

# Microwave-Assisted Concise Total Syntheses of Quinazolinobenzodiazepine Alkaloids

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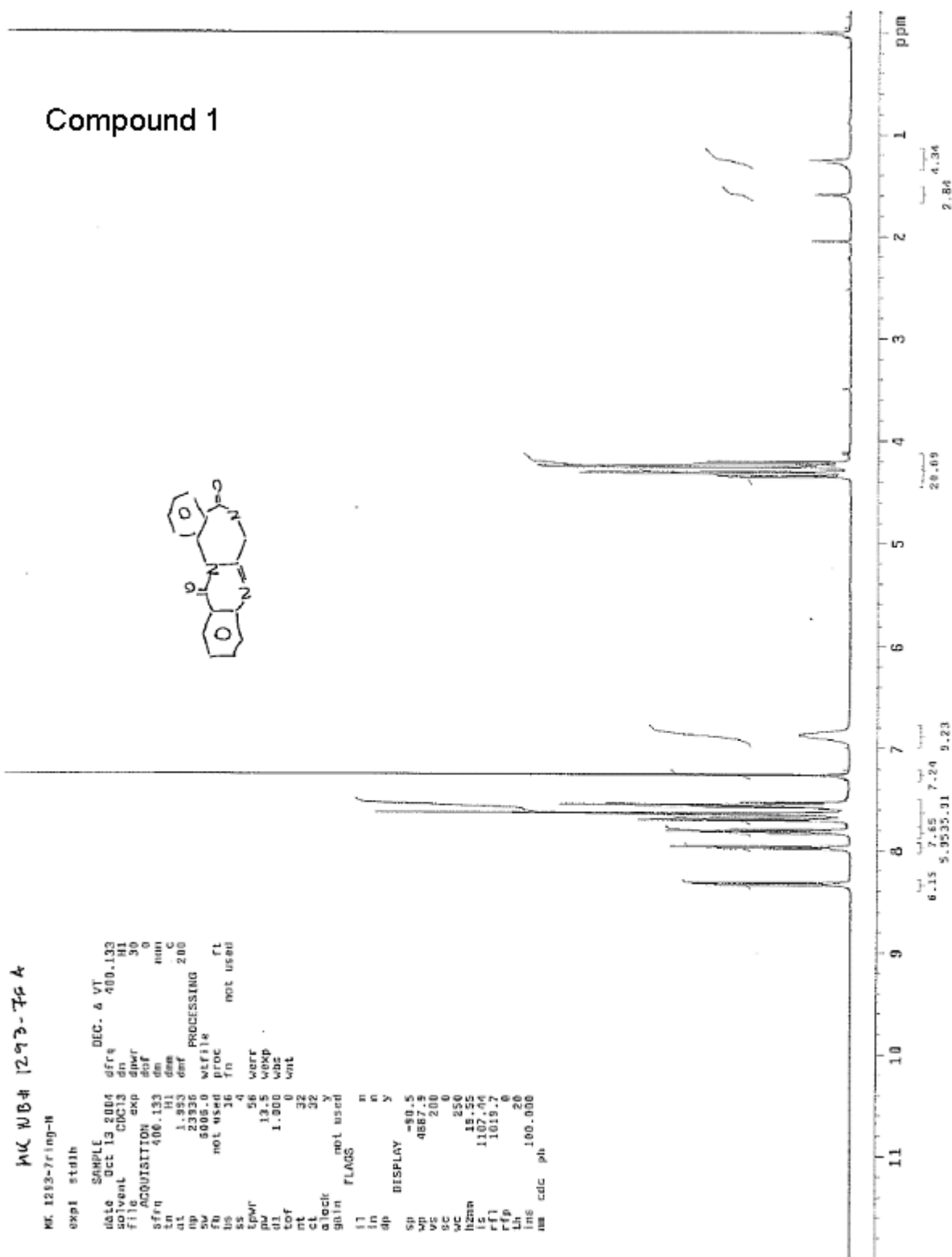
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## SUPPORTING INFORMATION

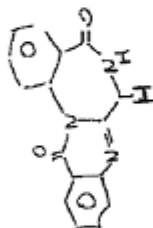
**General and materials.** All reagents and solvents were purchased and used without further purification. Biotage Smith Synthesizer<sup>TM</sup> was used for microwave reactions. Irradiation was initiated at 300 W to raise the temperature to the set point and then power was applied at intervals and levels to maintain the desired temperature. Reactions were run in sealed vessels. Reaction times reported included time for the vial to ramp to the desired temperature. The product isolation was performed on prep-TLC plates or on preparative HPLC. The Preparative HPLC was carried out employing a ProntoSIL 120-10-C18 column (50x20 mm). The flow rate was at 44 mL/min utilizing an acetonitrile/water mobile phase. <sup>1</sup>H NMR (400 MHz) and <sup>13</sup>C NMR (100 MHz) spectra were recorded in CDCl<sub>3</sub> solution (unless otherwise stated). Chemical shifts are given in parts per million (*ppm*) downfield from tetramethylsilane (TMS) as an internal reference, and coupling constants (*J*-values) are in hertz (Hz). Low-resolution mass spectra were recorded in the ES<sup>+</sup> mode. The high-resolution mass spectra (HRMS) were recorded in the FAB mode.



