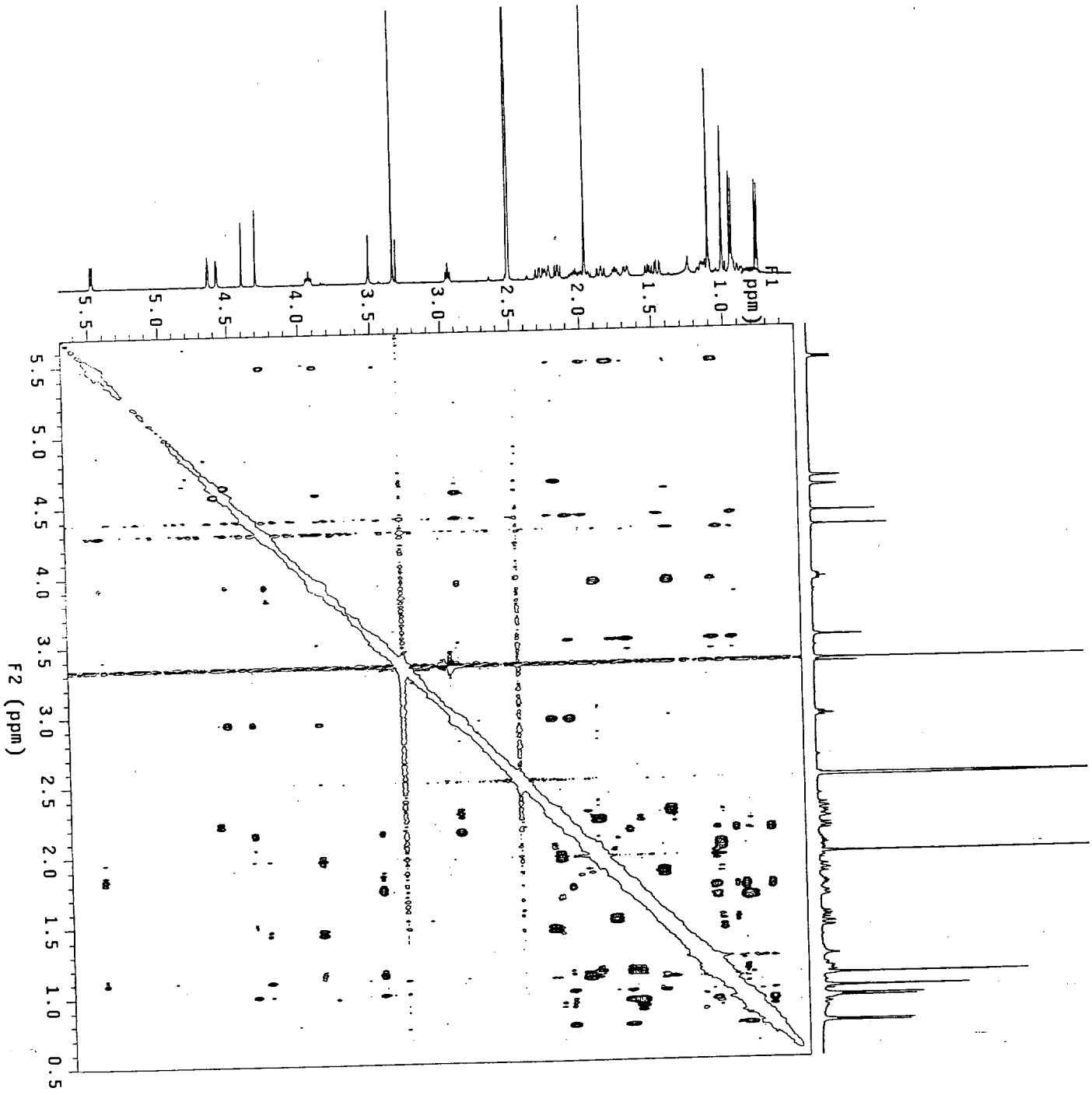
