

EXAMPLE

92
100

Unknown Report Sheet

Chemistry 136

Spring 1996

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

Name: _____

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 rotovaping 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 plate 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 an $rf = 0.75$ for this compound while the other one remained stationary. All TLC's were found to stain best with iodine

3. Physical constants (mp, bp, corrected values): bp. (liquid) = 90-92°C no value available

4. Formula: $C_{10}H_{16}O_3$ m.p. (solid) = 50-55°C (lit value = 66.5-67.0°C solid)
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 ~~the~~ 1 ml of unknown was dissolved in 1 ml 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 100 ml 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~~ 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, rotovaped, and then vacuum pumped down to ensure all solvent was removed from the compound.

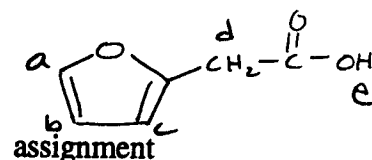
$$g(SiO_2) / g(SAMPLE)$$

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	asymmetrical C-H stretch
2677.4	symmetrical C-H stretch
1718.1	carbonyl C=O stretch
1604.7	C=C stretch
1506.2	in plane C-H stretch for alkene
1421.7	O-H stretch
1265.9	C-O stretch
1148.3	C-O-C asymmetrical 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)

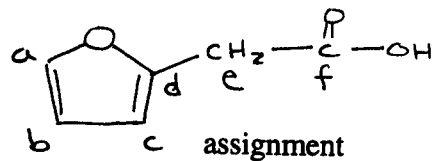


	Chemical Shift	integration	multiplicity/coupling constant	assignment
a	7.36	1	s (slight triplet) J=0.706	CH=C
e?	6.87	2	MAY INCLUDE SOME H ₂ O broad singlet	carboxylic acid proton (but has 2H's)
	6.279	2	6.4 → t (b) dd (c) J=19.49, 3.02	
	4.85			H ₂ O
	4.62			impurity - compound A
d	3.71	2	s +10	R-CH ₂ -C
	3.44	1	s	
	2.3-0.92			impurity - compound A

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



174.61	f
147.21	d
142.27	a
110.60	c
108.37	b
33.81	e
	+ 10

45

SOME IMPURITIES IN SPECTRA

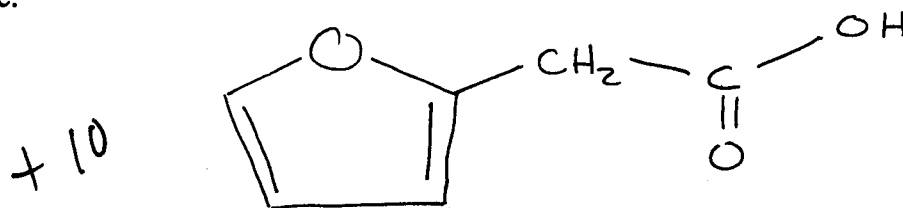
9. Other Data (UV, GCMS, Complex NMR, etc.)

none taken

10. Derivatives: none carried out

/

11. Proposed structure:



10

2-Furanacetic Acid

12. Relevant literature data supporting structure:

- 1) Silverstein, Bassler, Morrill 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

absence of methyl absorptions at 1400 and 1375

Carboxylic acid stretch at 3400 - 2400 cm

C=C stretch

C-O stretch

→ ~~the~~ chemical shifts & coupling constants are diagnostic for a heterocyclic aromatic

¹H NMR: Is screwed up

* shows an integration equal to 8 Hydrogens

→ VARIABLE, SOMETIMES HARD TO SEE

* absence 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, CH₂ coupled to nothing and a CH₂ couple to CH. Doesn't match up.

¹³C NMR

Carbonyl peak at 174 → can be ester, carboxylic acid. Confirmed by IR ⇒ Carboxylic Acid

