	Propose Structures	No structures proposed	A single compound proposed	At least two structural isomers considered	More than two structural isomers considered	All reasonable structural isomers considered
	Structure determination	No analysis completed on proposed structures	Analysis performed on single compound overlooking major flaws in proposed structure leading to an incorrect determination	Analysis performed on multiple structural isomers, however major inconsistencies between spectroscopic data and proposed structures overlooked leading to an incorrect determination	Analysis performed on multiple structural isomers, however minor inconsistencies between spectroscopic data and proposed structures overlooked leading to an incorrect determination	Proposed structures analyzed to correctly determine the correct structure

Problem 1: Positive EI Mass Spectrum



16
12
28

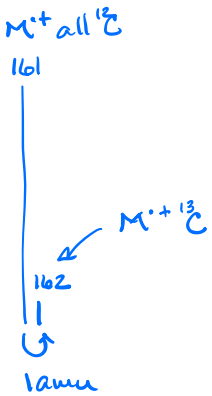
$M^{\cdot+}$ = highest m/z ratio less usually 1 amu for the ^{13}C isotope peak.

161

Base = 161

= Most abundant peak

Tallest = Base



$M^{\cdot+}$ = Base = Aromatic

$M^{\cdot+}$ = 161 = odd ⟹ Molecule has odd # N

Rule 13

$$13 \overline{)161} \quad 12 \text{ R } 5$$

13 | 31 | 26 | 5

$C_{12}H_{12+5} = C_{12}H_{17}$

$-CH_2$, $+N$ → $C_{11}H_{15}N$

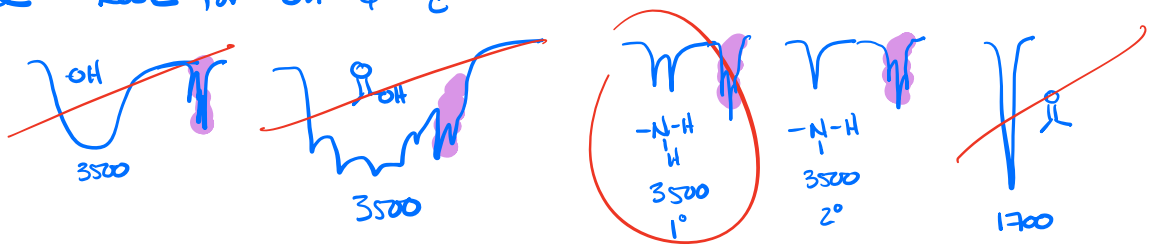
$N = 14 = CH_2$

$F = 19 = CH_5$

No oxygen

Look to other spectra for help on formula

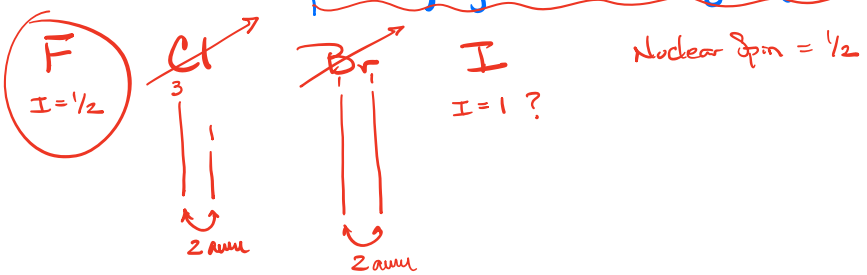
IR - Look for OH & $\overset{O}{\overset{\|}{C}}$

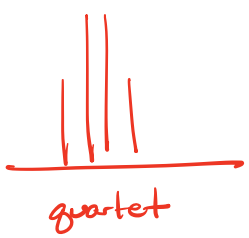


✓ ^{1}HNMR - Integrations absolute ⇒ # H's 6H's

✓ ^{13}CNMR - Chemical Environments ⇒ min # C at least 6C

✓ Hint - Regarding Halogen w/ I = ½ follows the N+1 rule for splitting just like hydrogen