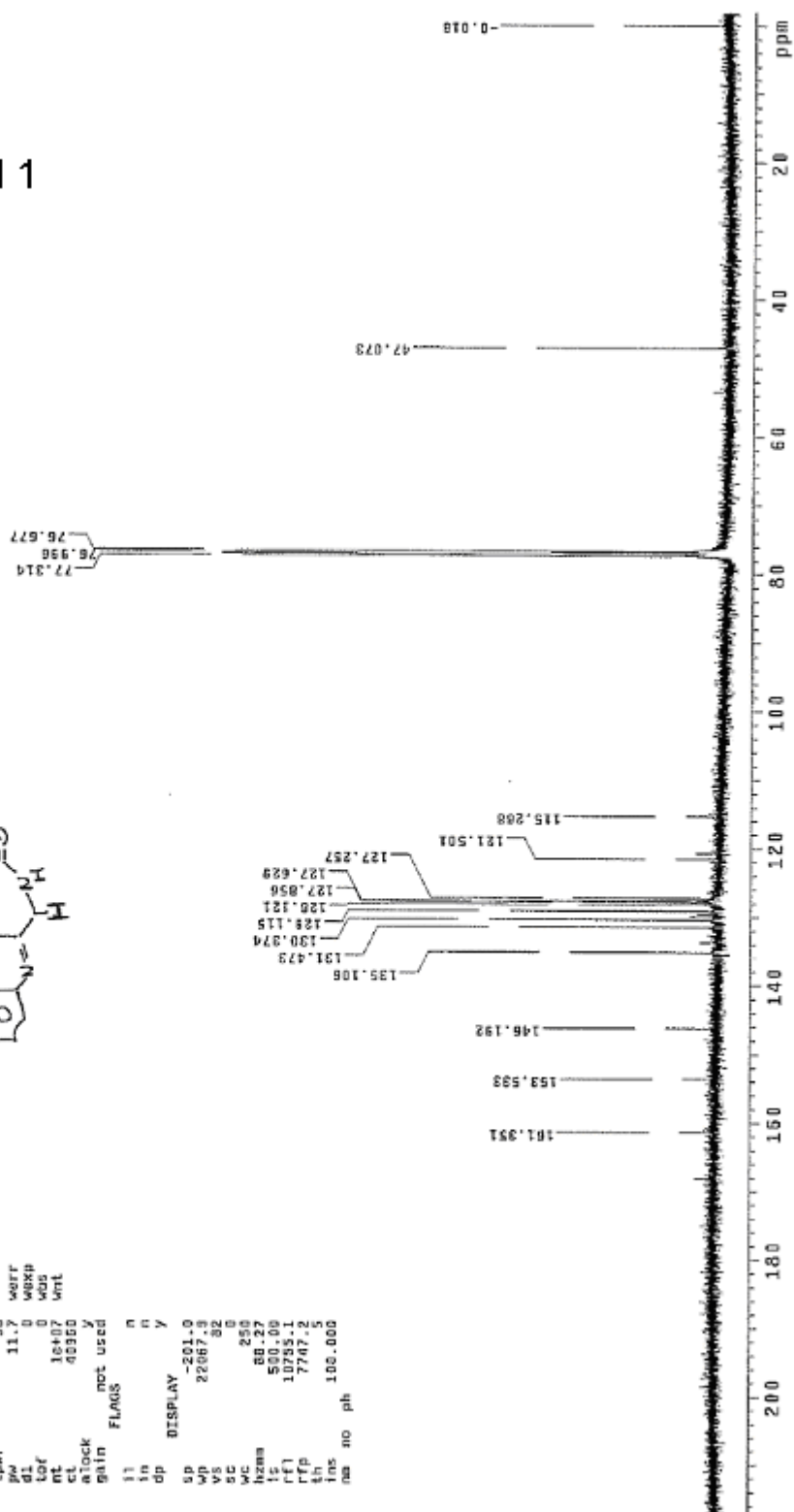
77-1  
28-13C  
MK WB-1292-76 A

exp1 61d13c

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filn	ACQUISITION	exp	37
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fb	13000	wtfile	not used
bs	16	proc	ft
ss	4	fn	not used
tpwr	58	weff	
pw	11.7	wexp	
d1	0	wdw	
tdf	10407	wdw	
ct	45350	wdw	
clock	not used		
gain	not used		
fl	0	flags	
in	0		
dp	0		
DISPLAY			
sp	-201.0		
wp	22067.8		
vs	32		
sc	0		
wc	250		
ncmm	50.2		
rf1	10755.1		
rf2	7747.2		
ti	5		
na	100.000		
na	no		
na	ph		



Compound 1

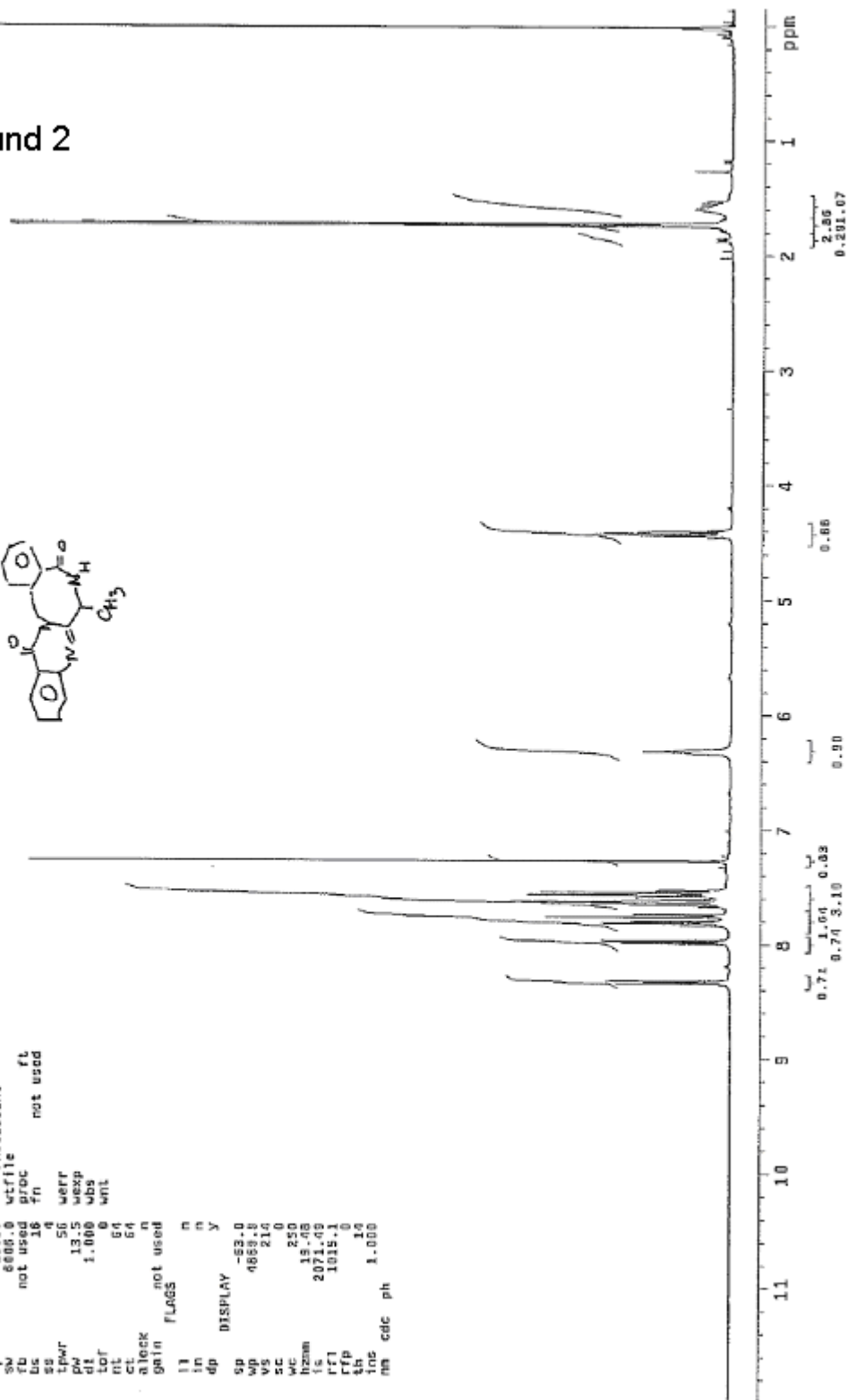
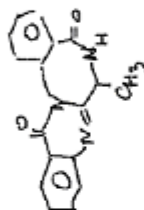


78-1 JFL 11/07/00 MK WD# 1296-76 B

exp1 sidh

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 file CDC13 exp 30  
 ACQUISITION exp 30  
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 in H1 den 200  
 dt 1.993 dnt PROCESSING  
 ap 23956 wtfle  
 ac 2000.0 not used  
 bc 16 proc  
 ss 14 in not used  
 twr 56 werr  
 pw 13.5 wexp  
 dt 1.000 wbs  
 tof 0 wnt  
 nt 64  
 ct 64  
 alock not used  
 gain n  
 flags  
 ll n  
 in n  
 dp y  
 DISPLAY  
 sp 52.0  
 wp 452.0  
 wpc 21.0  
 xc 0  
 wc 250  
 hzmm 15.08  
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 nm cdc ph

Compound 2

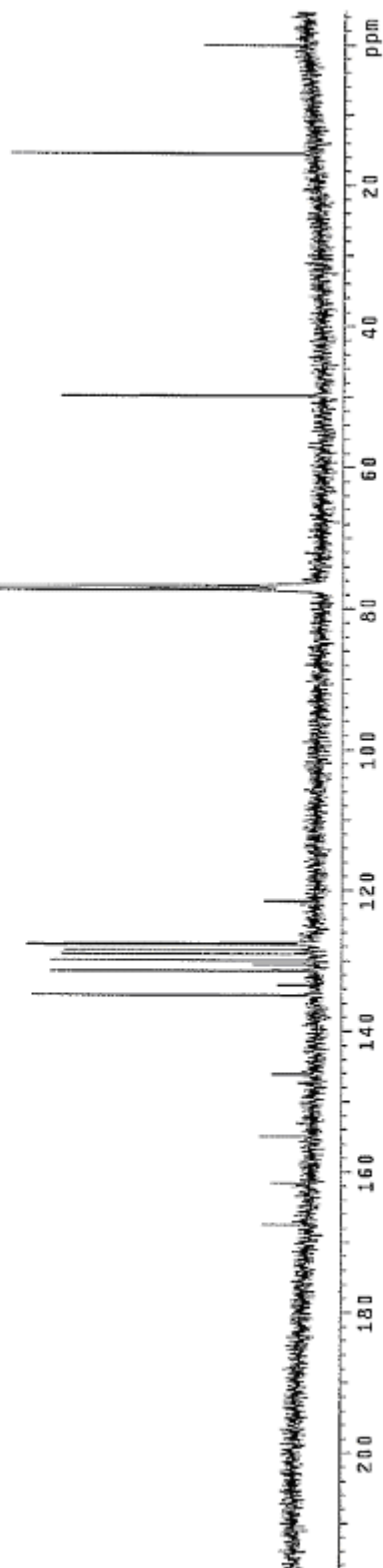
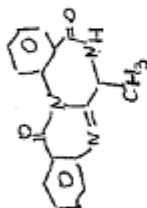