* 2 peaks at 140 → alkene α to a heteroatom

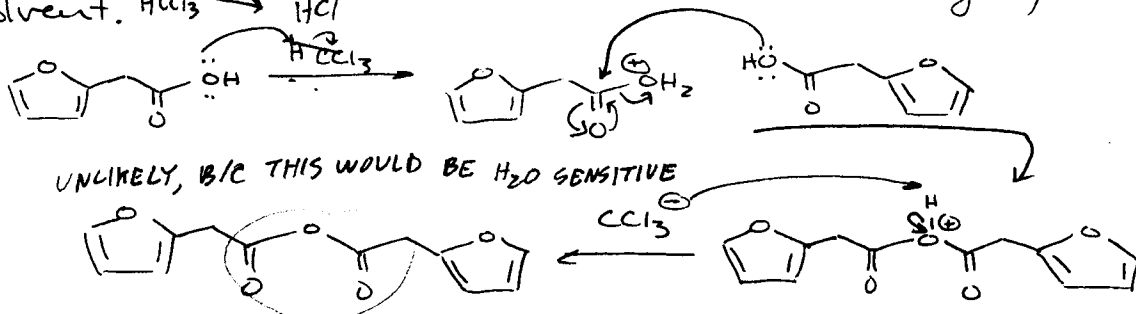
* 2 peaks at 110 → alkene

* 1 peak at 33 → characteristic of methylene -CH₂-

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

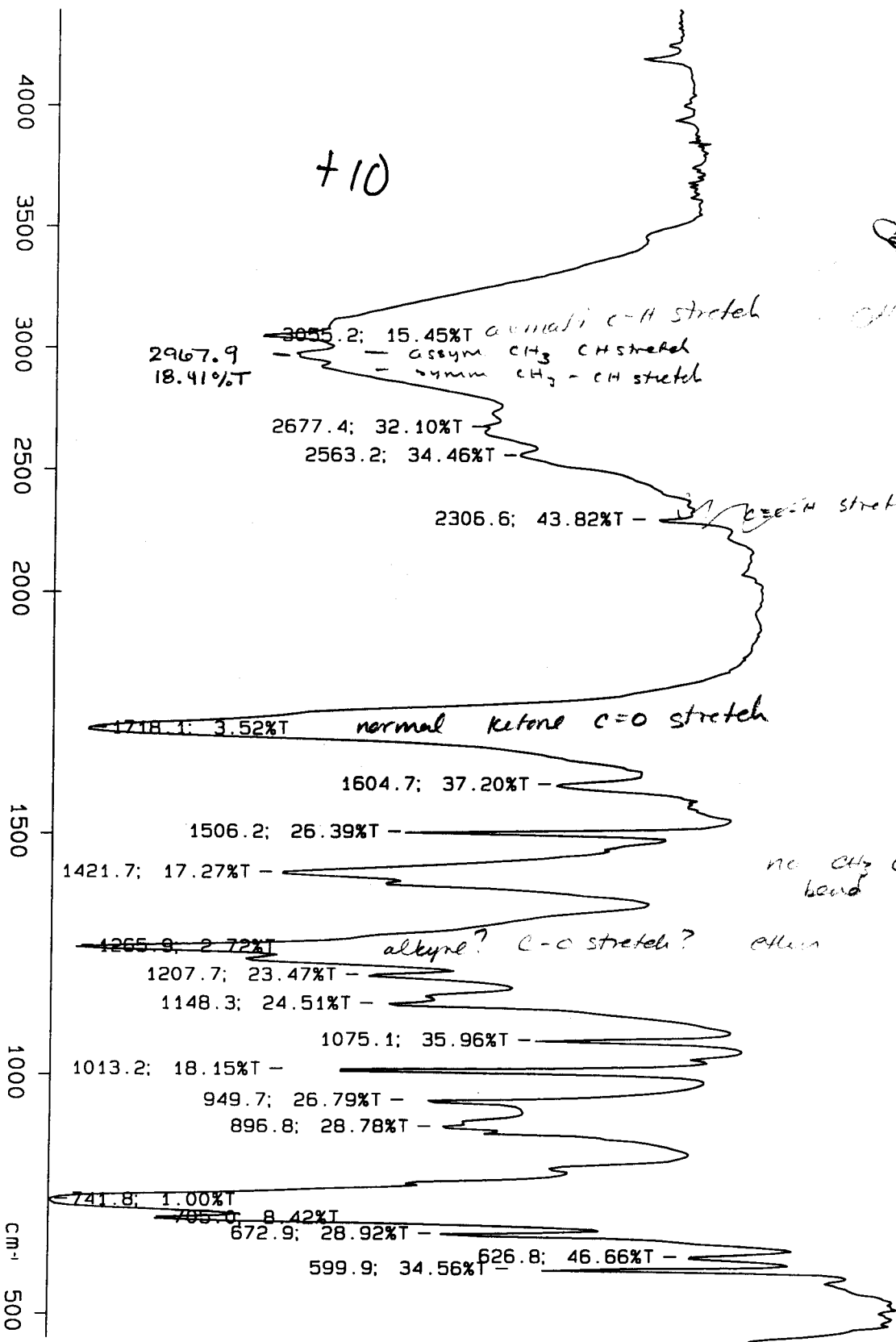
The only thing I could think would happen is it reacted w/ the deuterated chloroform. This is further substantiated since it changed from a liquid to a solid after drying off NMR solvent. $HCCl_3 \xrightarrow{H_2O} HCl$