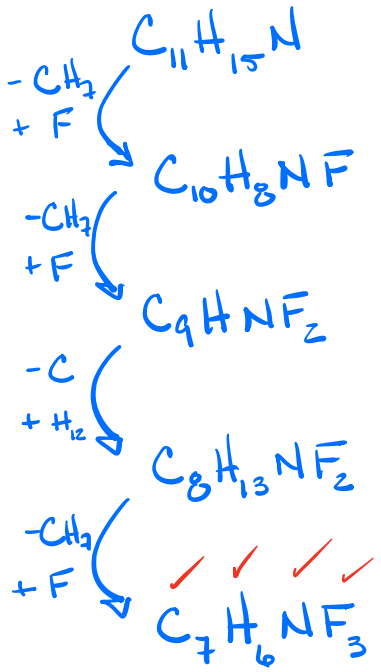




n+1 rule

How many neighbors?

= 3 neighbors



$7\times12+6+14+3\times19=161$ ✓

Molecular Formula = $C_7H_6NF_3$

Units of unsaturation $C_nH_{2n+2+N-X}$

$2(7)+2+\overset{N}{1}-\overset{3F}{3}=$

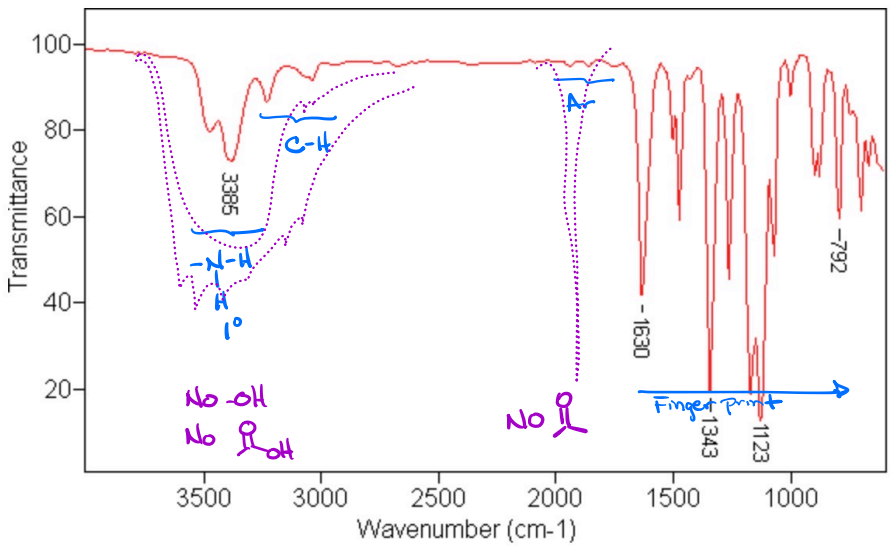
$14+2+1-3=14$

-6

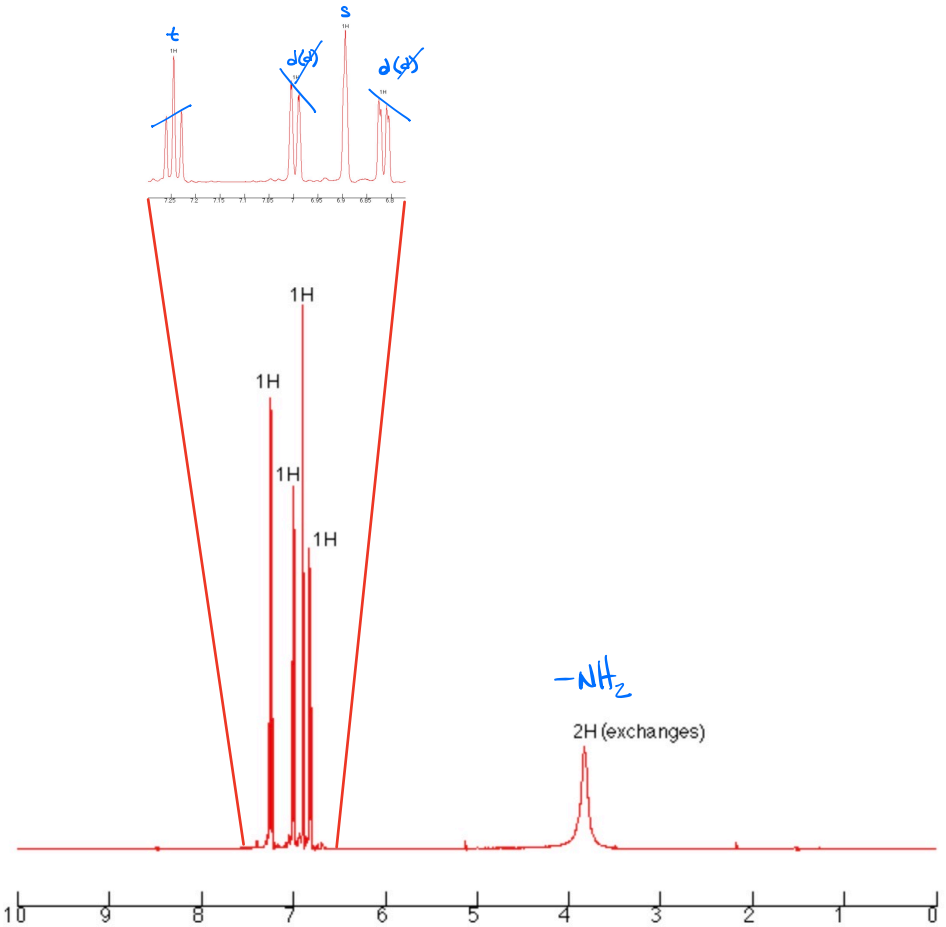
$2\,|\,8$