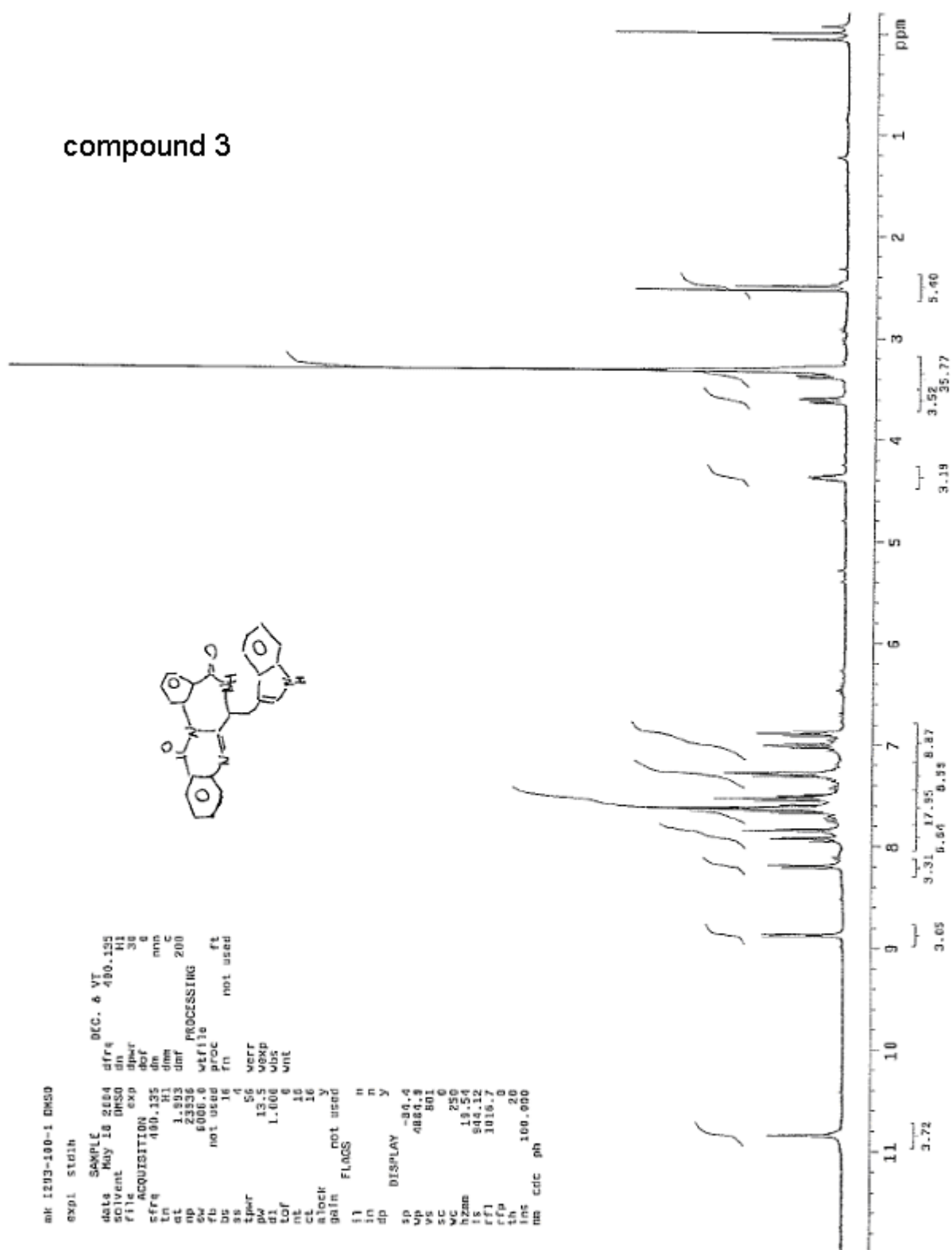
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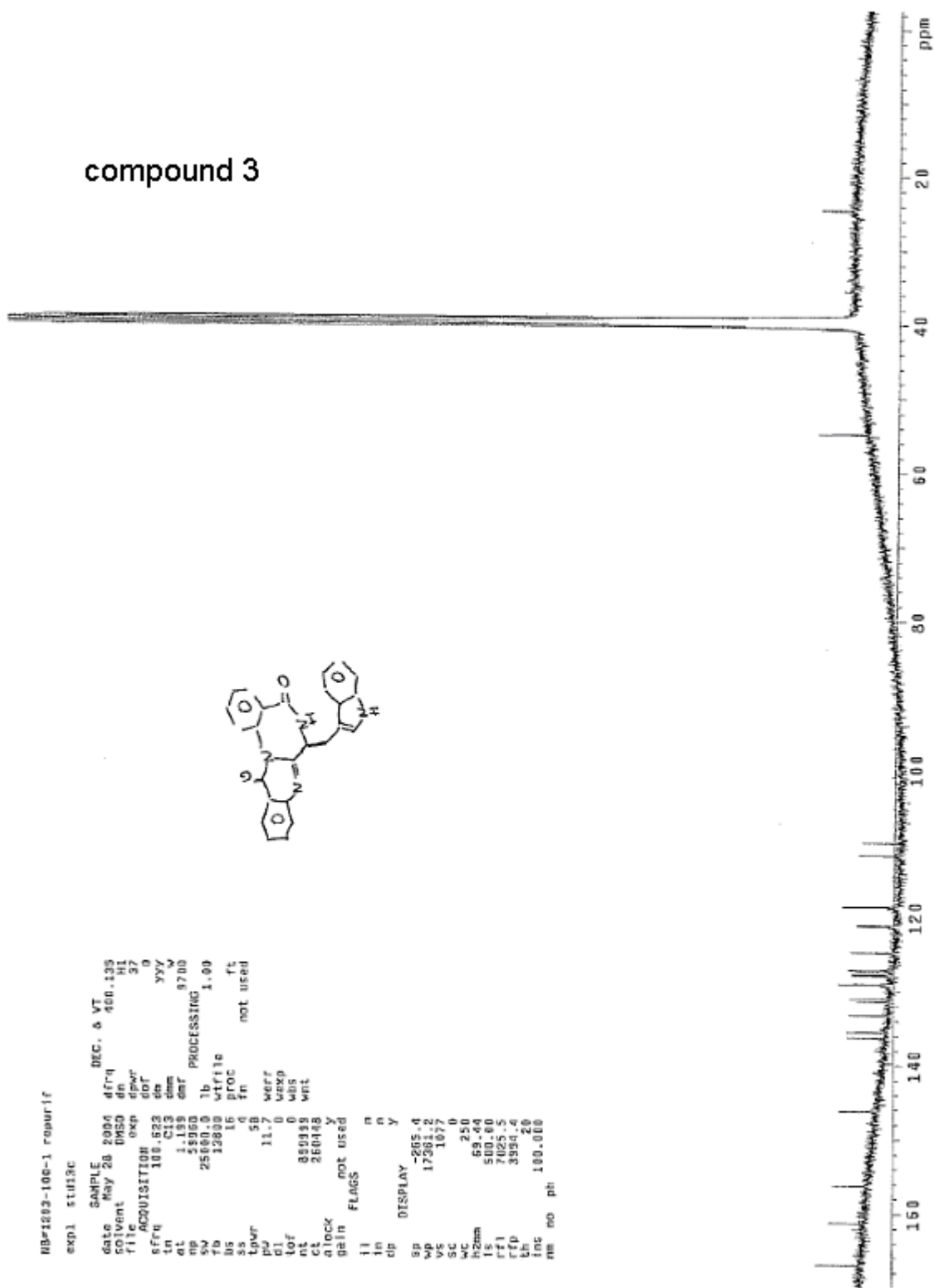
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 mirakas/mk-C13.f1~ ddr 0  
 d1 dm xvy w  
 ACQUISITION d dm xvy w  
 srfq 100.523 dfr 9700  
 in C13 dfr PROCESSING  
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 nu 50000 wfile  
 sw 25000.0 proc ft  
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 tpar 58 wexp  
 pw 11.7 wba  
 d1 0 wnt  
 lof 0  
 nt 789399  
 ci 13072  
 clock y  
 gain not used  
 FLAGS  
 fl n  
 in n  
 dp y  
 DISPLAY  
 sp -450.0  
 wp 22255.3  
 vs 17.0  
 vc 0  
 hzmm 89.18  
 ls 500.00  
 rfl 10755.7  
 rfp 7747.2  
 th 24  
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 nm no ph