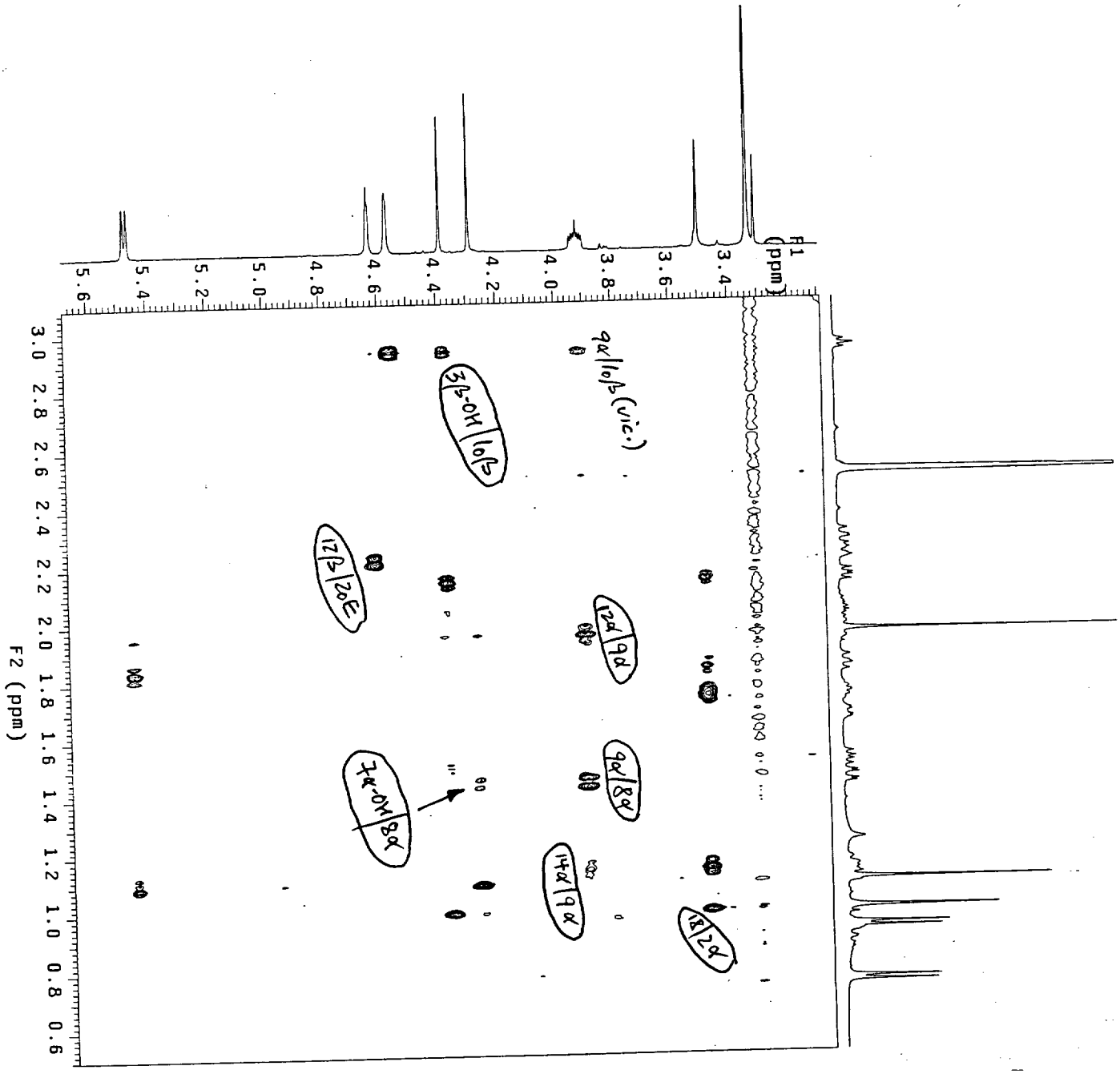
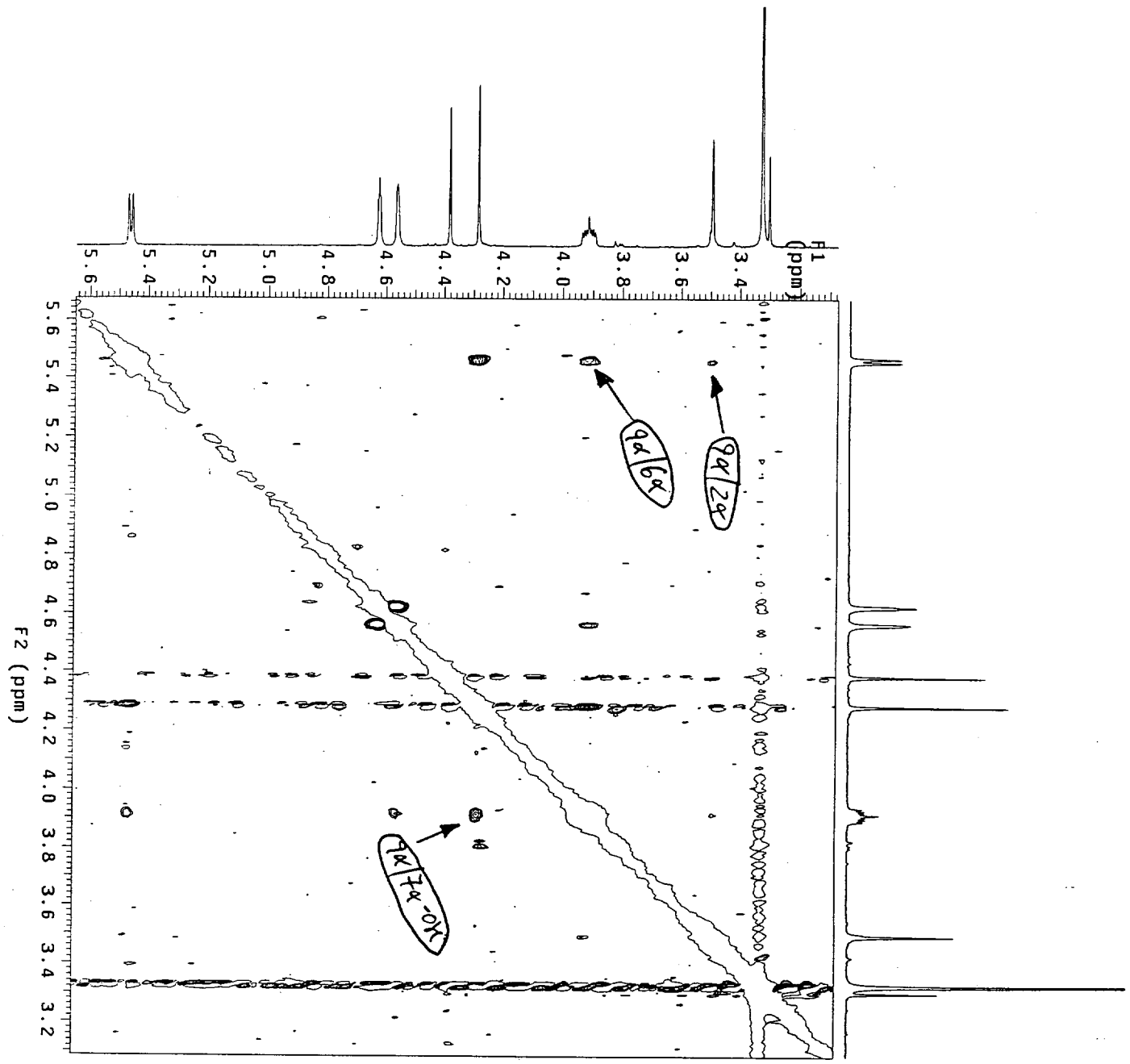