4 units unsat

Problem 1: IR spectrum

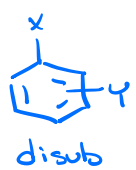


1° Amine

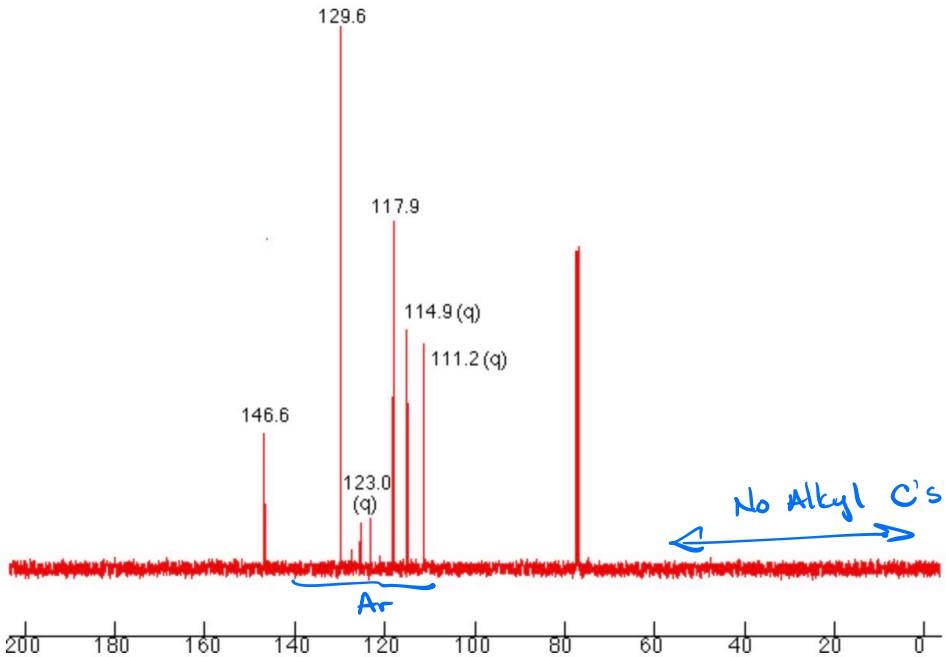


Environments = 5

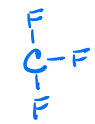
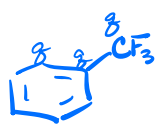
ppm	Int	mult	# neighbors	Assignments
3.9	2	S (exchanges)	∅	$-NH_2$
6.8	1	d(d)	1	Ar-H
6.9	1	s	∅	
7.0	1	d(d)	1	
7.25	1	t	2	