compound 3





## SUPPORTING INFORMATION

NPL7R-Intern-p-1532-q2-2H1

exp1 std1h

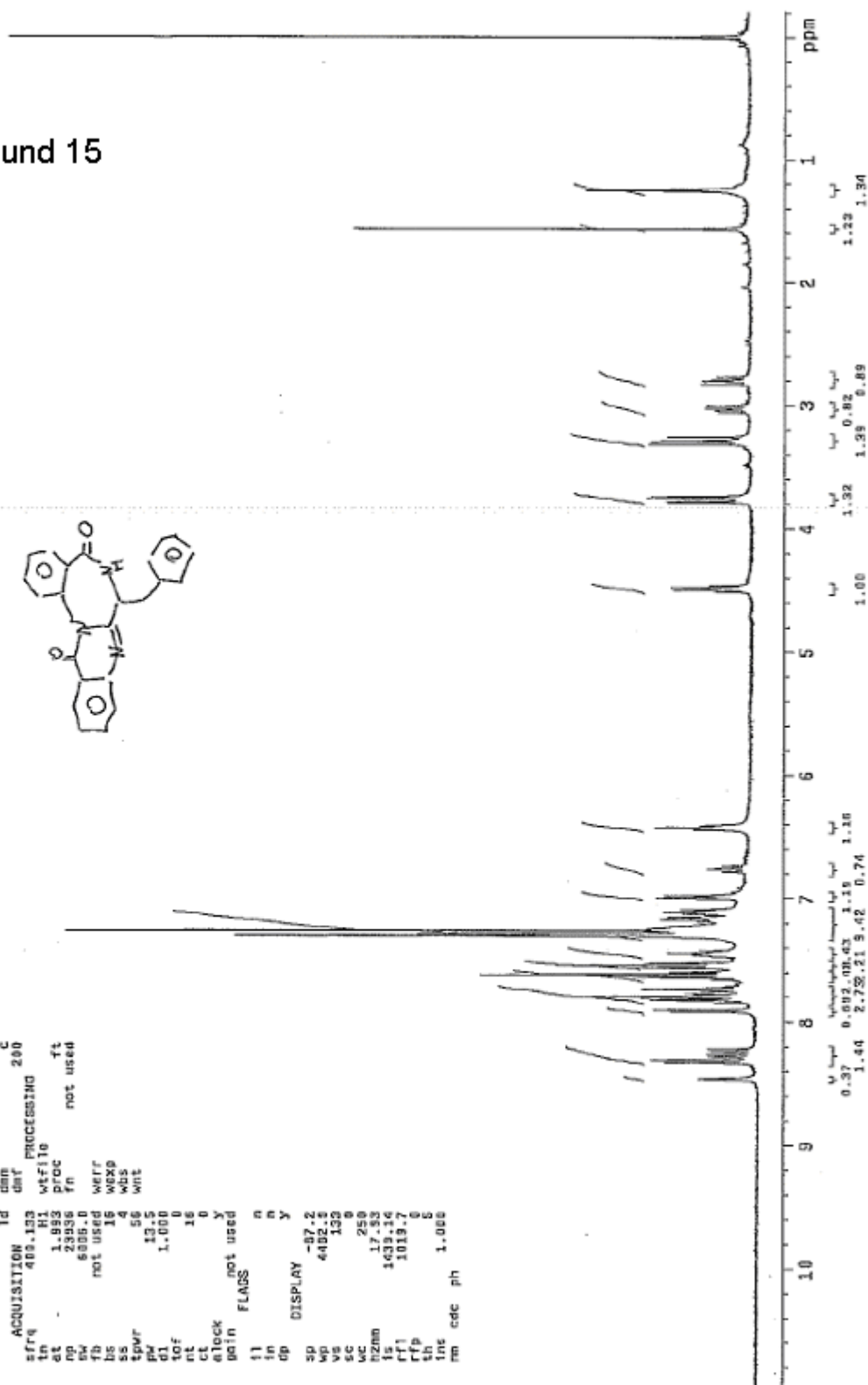
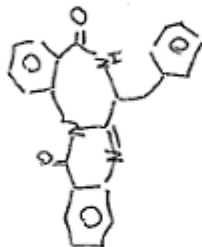
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 re-p-1532-q2-2H1.fw dm men  
 id dan c  
 daf 200

ACQUISITION  
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 tn H1 wfile  
 at 1.993 proc ft  
 pw 2398 fn not used  
 ts 6000.0 werr  
 bs not used werr  
 ss 16 w2p  
 spwr 56 wnt  
 gm 13.5  
 dl 1.000  
 tof 0  
 nt 16  
 ct 0  
 elock y  
 gain not used

