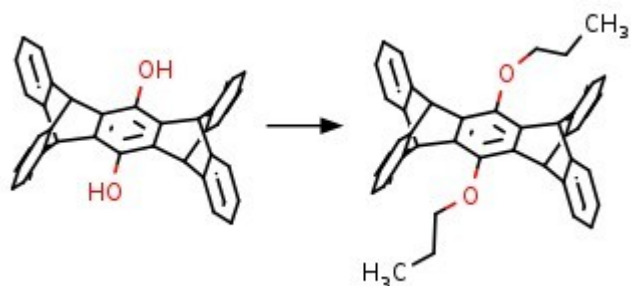


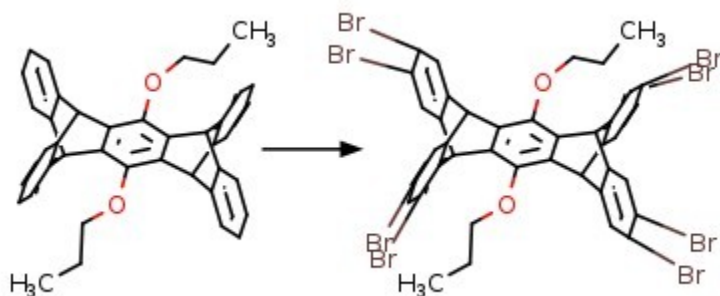
Semi Weekly Report July 12th, 2022

Reactions done:

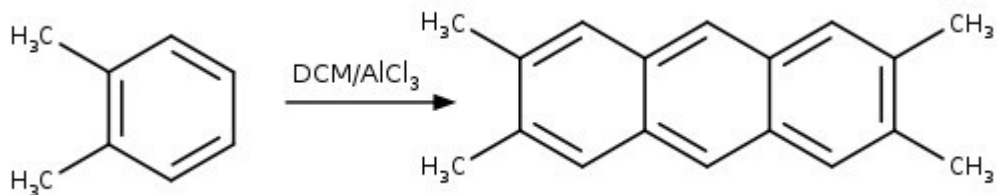
1. Alkylation of pentyptycene hydroquinone



2. Bromination of Dibutylether Pentyptycene



3. FC reaction of o-xylene with DCM (synthesis of 1,2,7,8-tetramethylantracene)



Reactions to be done:

1. N-methyl isoindole DA
2. Carboxytrip/amine coupling

Notebook pages:

Project No. _____
Book No. _____

GC-ELN-FC-091

6-29-
7-1-

FC reaction for 1,2,7,8 TMA

Reaction scheme:
c1ccc2c(c1)ccc3ccccc23 + CH2Cl2 $\xrightarrow[\text{RT}]{\text{AlCl}_3}$ c1ccc2c(c1)ccc3cc4ccccc4cc32
2,3 days at atm (1.96 crude prod)

O-xylene	10 ml
Dichloromethane	10 ml
Aluminum chloride	1.5 g

↳ To a round bottom flask stirring at RT was added 10 ml of o-xylene and 10 ml of DCM

↳ Was added portionwise 1.5 g of aluminum chloride

↳ Was stirred for 2 days under N_2 , then it was stirred for approx. 3 days under atmosphere

↳ An additional 50 ml of DCM was added and the solution was filtered through a pad of silica

↳ The solution was then rotovaped with hexanes and most of the o-xylene was removed

↳ The resultant product was recrystallized from hexanes (minimal DCM)

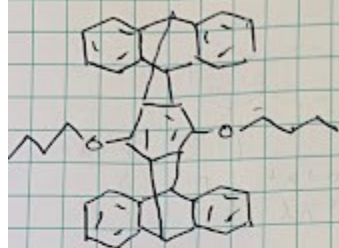
↳ Some impurities in recrystallized product (1.96 g crude)