Problem 1: ^{13}C NMR spectrum ($CDCl_3$, 125 MHz)

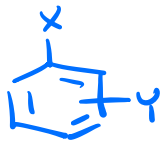


Enviroments = 6

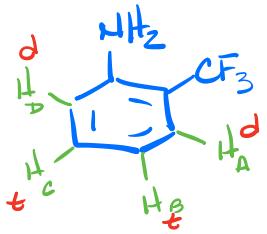
ppm	mult	Assignments
111.2	q	
114.9	q	Ar
117.9	s	
123.0	q	
129.6	s	
146.6	s	

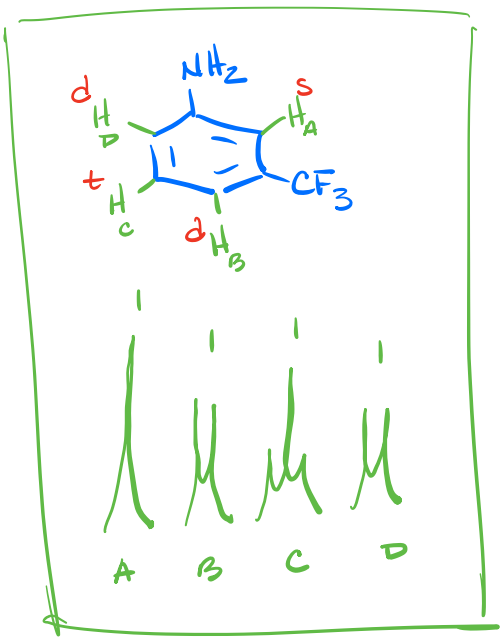
Parts

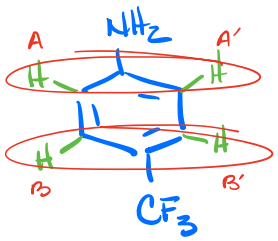
$C_7H_6NF_3$

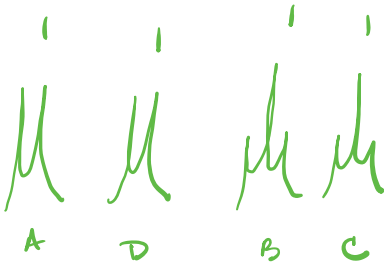


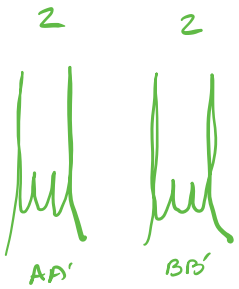
X = $-NH_2$ Y = $-CF_3$



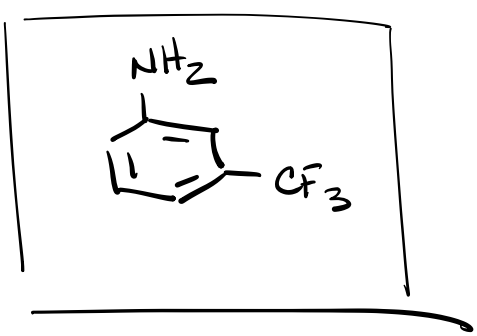








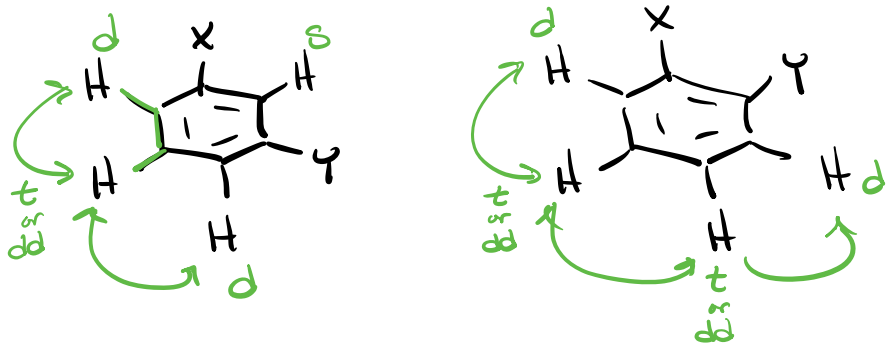
Answer



$-NH_2$ -16

$-CF_3$ -69

J_3 Neighbor



s	1				0 neighbors
d	1	1			1 neighbor
t	1	2	1		2 neighbors
