EXAMPLE

92/100

Unknown Report Sheet
Chemistry 136
Spring 1996

(One form for each component if a multi-component unknown is given)

Name: [redacted]

Date: June 4, 1996 Drawer #: C13

Unknown Number: 23 (2nd compound) Molecular weight: 126.11 g/mole

1. Physical Appearance (color, form of the crystals, etc.):

after purification { brown liquid before the NMR
after rotoraping of $CDCl_3$ from NMR it was a brown solid

impure: a brown liquid w/brown solid

2. TLC Analysis (SiO_2, solvent, UV lamp or stain)

TLC analysis was run on SiO_2 plates. Several solvent systems were tried.

1st: 75% Hexanes/25% Ethyl acetate → gave two spots with rf = 0.70 and rf = 0.30. The slower compound was a smear and the place where it was spotted on the plate showed brown compound still. An attempt to totally dissolve the compound was made. I found a 1:1 mixture of $CHCl_3$ and MeOH to be the best, resulting in a clear, dark red brown liquid. Further TLC were carried out using these solvents. Systems with both 1% and 2% MeOH gave extremely high rf's for the faster compound. Using only $CHCl_3$ gave and rf = 0.75 for this compound while the other one remained stationary. All TLC's were found to stain best with iodine

x5

3. Physical constants (mp, bp, corrected values): bp. (liquid) = 90-92°C no value available
M.P. (solid) = 50-55°C (lit value = 66.5-67.0°C sealed)

4. Formula: $C_6H_6O_3$ Degrees of unsaturation: 6 - 3 + 1 = 4

5. Method of separation or purification:

A silica column chromatography purification was carried out using two different solvent systems. First ~1ml of unknown was dissolved in 1ml of 1:1 mixture of $CHCl_3$ and MeOH and added to the column. The column was then flushed with 100 ml of $CHCl_3$ followed by 100ml of 20% MeOH/80% $CHCl_3$. The 1st solvent system eluted the 1st compound in Fractions 25-40. This was determined by running TLC tests and staining with iodine. The second system eluted the 2nd compound which was stationary in pure $CHCl_3$. TLC tests on this showed it was present in fractions 42-50. This report is on this 2nd compound. Fractions 42-50 were combined, rotoraped, and then vacuum pumped down to ensure all solvent was removed from the compound.

g (SiO_2)/g (SAMPLE)

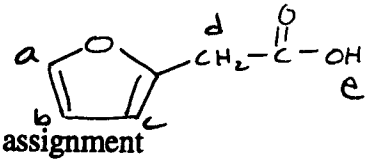
19

6. IR (attach the original at the end of the report)

band (cm^{-1})	assignment
broad band from 3500-2500	Carboxylic acid O-H
3055.2	ring C-H stretch
2967.9	assymetrical C-H stretch
2677.4	symmetrical C-H stretch
1718.1	carbonyl C=O stretch
1604.7	C=C stretch
1506.2	in plan C-H stretch for alkene
1421.7	O-H stretch
1265.9	C-O stretch
1148.3	C-O-C assymetrical stretch
1013.2	C-O-C symmetrical stretch
949.7 and 896.8	OOP C
741.8	OOP C-H bend

+10

NMR Spectra (attach the originals at the end of the report)

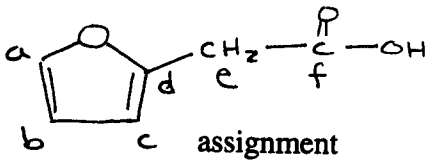


7. ^1H

	Chemical Shift	integration	multiplicity/coupling constant	assignment
a	7.36	1	S (slight triplet) J = 0.706	CH = C
e?	6.87	2	broad singlet (MAY INCLUDE SOME H_2O)	carboxylic acid proton (but has 2 Hs)
	6.279	2	(b) 6.4→t (c) 6.3→d J = 19.49, 3.02	
	4.85			H_2O
	4.62			impurity - compound A
d	3.71	2	S	$R-CH_2-\overset{O}{\overset{\|}{C}}$
	3.44	1	S	
	2.3-0.92			impurity - compound A

+10

The proton NMR does not integrate according to the given molecular structure. The splitting patterns do not match up either