To Page No. -

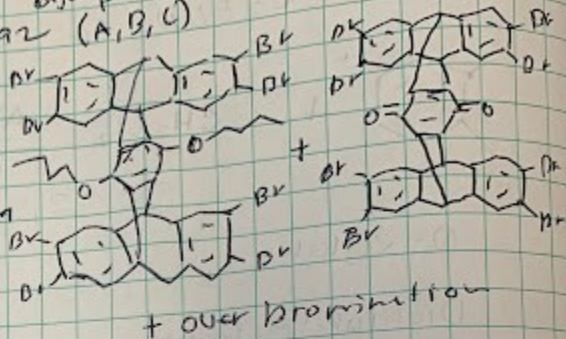
Assessed & Understood by me, _____ Date _____ Invented by: _____ Date _____

386
89204
of dibutyl

GC-ELN-FC-092 (A, B, C)
Hydro pentytyl quinone

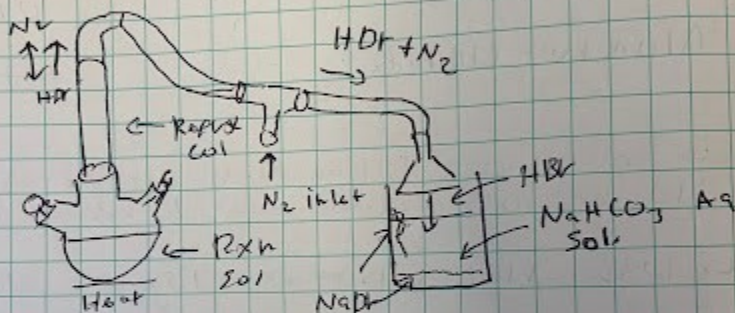


Br₂
Fe (cat.)
Chloroform
18 hrs.
24 hrs



Apparatus Setup:

A - ii (Pitcaud)
B - j
C - i



↳ To a 3 neck flask was added 600mg of product + obtained from GC-ELN-FC-90 and 50 ml of CHCl₃

↳ Was added about ~~10mg~~ 10mg of IRON shavings

↳ Was added 560 ml of Br₂ and allowed to stir under N₂ for approx 10 hrs under N₂ atmosphere

↳ Was added an additional amount of Br₂ (460 ml)

↳ After approx 24 hrs reaction was quenched with Ag. Sodium thiosulfate and organic layer extracted with DCM (3x) and rotated. NMR not good. APPE looked great on B

↳ Run a batch (spilled) and overbrominated

From previous page

Run B: 610 mg → 197 mg (no fraction)

Run C: 602 mg

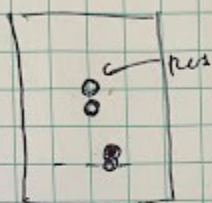
Purification:

→ check product
386 mg 1st crop
892 mg 2nd crop

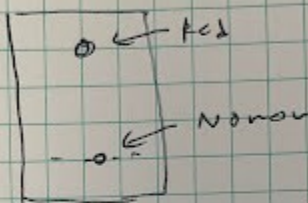
Via TLC



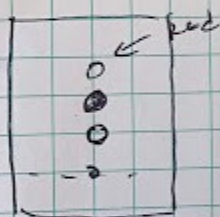
Acetone



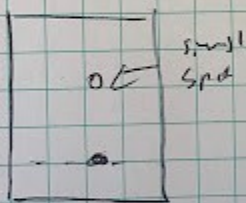
DCM



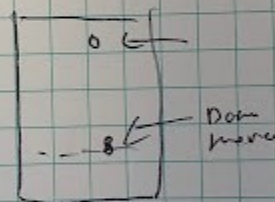
Et₂O



50/50 Acetone
DCM



Acetone
Et₂O



DCM
Et₂O

maybe also
soluble in THF
purification

1. Run silica
plus with I
2. Run DCM
3. Run Acet

- ↳ Titration with Acetone/DCM, Filtrate 1 yields 2
distinct crops
- ↳ Impurities bright Red to Dark Red