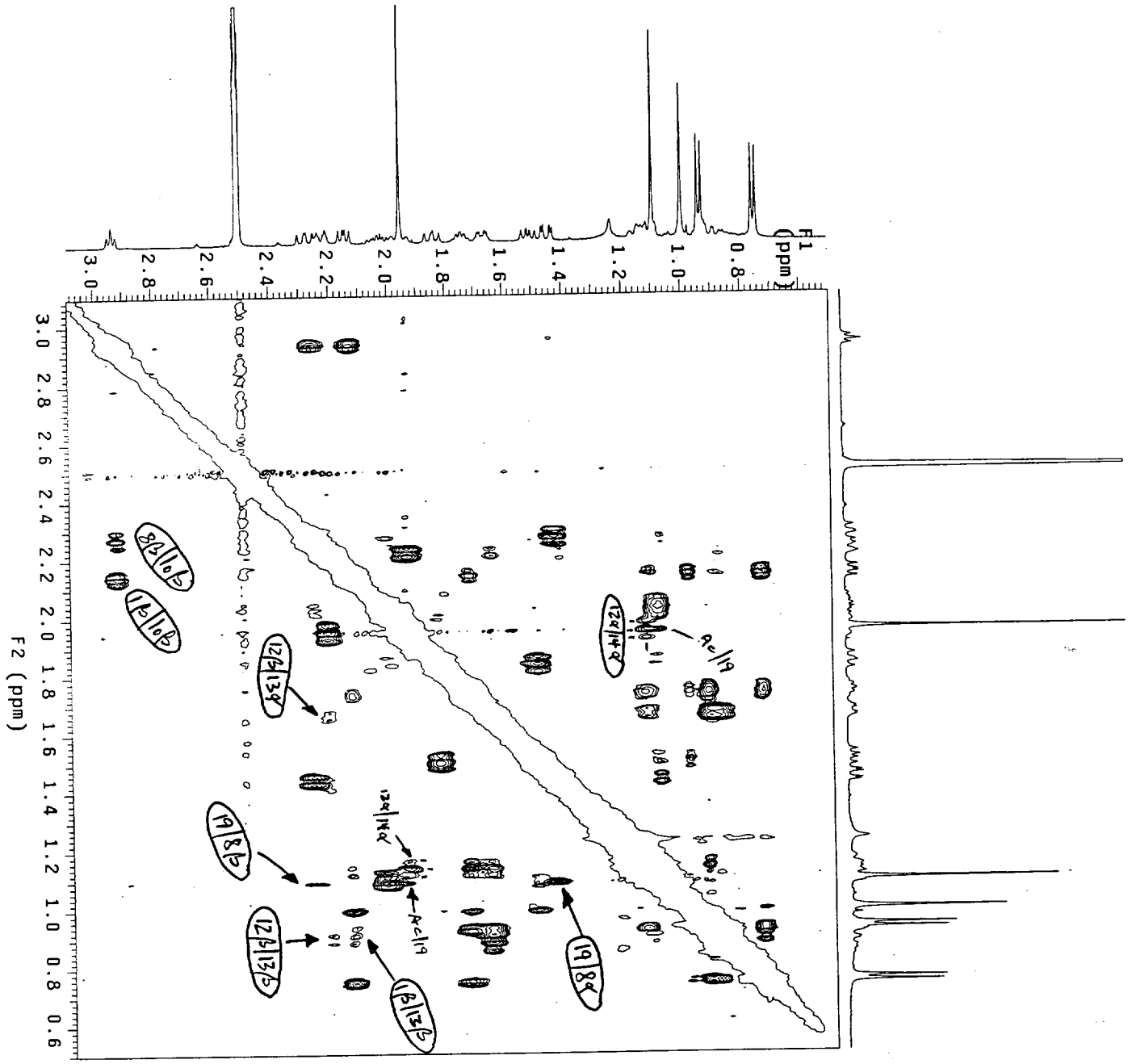