FLAGS  
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 in n  
 dp y

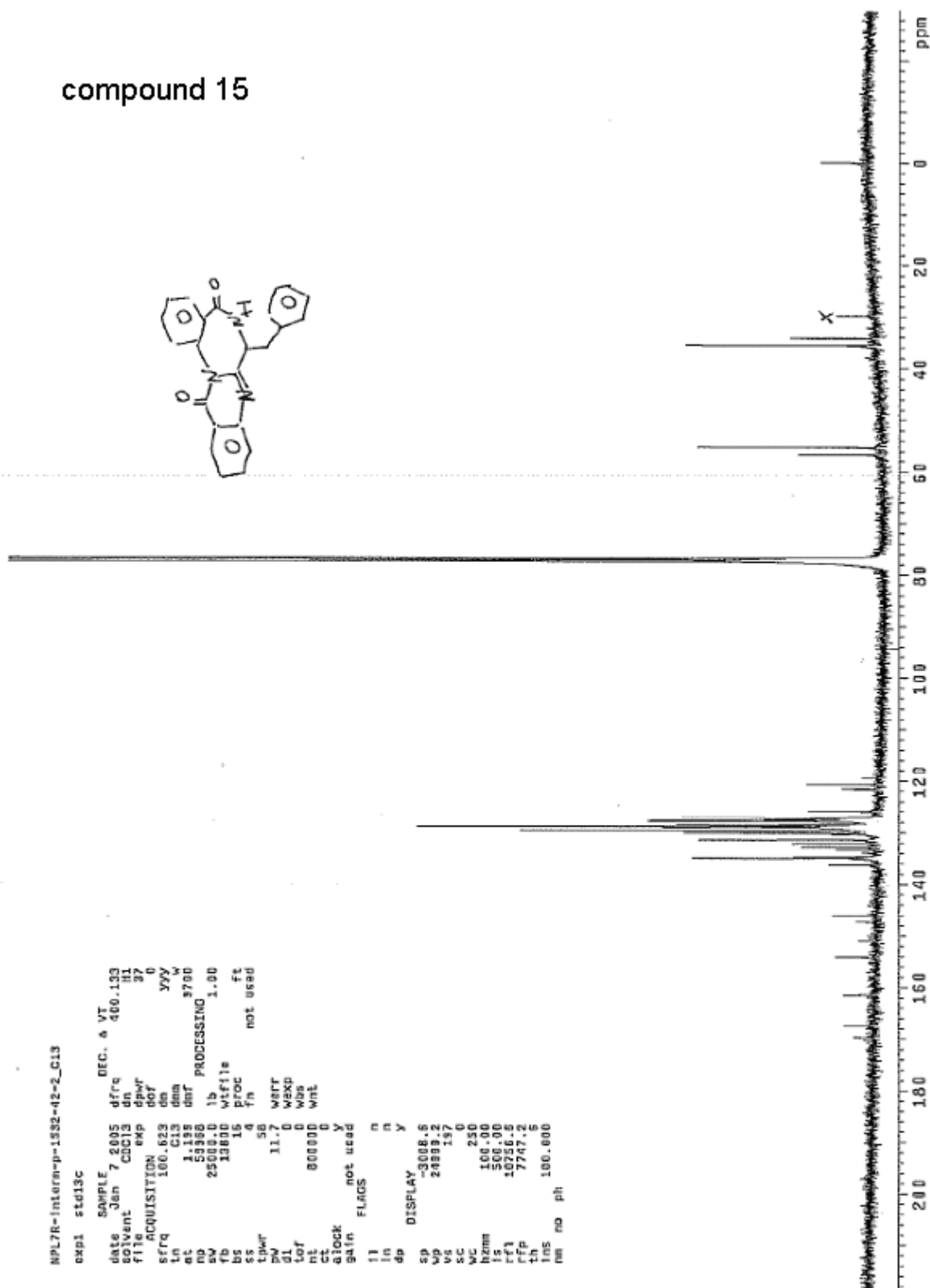
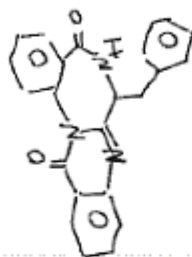
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 nm cdc ph

compound 15



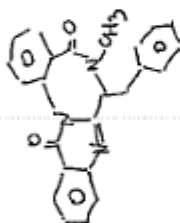
# SUPPORTING INFORMATION

compound 15

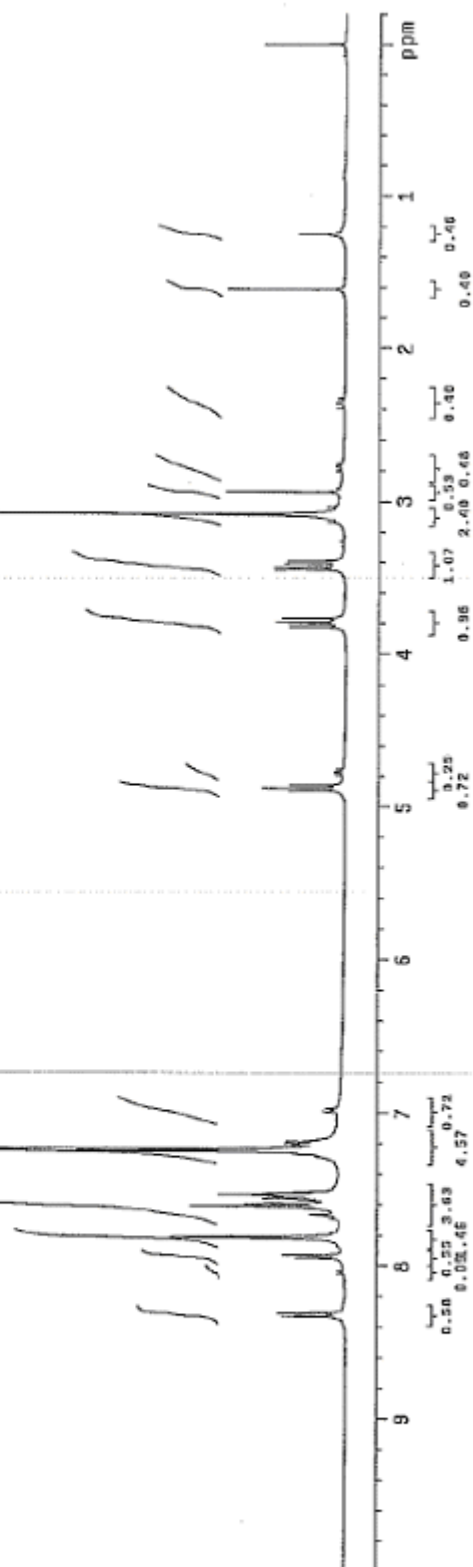


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 solvent CDCl<sub>3</sub> dn 400.133  
 file 37  
 ACQUISITION emp 37  
 sfrq 100.623 dm vvy  
 tn C13 dea w  
 at 1.185 dm 3700  
 nd 53360  
 sw 25000.0 lb PROCESSING 1.00  
 fb 13800 wfile  
 bs 16 proc ft  
 ss a fn not used  
 tpar 56  
 pw 11.7 werr  
 di 0 wexp  
 of 0 wse  
 ct 80000 wnt  
 clock 0  
 gain not used  
 flags  
 ll n  
 in n  
 dp y  
 DISPLAY  
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 vs 157  
 sc 0  
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 fs 500.00  
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 rms 100.000  
 nm no ph

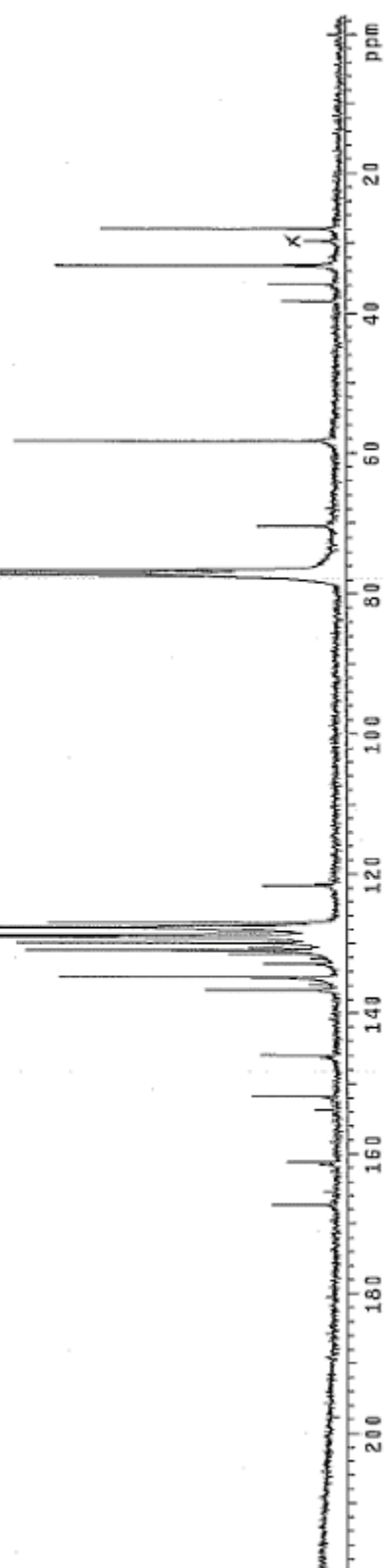
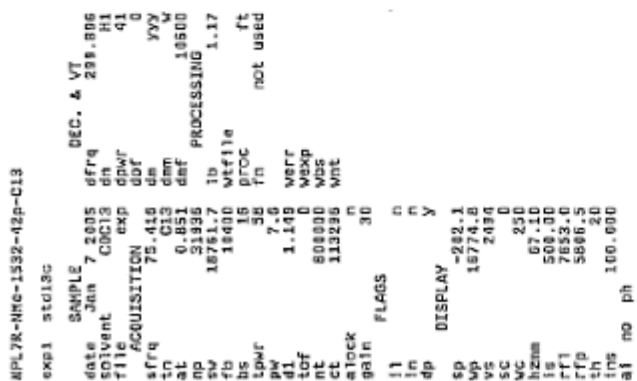
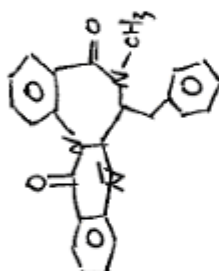
compound 4