Witnessed & Understood by me, _____

Date _____

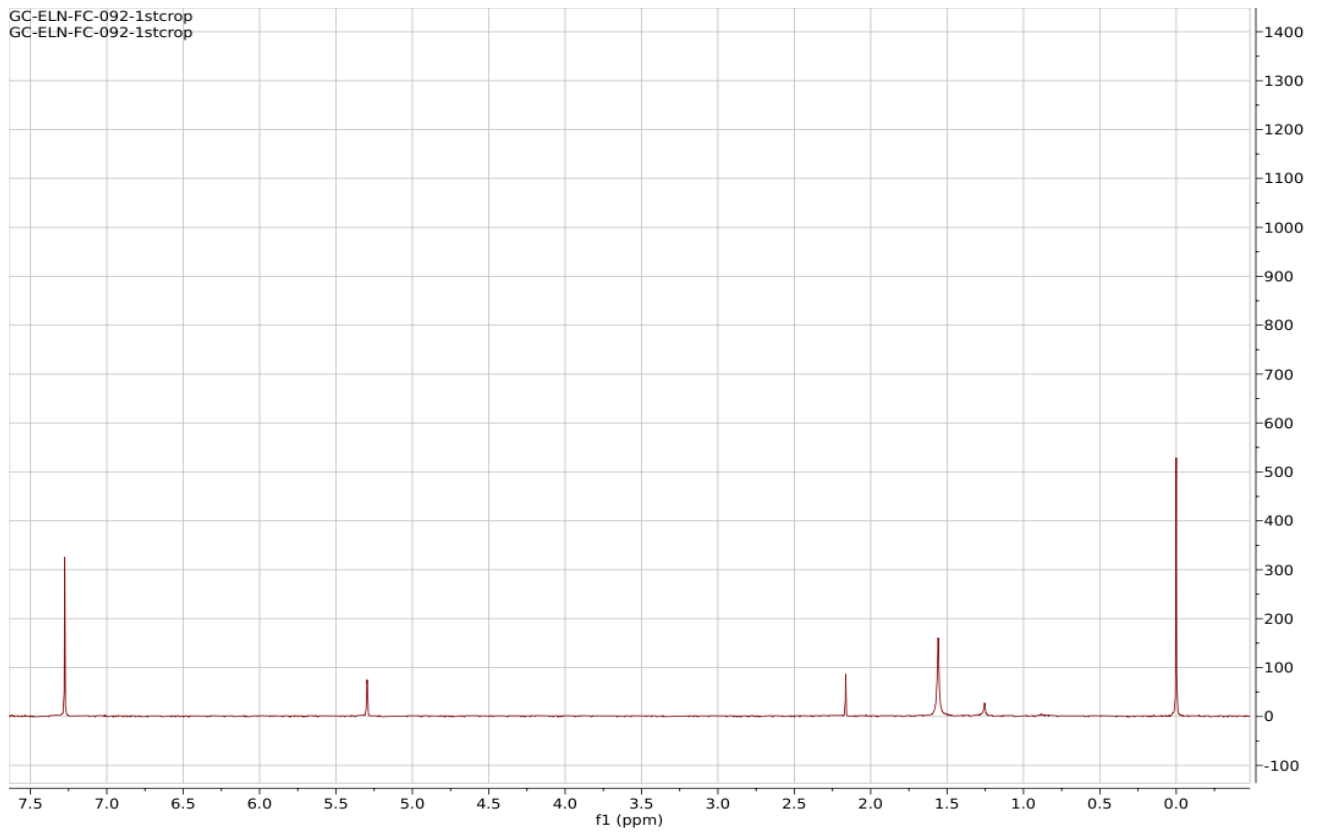