example rxn



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

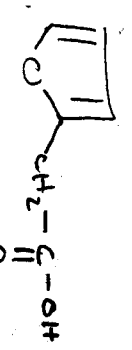
96/05/23 22:16 unspecified
SCAN: 1 scan, 4.0cm-1



Carbonyl's

copy unknown 7/05

+10



5 H+ peaks
to C peaks

2-Furanacetic acid

M.P = 66.5 - 67.0 °C

MW = 126.11

C₆H₆O₃

DOS = 6-3+1=4



C6H6O3

M2120F.118
AU PPO6:
X00.AU
DATE 21-5-96
TIME 13.39

SF 200.132
Q1 3545.000
SI 16384
TD 16384
SM 4032.258
HZ/PT 492

PW 0.0
RD 0.0
AG 2.032
RG 4
NS 8

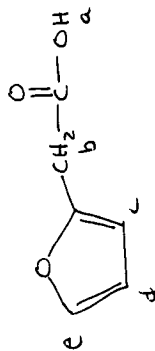
DE 155.0
DR 12
DM 124
FW 5100
Q2 0.0
DP 63L P0

LB -1.300
NC 1
CX 32.00
CY 18.00
F1 9.402P
F2 -598P
MI 0.0
HZ/CM 62.543
PPM/CM .313
IS 4
SR 2340.56

D1 1.0000000
P0 2.4
RGA 0.0
RD 0.0
PW 155.0
NS 8
DS 0

1H-GYPSY-UNKNOWN#23B

7.36508	7.36041	7.35688	7.26093	7.07294	6.34549	6.33100	6.32213	6.23980	6.22469	5.90738	5.65922	4.84929	4.63716	4.61584	4.13403	4.09879	4.03108	3.9027	3.8085	3.79146	3.7311	4.3990	3.8261	3.8184	3.3374	3.299	3.285	3.281	3.277	3.273	3.269	3.265	3.261	3.257	3.253	3.249	3.245	3.241	3.237	3.233	3.229	3.225	3.221	3.217	3.213	3.209	3.205	3.201	3.197	3.193	3.189	3.185	3.181	3.177	3.173	3.169	3.165	3.161	3.157	3.153	3.149	3.145	3.141	3.137	3.133	3.129	3.125	3.121	3.117	3.113	3.109	3.105	3.101	3.097	3.093	3.089	3.085	3.081	3.077	3.073	3.069	3.065	3.061	3.057	3.053	3.049	3.045	3.041	3.037	3.033	3.029	3.025	3.021	3.017	3.013	3.009	3.005	3.001	2.997	2.993	2.989	2.985	2.981	2.977	2.973	2.969	2.965	2.961	2.957	2.953	2.949	2.945	2.941	2.937	2.933	2.929	2.925	2.921	2.917	2.913	2.909	2.905	2.901	2.897	2.893	2.889	2.885	2.881	2.877	2.873	2.869	2.865	2.861	2.857	2.853	2.849	2.845	2.841	2.837	2.833	2.829	2.825	2.821	2.817	2.813	2.809	2.805	2.801	2.797	2.793	2.789	2.785	2.781	2.777	2.773	2.769	2.765	2.761	2.757	2.753	2.749	2.745	2.741	2.737	2.733	2.729	2.725	2.721	2.717	2.713	2.709	2.705	2.701	2.697	2.693	2.689	2.685	2.681	2.677	2.673	2.669	2.665	2.661	2.657	2.653	2.649	2.645	2.641	2.637	2.633	2.629	2.625	2.621	2.617	2.613	2.609	2.605	2.601	2.597	2.593	2.589	2.585	2.581	2.577	2.573	2.569	2.565	2.561	2.557	2.553	2.549	2.545	2.541	2.537	2.533	2.529	2.525	2.521	2.517	2.513	2.509	2.505	2.501	2.497	2.493	2.489	2.485	2.481	2.477	2.473	2.469	2.465	2.461	2.457	2.453	2.449	2.445	2.441	2.437	2.433	2.429	2.425	2.421	2.417	2.413	2.409	2.405	2.401	2.397	2.393	2.389	2.385	2.381	2.377	2.373	2.369	2.365	2.361	2.357	2.353	2.349	2.345	2.341	2.337	2.333	2.329	2.325	2.321	2.317	2.313	2.309	2.305	2.301	2.297	2.293	2.289	2.285	2.281	2.277	2.273	2.269	2.265	2.261	2.257	2.253	2.249	2.245	2.241	2.237	2.233	2.229	2.225	2.221	2.217	2.213	2.209	2.205	2.201	2.197	2.193	2.189	2.185	2.181	2.177	2.173	2.169	2.165	2.161	2.157	2.153	2.149	2.145	2.141	2.137	2.133	2.129	2.125	2.121	2.117	2.113	2.109	2.105	2.101	2.097	2.093	2.089	2.085	2.081	2.077	2.073	2.069	2.065	2.061	2.057	2.053	2.049	2.045	2.041	2.037	2.033	2.029	2.025	2.021	2.017	2.013	2.009	2.005	2.001	1.997	1.993	1.989	1.985	1.981	1.977	1.973	1.969	1.965	1.961	1.957	1.953	1.949	1.945	1.941	1.937	1.933	1.929	1.925	1.921	1.917	1.913	1.909	1.905	1.901	1.897	1.893	1.889	1.885	1.881	1.877	1.873	1.869	1.865	1.861	1.857	1.853	1.849	