SAMPLE		DEC. & VT
date can e sans	dfr	460.193
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solvent	can	33
file export/hot/~	dwr	33
jennan/mpt-78-NCH-	usf	nrm
Sp-1532-42-fid	dm	c
ACQUISITION	dm	200
afrq	dfr	PROCESSING
460.193	H1	
1.503	wflla	fl
28396	proc	not used
6085.0	fn	
not used		
16	werr	
56	wexp	
136	wes	
1.000	wnt	
15	e	
15	et	
0	e	
n	n	
not used	n	
FLAGS		
n	n	
n	n	
y	y	
DISPLAY		
-82.4		
4077.6		
167		
0		
230		
153		
1739.40		
1019.7		
0		
f-p		
h		
s		
ins		
cdc		
ph		
1.000		



compound 4



p.1 OMe.

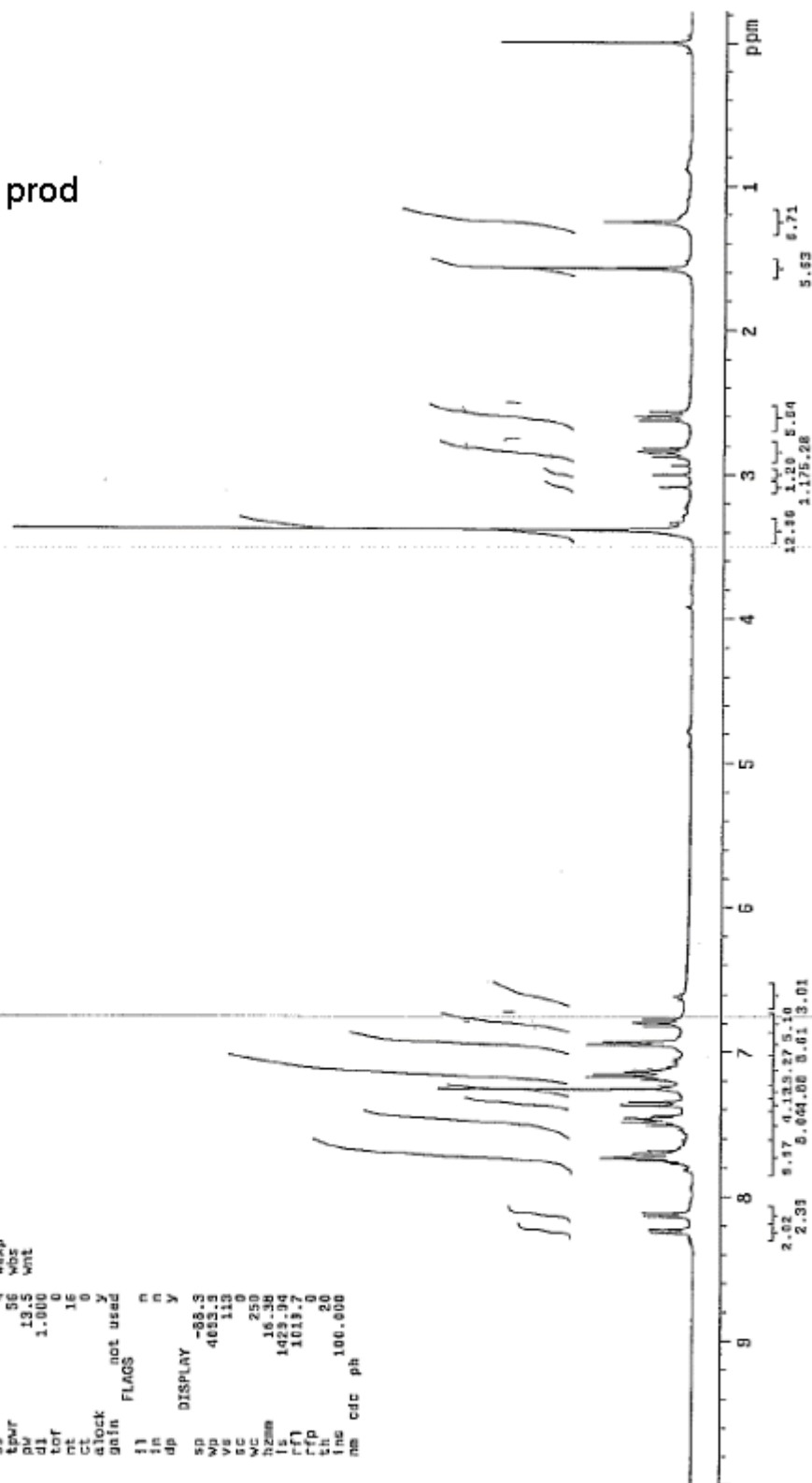
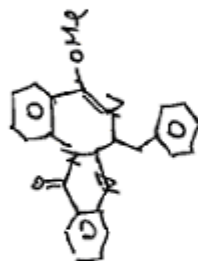
NPL7R-NCN3p-2-1532-42

```

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solvent CDCl3 dn
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jacking/NPL7R-NCN3p-2-1532-42.fid an 0
sp-2-1532-42.fid an min c
ACQUISITION
dfrq 400.133 chr PROCESSING 200
ft 1.000
at 1.801 wdf16
ap 21836 proc ft
sw 6000.0 fn not used
fb not used
ls 16 warr
ss 4 wexp
tpwr 56 wbs
pv 13.5 wnt
d1 1.000
tof 0
nt 16
ct 0
alock y
gain not used
FLAGS
ll n
in n
dp y
sp -85.3
wp 4651.3
vc 110
mc 250
hzmm 16.38
ls 1423.94
rfi 1013.7
rfp 0
th 20
fns 100.000
nm cdc ph