8. ^{13}C Chemical Shift assignment

174.61	f
147.21	d
142.27	a
110.60	c
108.37	b
33.81	e

+ 10

45

SOME IMPURITIES IN SPECTRA

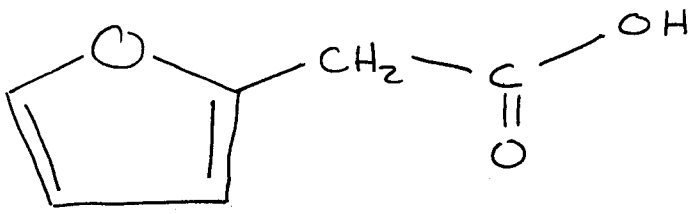
9. Other Data (UV, GCMS, Complex NMR, etc.)

none taken

10. Derivatives: none carried out

/

11. Proposed structure:



+10

10 2-Furanacetic Acid

12. Relevant literature data supporting structure:

1) Silverstein, Bassler, Morril Spectrometric Identification of Organic Compounds 5th Ed.

2) Sadtler Spectral Index - Sadtler Research Laboratories Only IR spectra was available to compare

+5

13. Flow diagram of logic used in deriving structure:

(less than 1 page in most cases. By citing specific data you have obtained, prove that your structure is correct)

Look at Formula $C_6H_6O_3$

$\Rightarrow$ DUS = 6 − 3 + 1 = 4 +20

IR: C=O stretch at 1718
absense of methyl absorptions at 1400 and 1375
Carboxylic acid stretch at 3400 − 2400cm
C=C stretch
C−O stretch → chemical shifts & coupling constants are diagnostic for a heterocyclic aromatic

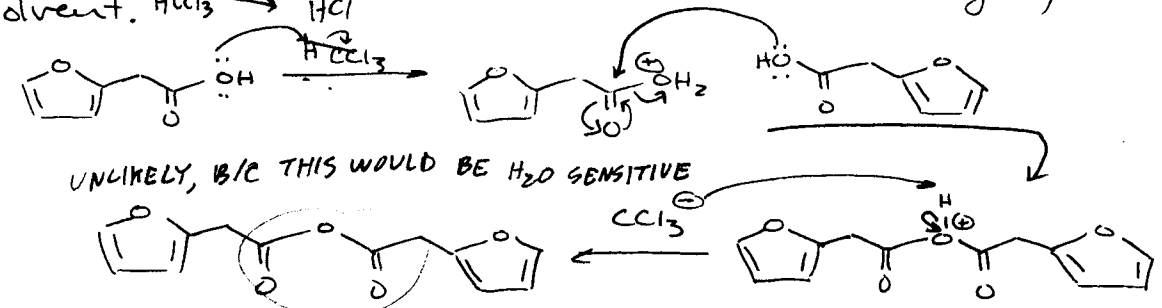
^{1}H NMR: Is screwed up
* shows an integration equal to 8 Hydrogens → VARIABLE, SOMETIMES HARD TO SEE
* absense of carboxylic proton peak in usual 13−10 ppm range
* shows three singlets, which should not happen for the proposed structure. shows 4 CH's, CH_2 coupled to nothing and a CH_2 couple to CH. Doesn't match up.

^{13}C NMR
Carbonyl peak at 174 → can be ester, carboxylic acid. Confirmed by IR $\Rightarrow$ Carboxylic Acid
* 2 peaks at 140 → alkene α to a heteroatom
* 2 peaks at 110 → alkene
* 1 peak at 33 → characteristic of methylene $-CH_2-$

* Could figure out by myself. Lance gave me the specific structure for 2-furanacetic acid.

The only thing I could think would happen is it reacted w/ the deuterated chloroform. This is further substantiated since it changed from a liquid to a solid after drying off NMR solvent. $HCCl_3 \xrightarrow{H_2O} HCl$