1.845	1.841	1.837	1.833	1.829	1.825	1.821	1.817	1.813	1.809	1.805	1.801	1.797	1.793	1.789	1.785	1.781	1.777	1.773	1.769	1.765	1.761	1.757	1.753	1.749	1.745	1.741	1.737	1.733	1.729	1.725	1.721	1.717	1.713	1.709	1.705	1.701	1.697	1.693	1.689	1.685	1.681	1.677	1.673	1.669	1.665	1.661	1.657	1.653	1.649	1.645	1.641	1.637	1.633	1.629	1.625	1.621	1.617	1.613	1.609	1.605	1.601	1.597	1.593	1.589	1.585	1.581	1.577	1.573	1.569	1.565	1.561	1.557	1.553	1.549	1.545	1.541	1.537	1.533	1.529	1.525	1.521	1.517	1.513	1.509	1.505	1.501	1.497	1.493	1.489	1.485	1.481	1.477	1.473	1.469	1.465	1.461	1.457	1.453	1.449	1.445	1.441	1.437	1.433	1.429	1.425	1.421	1.417	1.413	1.409	1.405	1.401	1.397	1.393	1.389	1.385	1.381	1.377	1.373	1.369	1.365	1.361	1.357	1.353	1.349	1.345	1.341	1.337	1.333	1.329	1.325	1.321	1.317	1.313	1.309	1.305	1.301	1.297	1.293	1.289	1.285	1.281	1.277	1.273	1.269	1.265	1.261	1.257	1.253	1.249	1.245	1.241	1.237	1.233	1.229	1.225	1.221	1.217	1.213	1.209	1.205	1.201	1.197	1.193	1.189	1.185	1.181	1.177	1.173	1.169	1.165	1.161	1.157	1.153	1.149	1.145	1.141	1.137	1.133	1.129	1.125	1.121	1.117	1.113	1.109	1.105	1.101	1.097	1.093	1.089	1.085	1.081	1.077	1.073	1.069	1.065	1.061	1.057	1.053	1.049	1.045	1.041	1.037	1.033	1.029	1.025	1.021	1.017	1.013	1.009	1.005	1.001	0.997	0.993	0.989	0.985	0.981	0.977	0.973	0.969	0.965	0.961	0.957	0.953	0.949	0.945	0.941	0.937	0.933	0.929	0.925	0.921	0.917	0.913	0.909	0.905	0.901	0.897	0.893	0.889	0.885	0.881	0.877	0.873	0.869	0.865	0.861	0.857	0.853	0.849	0.845	0.841	0.837	0.833	0.829	0.825	0.821	0.817	0.813	0.809	0.805	0.801	0.797	0.793	0.789	0.785	0.781	0.777	0.773	0.769	0.765	0.761	0.757	0.753	0.749	0.745	0.741	0.737	0.733	0.729	0.725	0.721	0.717	0.713	0.709	0.705	0.701	0.697	0.693	0.689	0.685	0.681	0.677	0.673	0.669	0.665	0.661	0.657	0.653	0.649	0.645	0.641	0.637	0.633	0.629	0.625	0.621	0.617	0.613	0.609	0.605	0.601	0.597	0.593	0.589	0.585	0.581	0.577	0.573	0.569	0.565	0.561	0.557	0.553	0.549	0.545	0.541	0.537	0.533	0.529	0.525	0.521	0.517	0.513	0.509	0.505	0.501	0.497	0.493	0.489	0.485	0.481	0.477	0.473	0.469	0.465	0.461	0.457	0.453	0.449	0.445	0.441	0.437	0.433	0.429	0.425	0.421	0.417	0.413	0.409	0.405	0.401	0.397	0.393	0.389	0.385	0.381	0.377	0.373	0.369	0.365	0.361	0.357	0.353	0.349	0.345	0.341	0.337	0.333	0.329	0.325	0.321	0.317	0.313	0.309	0.305	0.301	0.297	0.293	0.289	0.285	0.281	0.277	0.273	0.269	0.265	0.261	0.257	0.253	0.249	0.245	0.241	0.237	0.233	0.229	0.225	0.221	0.217	0.213	0.209	0.205	0.201	0.197	0.193	0.189	0.185	0.181	0.177	0.173	0.169	0.165	0.161	0.157	0.153	0.149	0.145	0.141	0.137	0.133	0.129	0.125	0.121	0.117	0.113	0.109	0.105	0.101	0.097	0.093	0.089	0.085	0.081	0.077	0.073	0.069	0.065	0.061	0.057	0.053	0.049	0.045	0.041	0.037	0.033	0.029	0.025	0.021	0.017	0.013	0.009	0.005	0.001
---------	---------	---------	---------	---------	---------	---------	---------	---------	---------	---------	---------	---------	---------	---------	---------	---------	---------	--------	--------	---------	--------	--------	--------	--------	--------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------	-------