q	1	3	3	1	3 neighbors

CoOP # 2

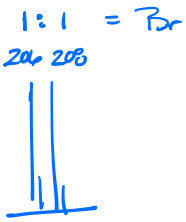
IR → no OH, no  , No 

→ Just Hydrocarbon

HNMR → All aromatic

CNMR → All aromatic

Mass Spec → $M^{+} = 206$



Challenge is in the HNMR

⇒ Recomendation is to Consider molecule options & look to differentiate in HNMR

CoOP #3

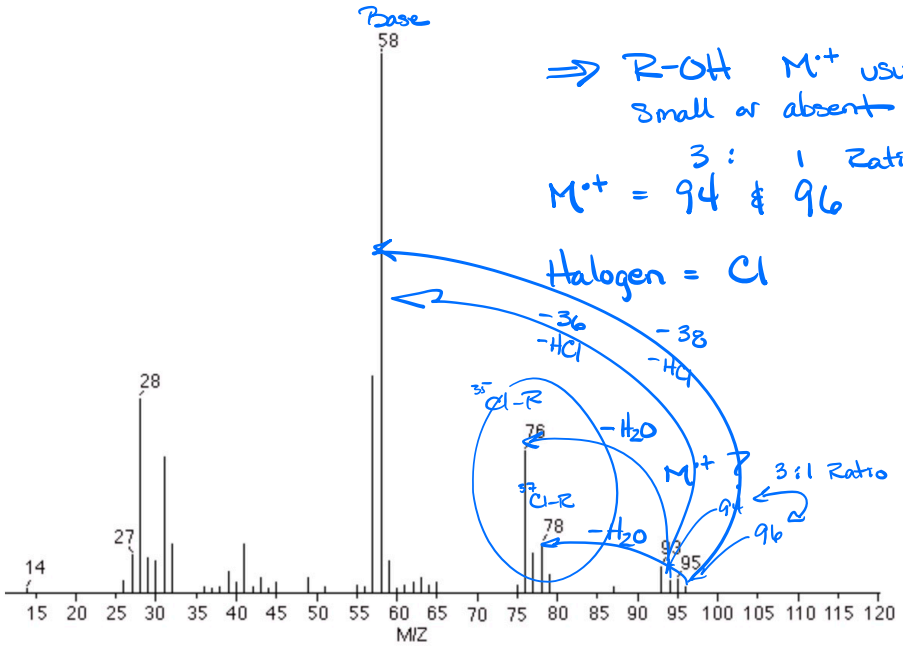
IR - Alcohol
No π

^{1}HNMR - All alkyl → no aromatic

^{13}CNMR - All alkyl → all $-\overset{+}{C}H_2-$

Mass Spec

Problem 3: Positive EI Mass Spectrum



⇒ R-OH M$^{+\cdot}$ usually very small or absent

3 : 1 Ratio

M$^{+\cdot}$ = 94 & 96

Halogen = Cl

^{35}Cl 94

^{37}Cl 96

^{35}Cl 93

^{37}Cl 95