Example rxn



would give 6 carbon peaks
and 4 H peaks → or 5 if methylene H's are different

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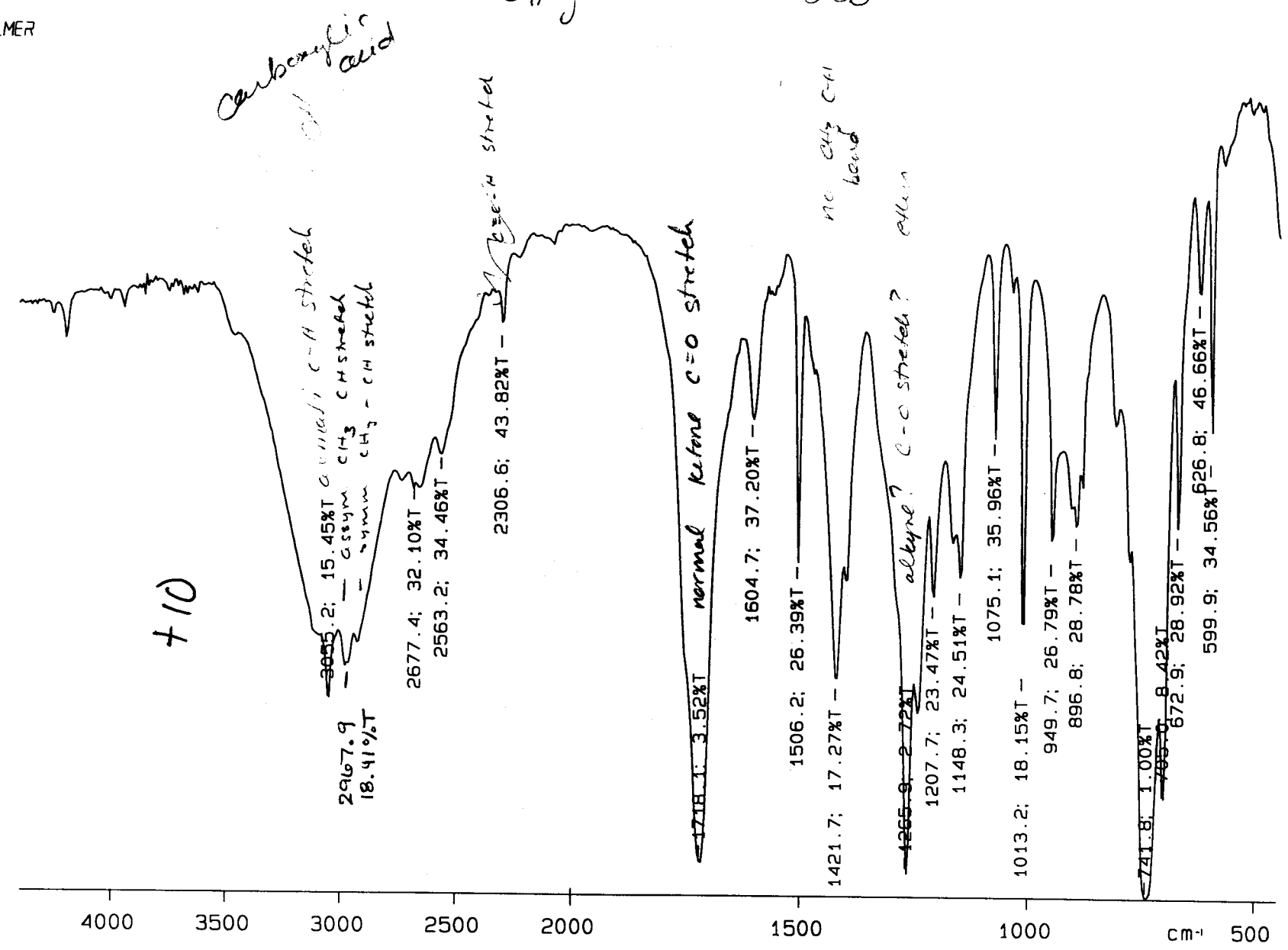
PERKIN ELMER