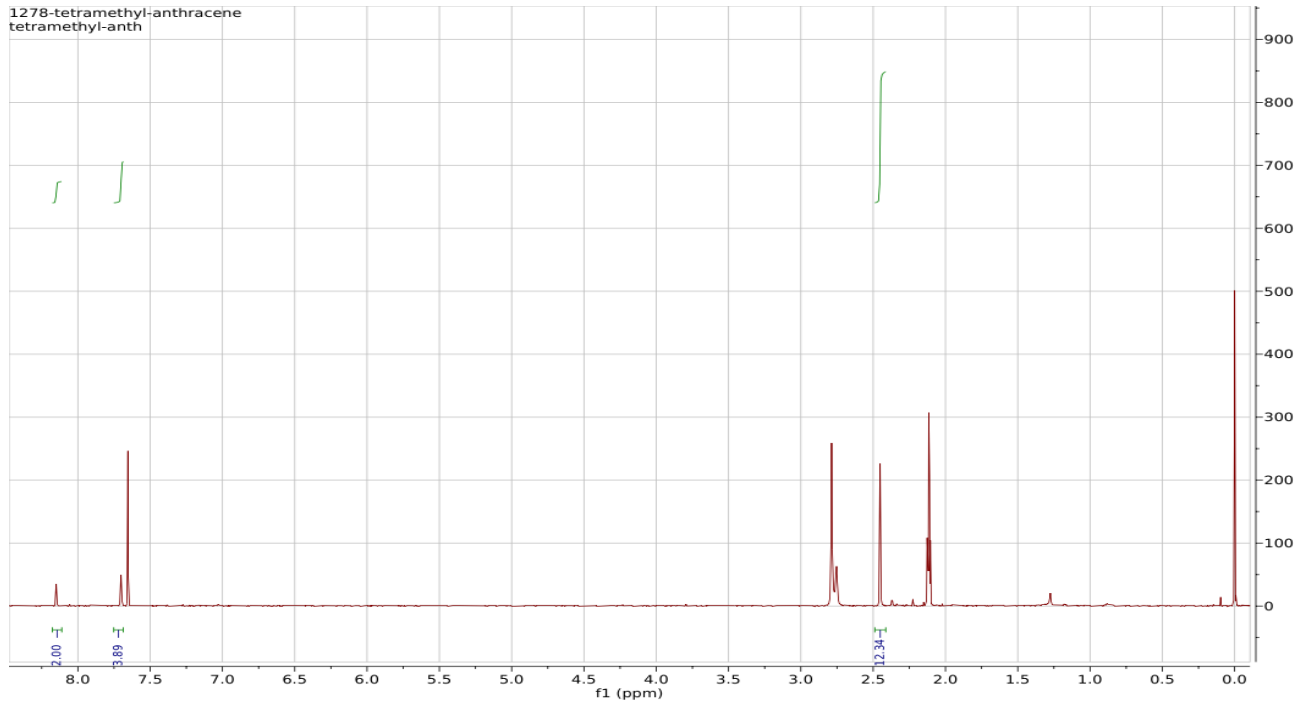
Invented by: _____