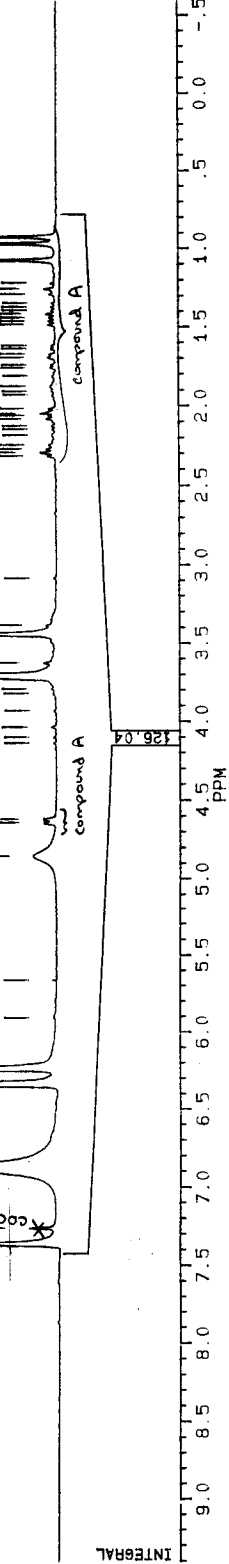
(b)

aromatic, aliphatic

10+

(p)

H₂O



C₆H₆O₃

DVS = 4

6 different carb



M2124F.118
AU BR06
X02.AU
DATE 21-5-96
TIME 13.46

SF 50.323
F1 8349.000
SI 32768
TD 32768
SW 12500.000
HZ/PT :763

PW 0.0
RD 0.0
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
WI 0.0
HZ/CM 320.616
PPM/CM 6.375
IS 1
SR 3315.00