NOESY
500 MHz
DMSO-d6


F1 (ppm)
F2 (ppm)
9α/10β (vic.)
3β-OH/10β
12α/9α
9α/8α
14α/9α
18/2α
17β/20E
7α-OH/8α




F1 (ppm)
F2 (ppm)
3.0 2.8 2.6 2.4 2.2 2.0 1.8 1.6 1.4 1.2 1.0 0.8 0.6
8β/10β
1β/10β
12α/13β
19/8β
12β/13β
12α/14α
9α/19
1β/13β
19/8α

NOESY_DMSO
18A_acetate
Solvent: DMSO
Operator: DF
gain=40/ct=64

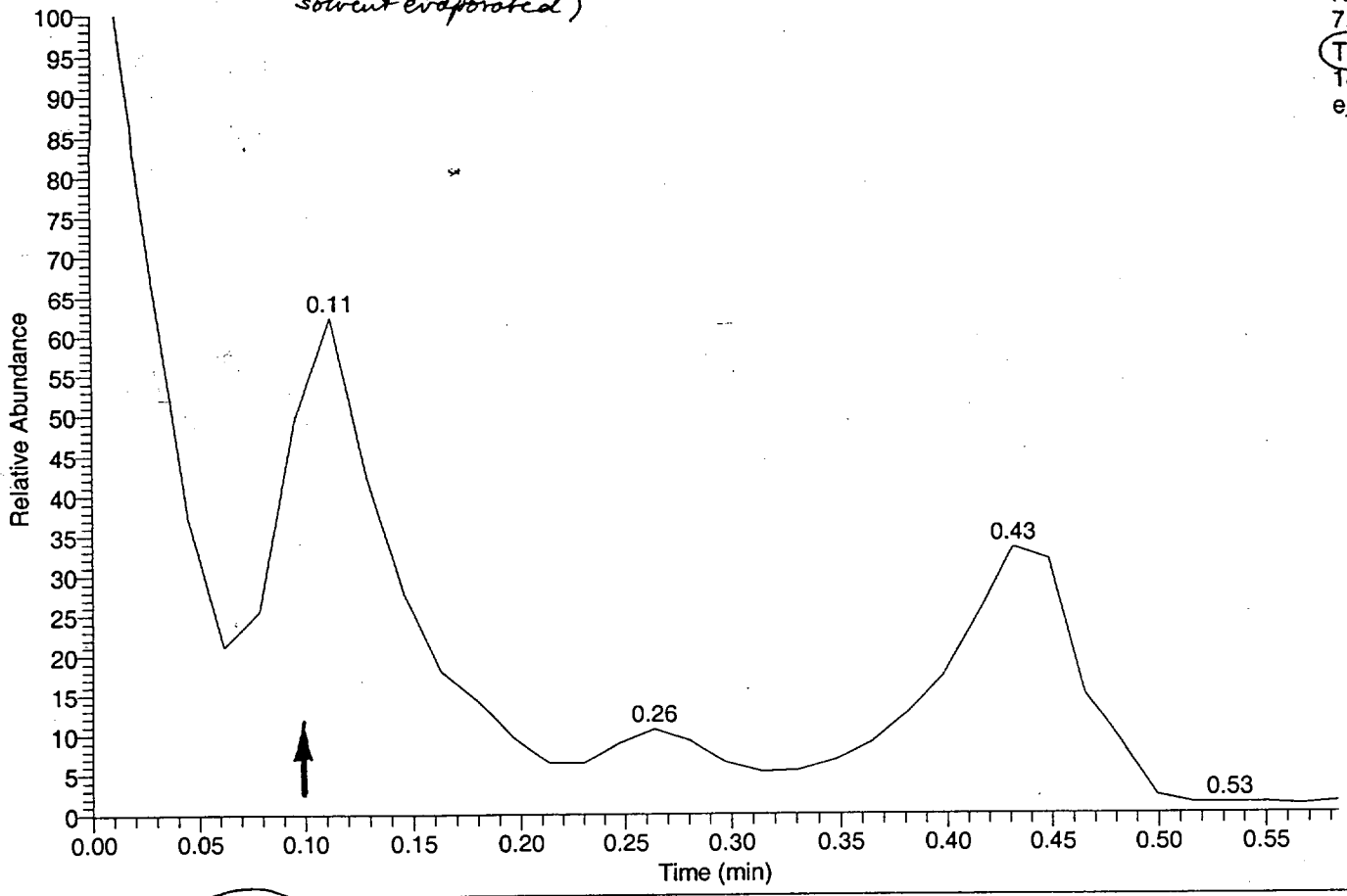
exp8 NOESY

SAMPLE		DEC. & VT	
date	Jan 14 2000	dn	H1
solvent	DMSO	dof	0
file	exp	dm	n
ACQUISITION		dmm	c
sfrq	499.881	dmf	200
tn	H1	temp	25.0
at	0.293	PROCESSING	
np	2048	gf	0.135
sw	3499.3	gfs	not used
fb	2000	fn	2048
ss	8	math	i
pw	9.2		
pw	9.2	werr	
tpwr	54	wexp	
d1	1.000	wbs	
tof	-1007.1	wnt	
nt	64	2D PROCESSING	
ct	64	gf1	0.034
alock	n	gfs1	not used
gain	40	fn1	1024
FLAGS		DISPLAY	
il	n	sp	249.5
in	n	wp	2596.2
dp	y	vs	3429
hs	n	sc	62
2D ACQUISITION		wc	130
sw1	3499.3	hzmm	19.97
ni	256	is	428.57
2D DISPLAY		rf1	1499.6
sp1	250.0	rfp	1249.7
wp1	2595.3	th	2
sc2	0	ins	0.857
wc2	130	ai cdc ph	
rf11	1499.6		
rfp1	1249.7		

18a_acetate_ci
Probe CI,.ecurr=200
RT: 0.00 - 0.58

(CDCl3 soln. applied to probe; solvent evaporated)