```

O-methylation prod

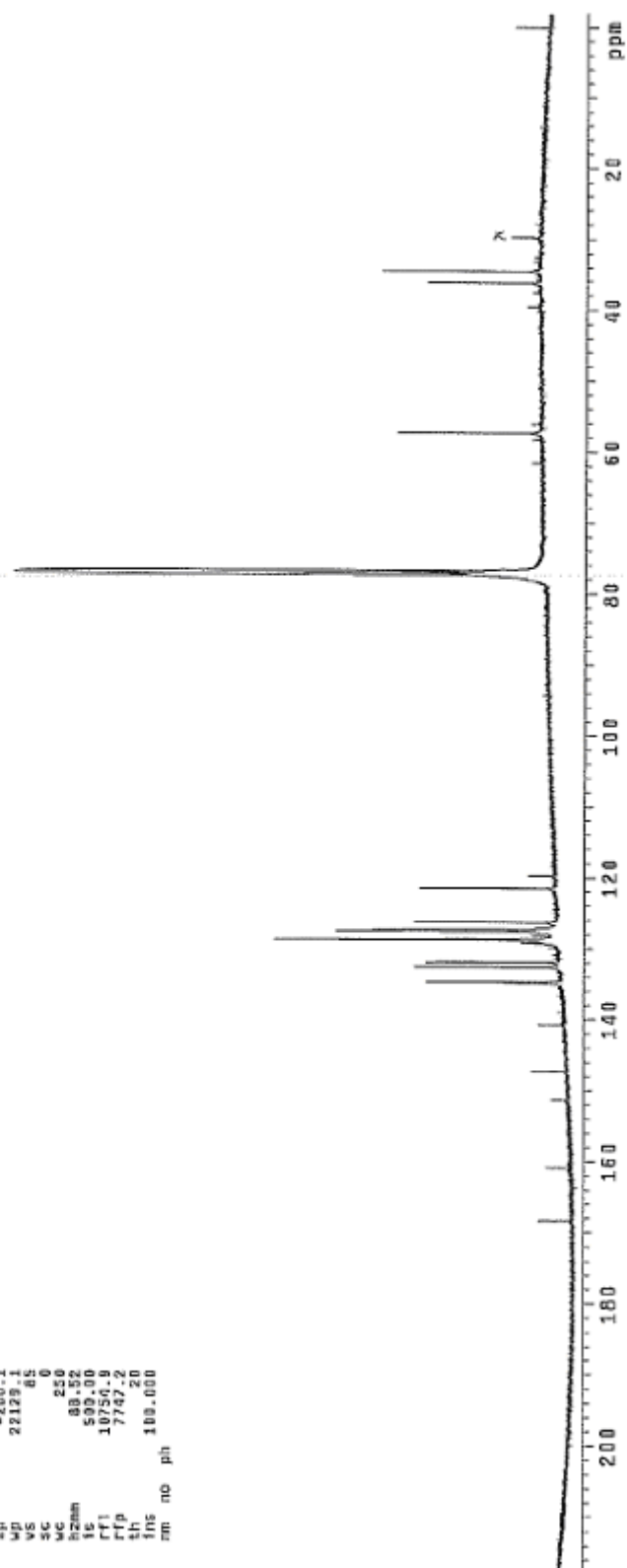
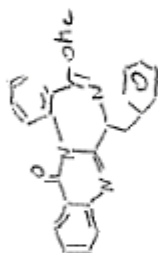


JNC-1532-42-1\_OMe-TLC-C13

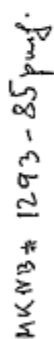
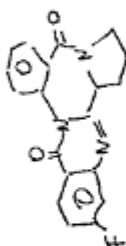
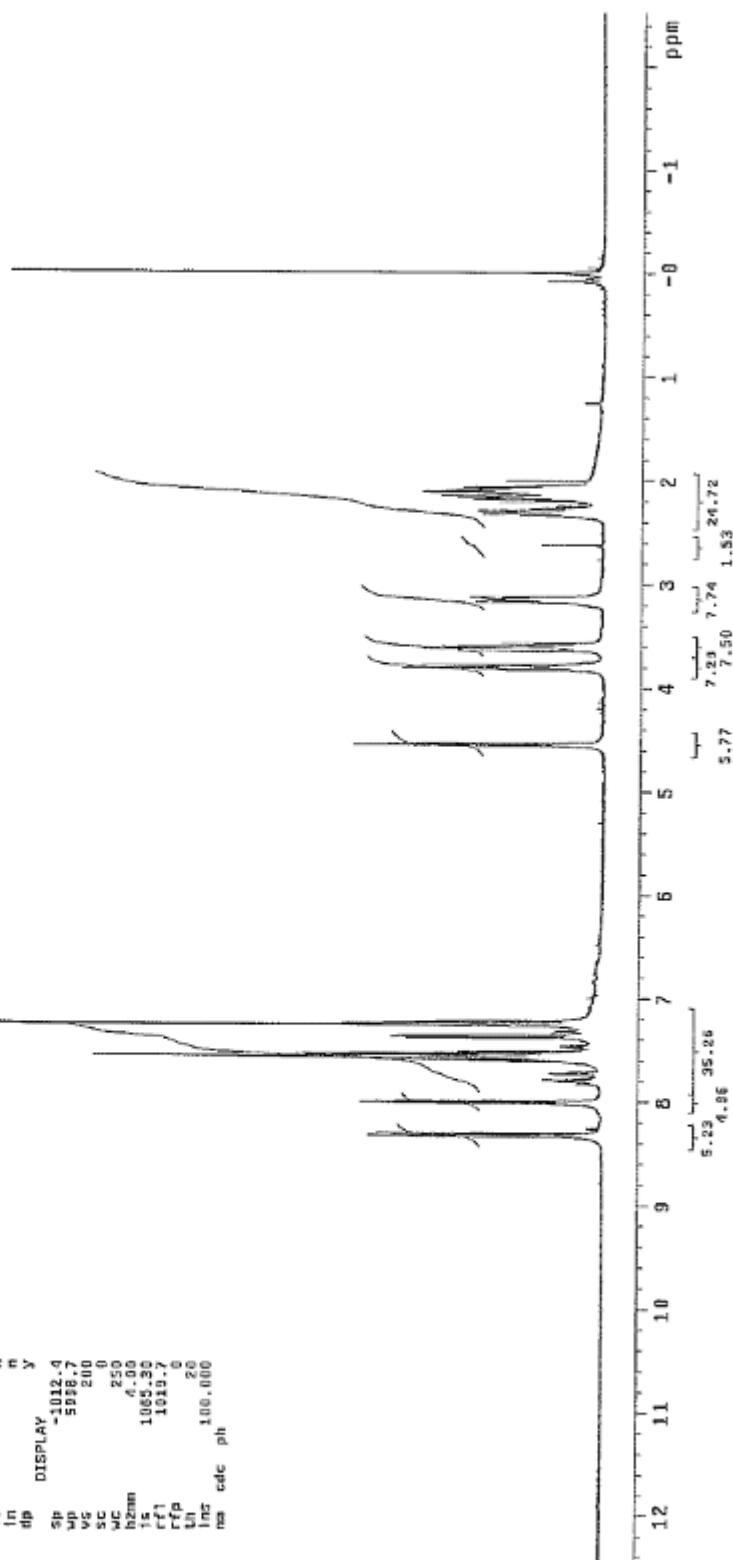
ex01 std13C

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 tn C13 dm yyy  
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 sp 53868 1b PROCESSING 1.03  
 cp 250000 1b file  
 fb 13000 1b proc  
 bs 15 1b not used  
 ss 58  
 tpr 11.7 werr  
 pw 0 wexp  
 d1 0 wds  
 tcf 50000 wnt  
 nt 250000  
 ct 250000  
 atock y  
 gain not used  
 FLAGS  
 fl n  
 in n  
 dp y  
 DISPLAY  
 sr -200.1  
 wp 22129.1  
 vs 85  
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 fl 19750.0  
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 th 103.000  
 ins no ph

O-methylation prod



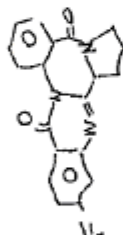
compound 16

[illegible]