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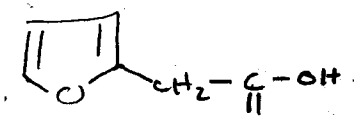
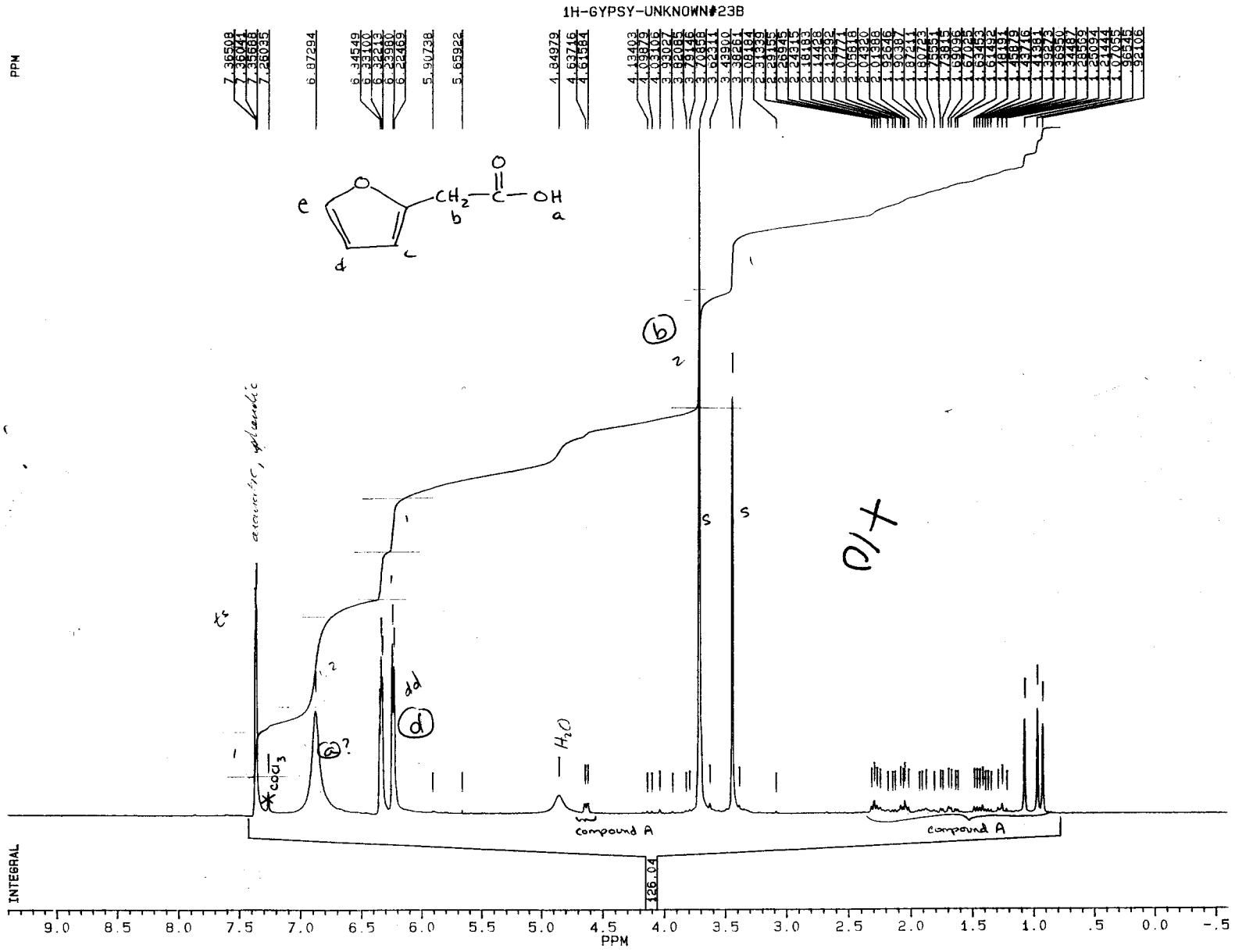


5 H peaks
6 C peaks

2-Furanacetic acid
m.p = 66.5 – 67.0 °C
MW = 126.11

$C_6H_6O_3$

DUS = 6 - 3 + 1 = 4



BRUKER

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DR	12
DW	124
FW	5100
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LB	.300
NC	-1
CX	32.00
CY	18.00
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F2	-.598P
MI	0.0
HZ/CM	62.543
PPM/CM	.313
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SR	2340.56
D1	1.0000000
PO	2.4
RGA	
RD	0.0
PW	0.0
DE	155.0
NS	8
DS	0