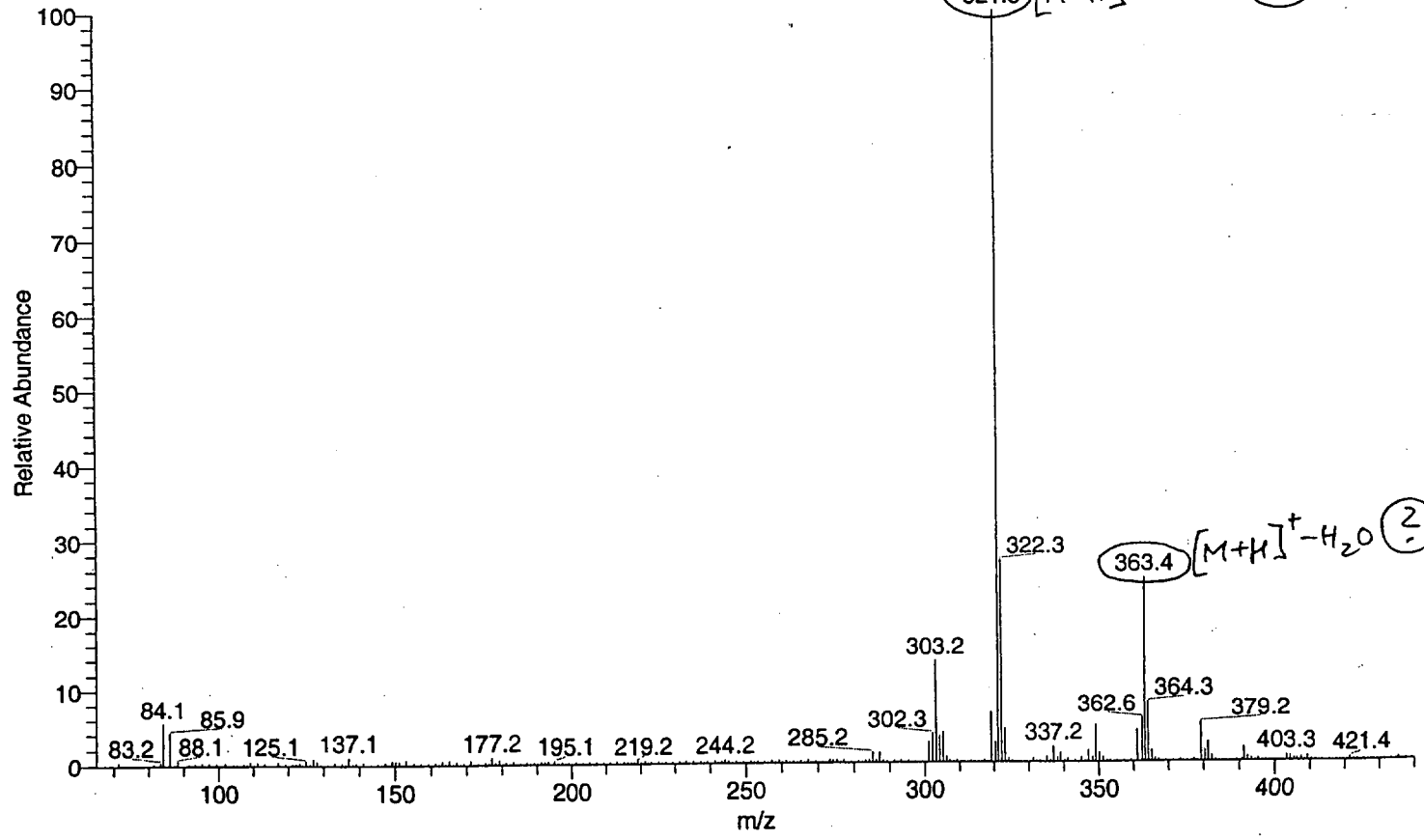
NL:
7.86E7
TIC MS
18a_acetat
e_ci



18a_acetate_ci#1 RT: 0.01 AV: 1 NL: 2.53E7
T: + c CI ms [64.99-899.98]