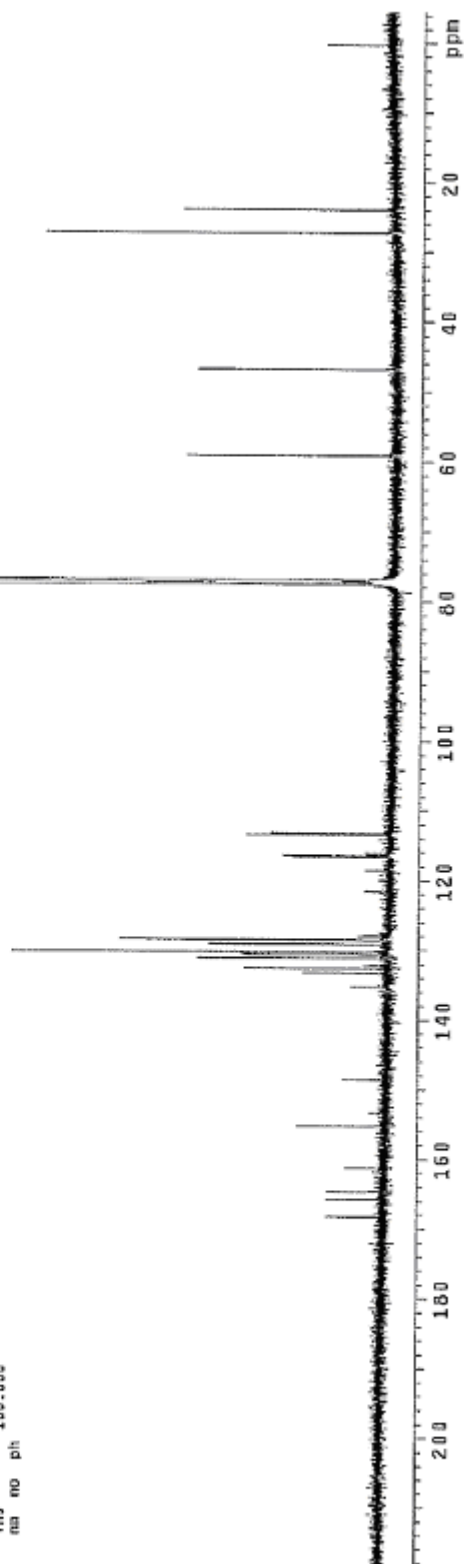
KV NB # 1293-85B

13C OBSERVE  
DOS 85B *ref.*  
expt 1293-85B

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solvent	CDCl <sub>3</sub>	dn	H1
flia	exp	dpcr	37
ACQUISITION		exp	0
sfreq	100.623	de	VVY
in	C13	dam	W
at	1.199	def	8700
np	58968		
sw	25000.0	lb	PROCESSING
fo	13800	wtfile	not used
ps	16	proc	
tsmr	58	fn	not used
pw	11.7	mcrr	
d1	0	mcsp	
tof	78959	mba	
nt	44192	mnt	
cl	not used		
clock	Y		
gain	not used		
FLAGS			
l1	n		
l2	n		
l3	n		
dp	Y		
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vs	112		
sc	0		
vc	250		
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rfp	0		
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ins	100.000		
na	no	ph	



compound 16





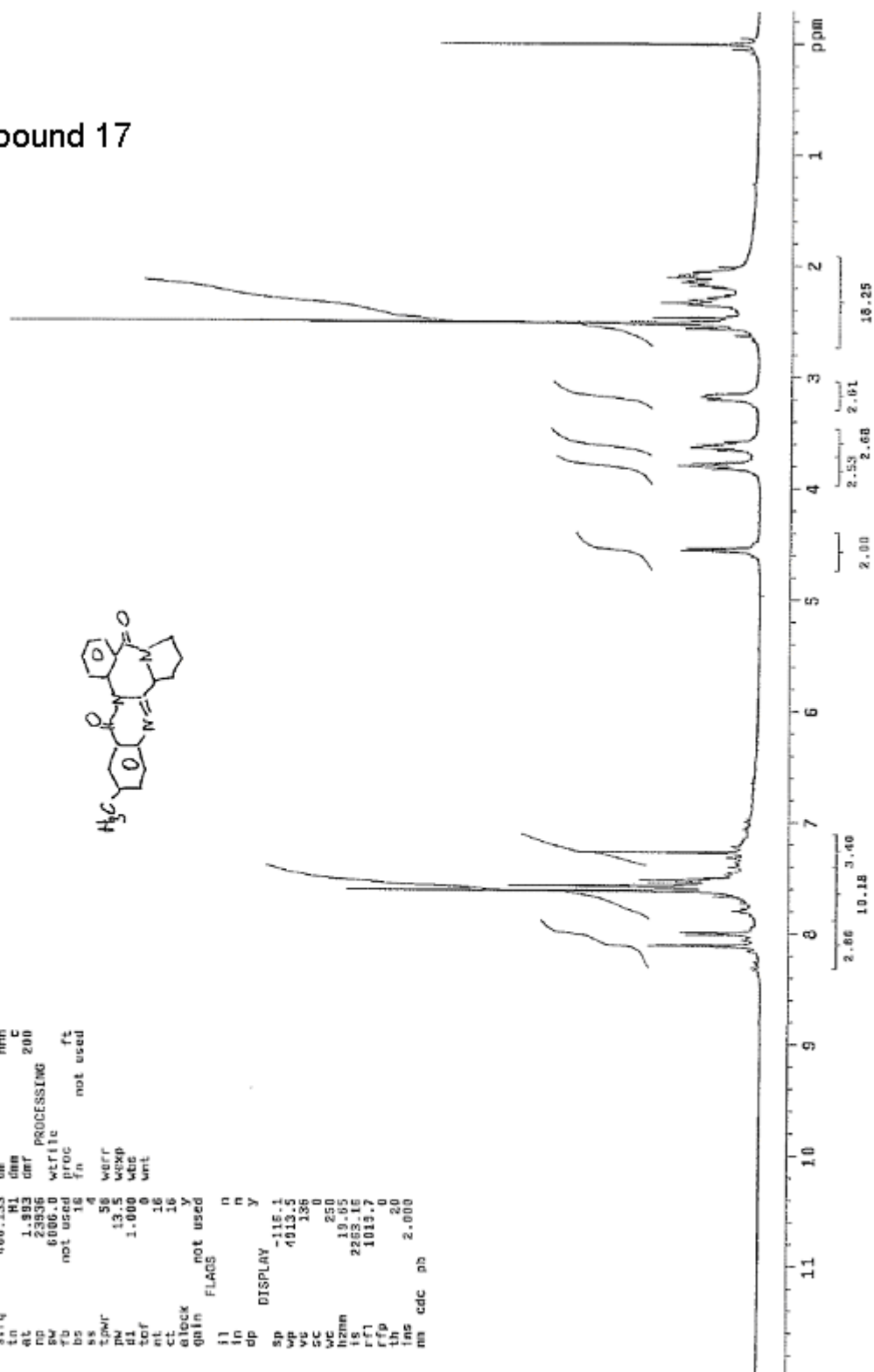
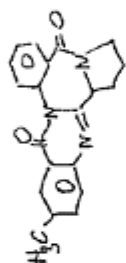
WV NB # 1293 - 86B

DOS 2 86B-2

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 pw 13.5 wexp  
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 ct 16  
 atlock not used  
 gain y  
 flags  
 il n  
 in n  
 op y  
 DISPLAY  
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 vc 136  
 sc 0  
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 ma cdc pb

compound 17



KX NB# 1293 -86 B

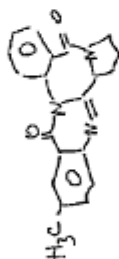
13C OBSERVE

D52-82B2

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 nt 50000  
 sw 25000.0  
 fb 13000  
 bs 16  
 ss 4  
 tpwr 58  
 pw 11.7  
 di 0  
 tof 1e+07  
 ct 42304  
 alock y  
 gain not used  
 flags n  
 il n  
 in n  
 dp y  
 sp -201.1  
 wv 22101.1  
 vs 138  
 sc 0  
 wc 250  
 hznm 88.44  
 ls 500.03  
 rf1 3008.0  
 rfp 0  
 th 20  
 ins 100.000  
 nm no ph

DEC. 6 VT  
 dfrq 400.133  
 da H1  
 dpr 37  
 dpr 0  
 dpr yyy  
 dpr W  
 dpr 9700  
 dpr 1.00  
 dpr not used



compound 17