Recorded by: _____

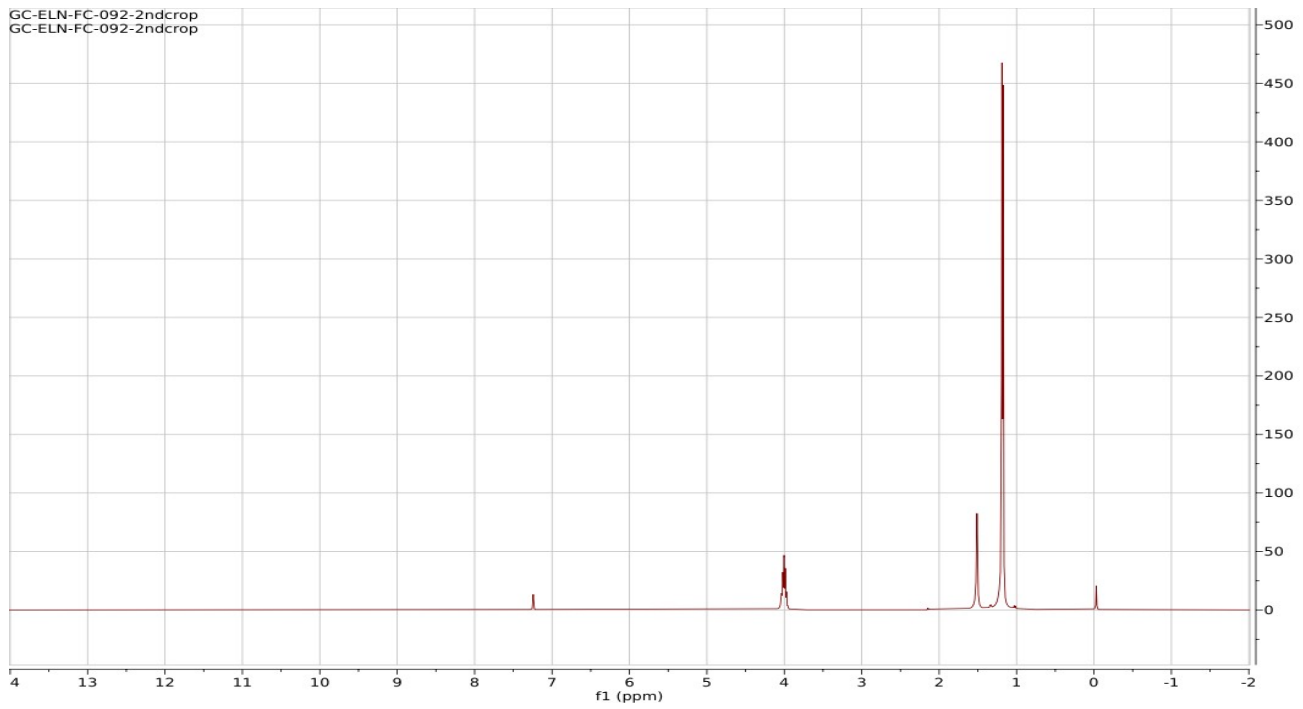
Date _____

To Page N _____

Spectra:



GC-ELN-FC-092-2ndcrop
GC-ELN-FC-092-2ndcrop

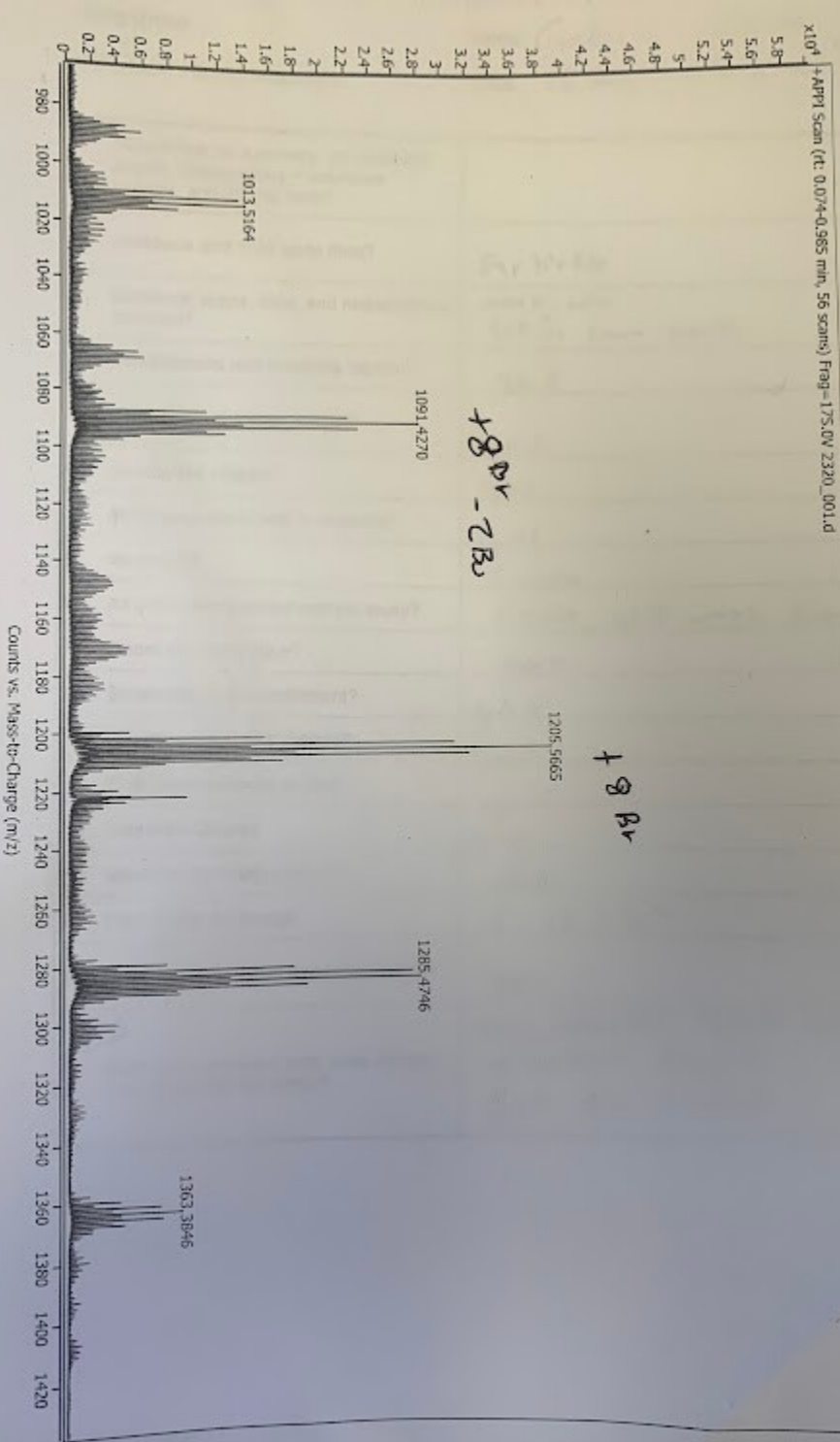


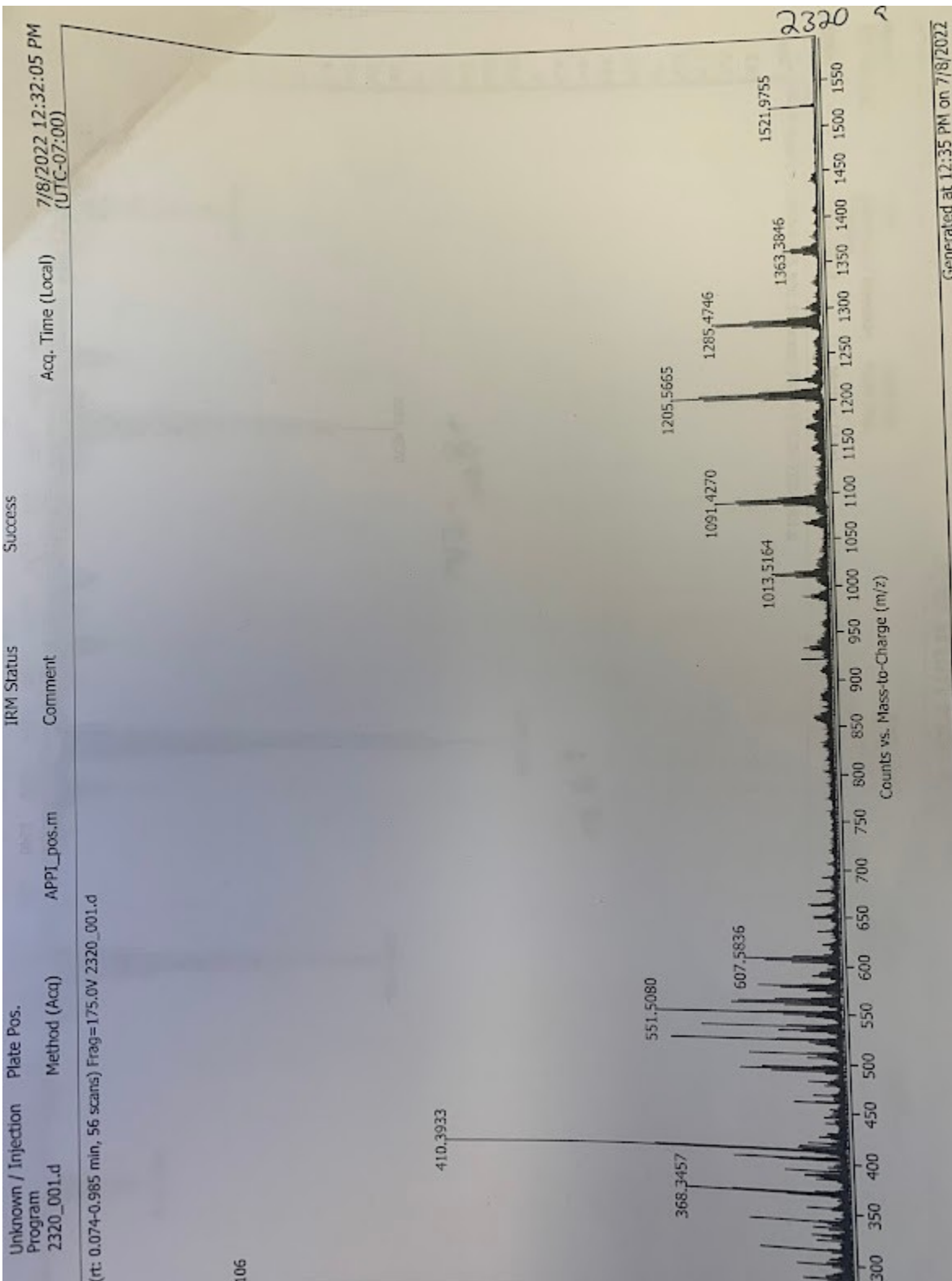
.

Spectrum Plot Report



Name	2320	Rack Pos.	Instrument	Operator
Inj. Vol. (µl)	Unknown / Injection	Plate Pos.	IRM Status	
Data File	2320_001.d	Method (Acq)	Comment	Acq. Time (Local)
				7/8/2022 12:32:05 PM
				(UTC-07:00)





Weekly inspection:

King Group

Individual Weekly Inspection

Name: Gabriel Cabrera
Date: Today! (7-11-22)

1. Report has (a) summary, (b) notebook pages, (c) supporting + reference spectra, and (d) this form?	
2. Notebook and TOC up to date?	partially
3. Notebook scans, data, and metadata are archived?	partially yes, have some
4. All containers and reactions labeled?	yes
5. All samples correctly labeled?	yes
6. All volatiles capped?	yes
7. All flammables stored in cabinets?	yes
8. No clutter?	some
9. All glassware washed and put away?	some, will wash tomorrow
10. Floors and aisle clear?	yes
11. Secondary spill containment?	yes
12. Hoses secured with clamps?	yes
13. Is all waste properly stored?	yes
14. Cylinders secure?	yes
15. Manifold operating pressure:	600
16. Date of last oil change:	5-16-22
17. Did you always wear appropriate PPE?	yes
18. Aside from assigned jobs, what did you do to make the lab better?	Regulates THE STILLS MAPPED FLOORS Got N ₂ tanks