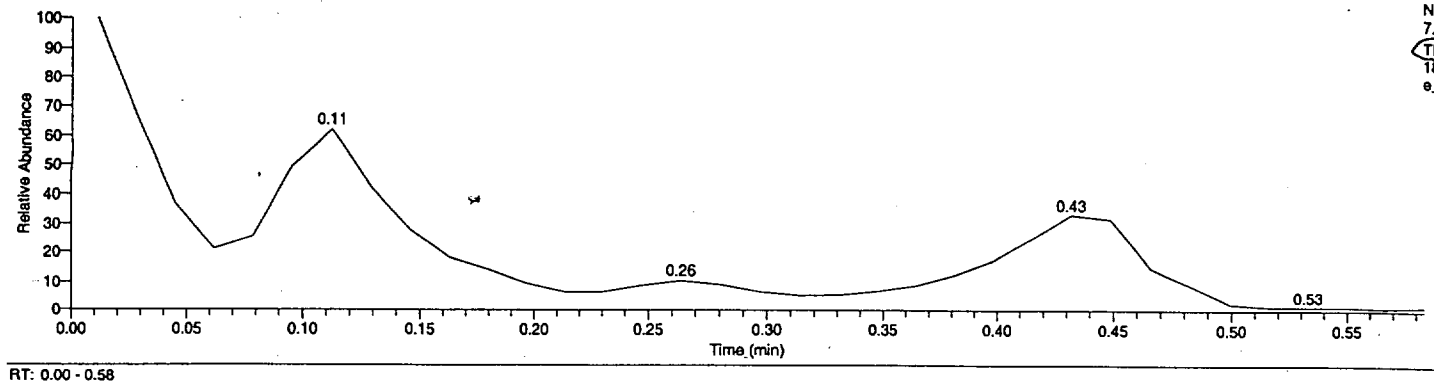
321.3 $[M+H]^+ - HOAc$ (2+)

363.4 $[M+H]^+ - H_2O$ (2+)