D1 2.0000000
S1 17H
D5 .0010000
P0 2.0
RGA 0.0
RD 0.0
PW 0.0
DE 50.0
NS 64
DS 0

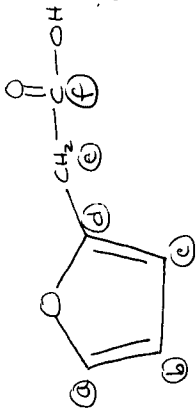
13C-GYPSY-UNKNOWN#236B

77.746
76.473

110.601
108.373

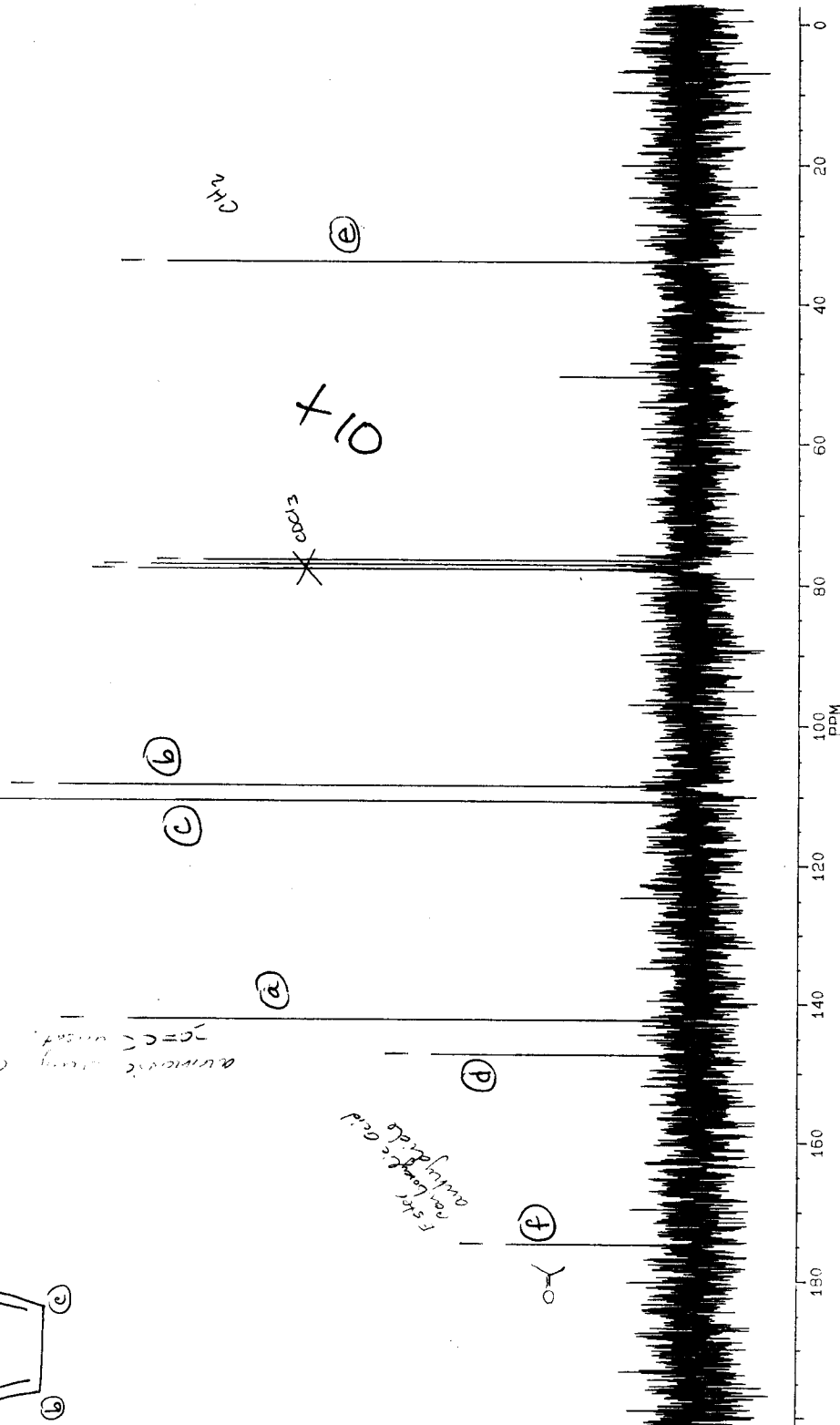
148.266
147.214

174.610



aromatic ring C
C=O carbonyl

ester carboxylic acid



33.811

CH2

e

174.6

77.746

b

c

a

f

f