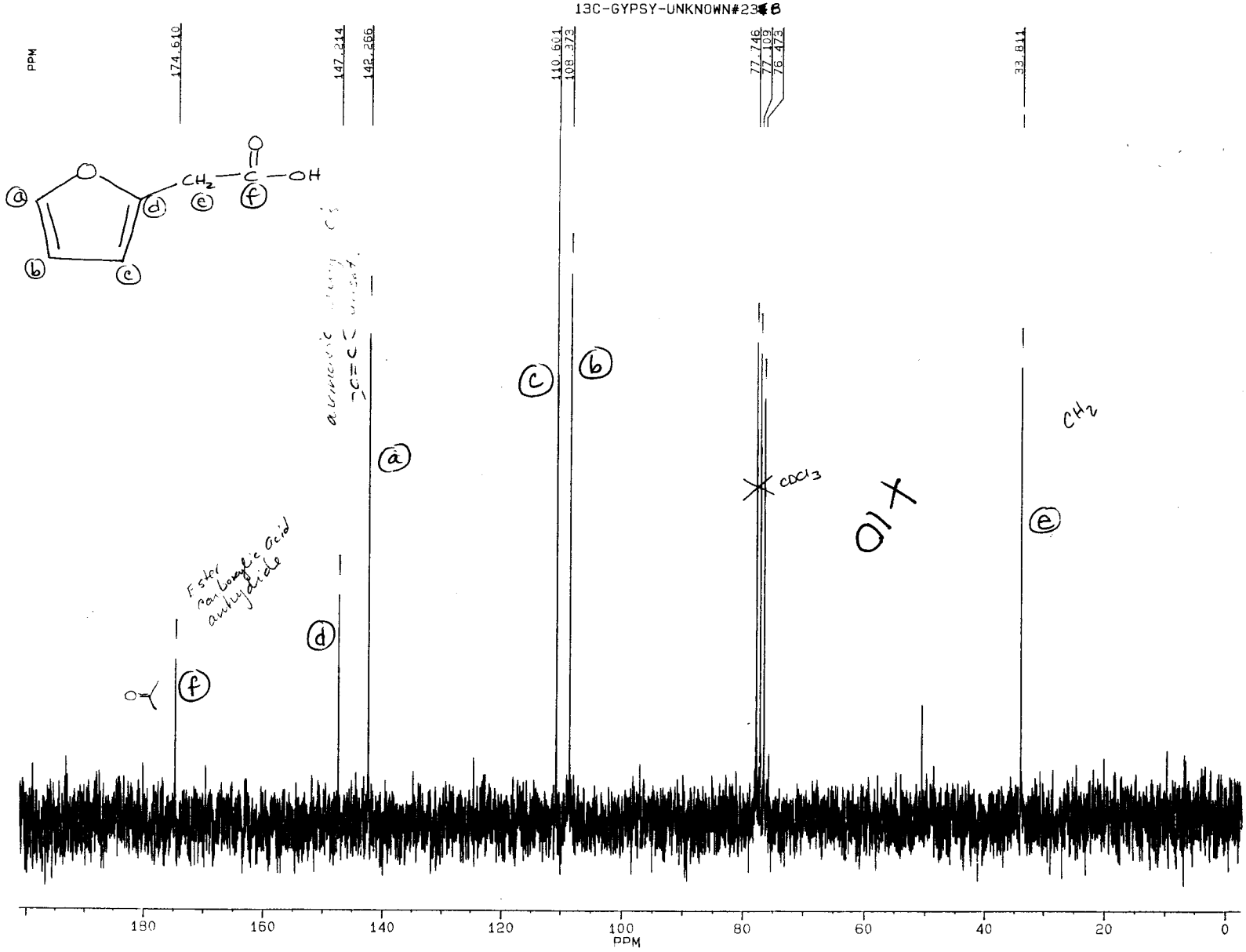
$C_6H_6O_3$

$C_6H_6O_3$

DUS = 4

6 different Carb

13C-GYPSY-UNKNOWN#23[illegible]B



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AQ	1.311
RG	200
NS	64
DE	50.0
DR	12
DW	40
FW	15700
O2	3545.000
DP	17H BB
LB	1.000
NC	1
CX	32.00
CY	18.00
F1	201.034P
F2	-2.969P
MI	0.0
HZ/CM	320.816
PPM/CM	6.375
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SR	3315.00
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S1	17H
D5	.0010000
P0	2.0
RGA	
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