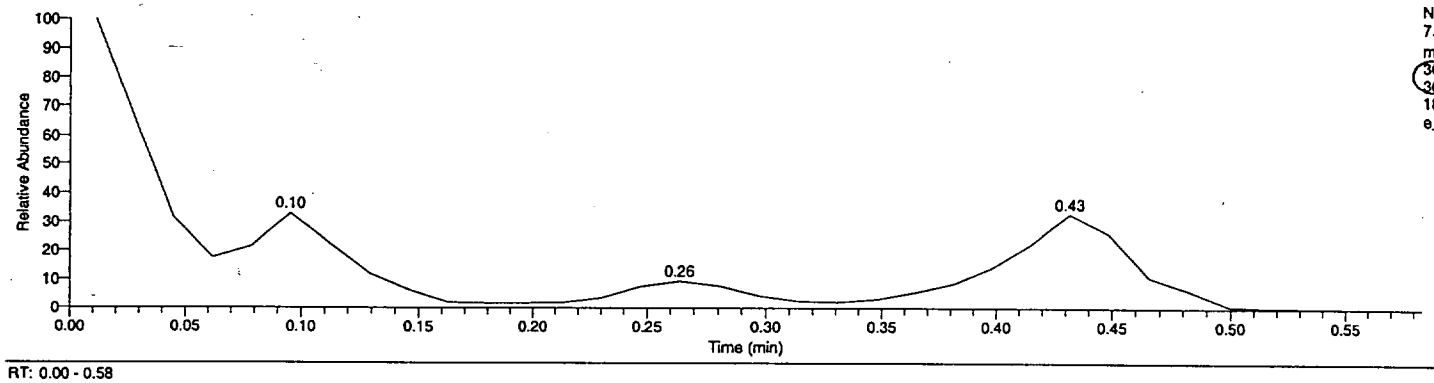


Probe CI,.ecurr=200

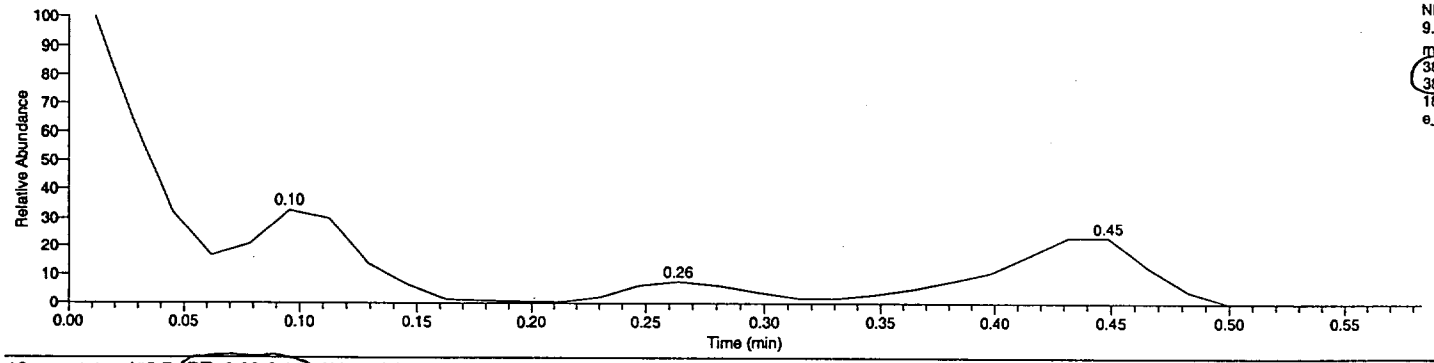
RT: 0.00 - 0.58



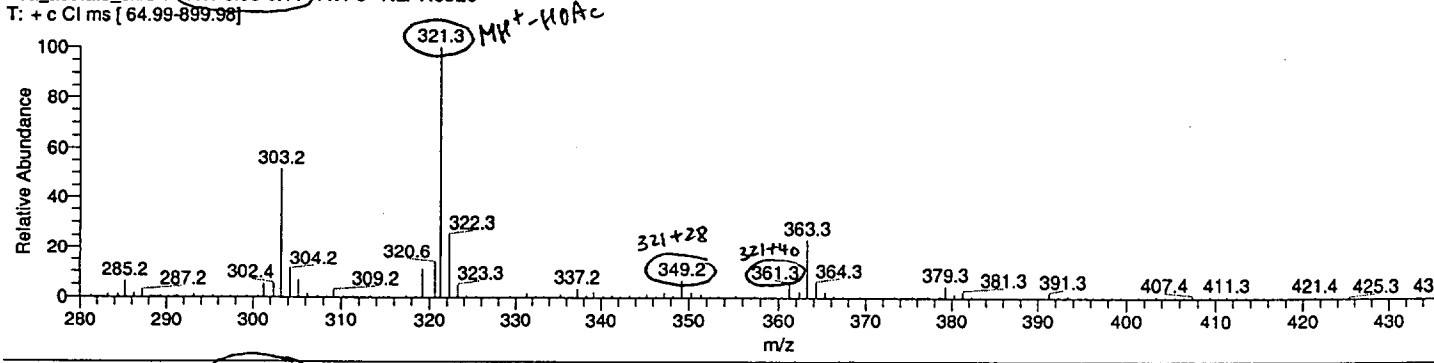
RT: 0.00 - 0.58



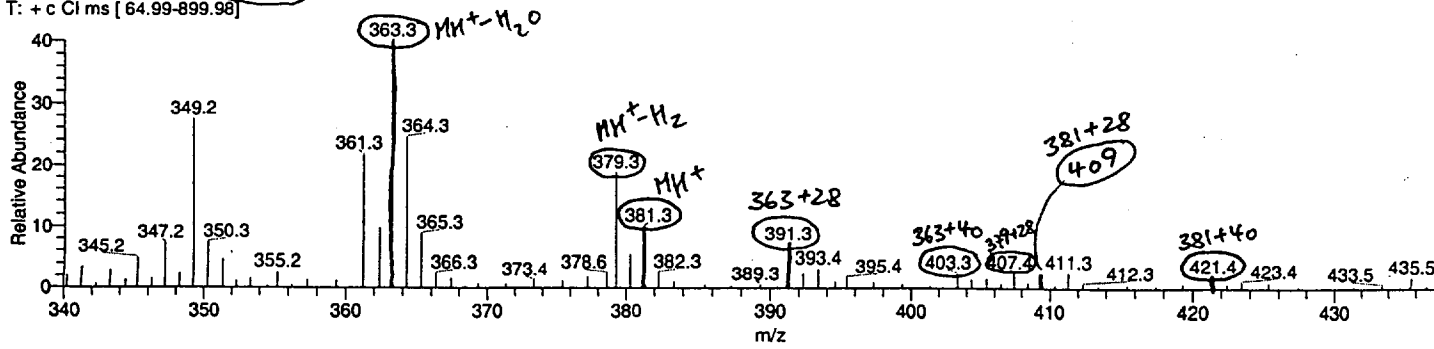
RT: 0.00 - 0.58



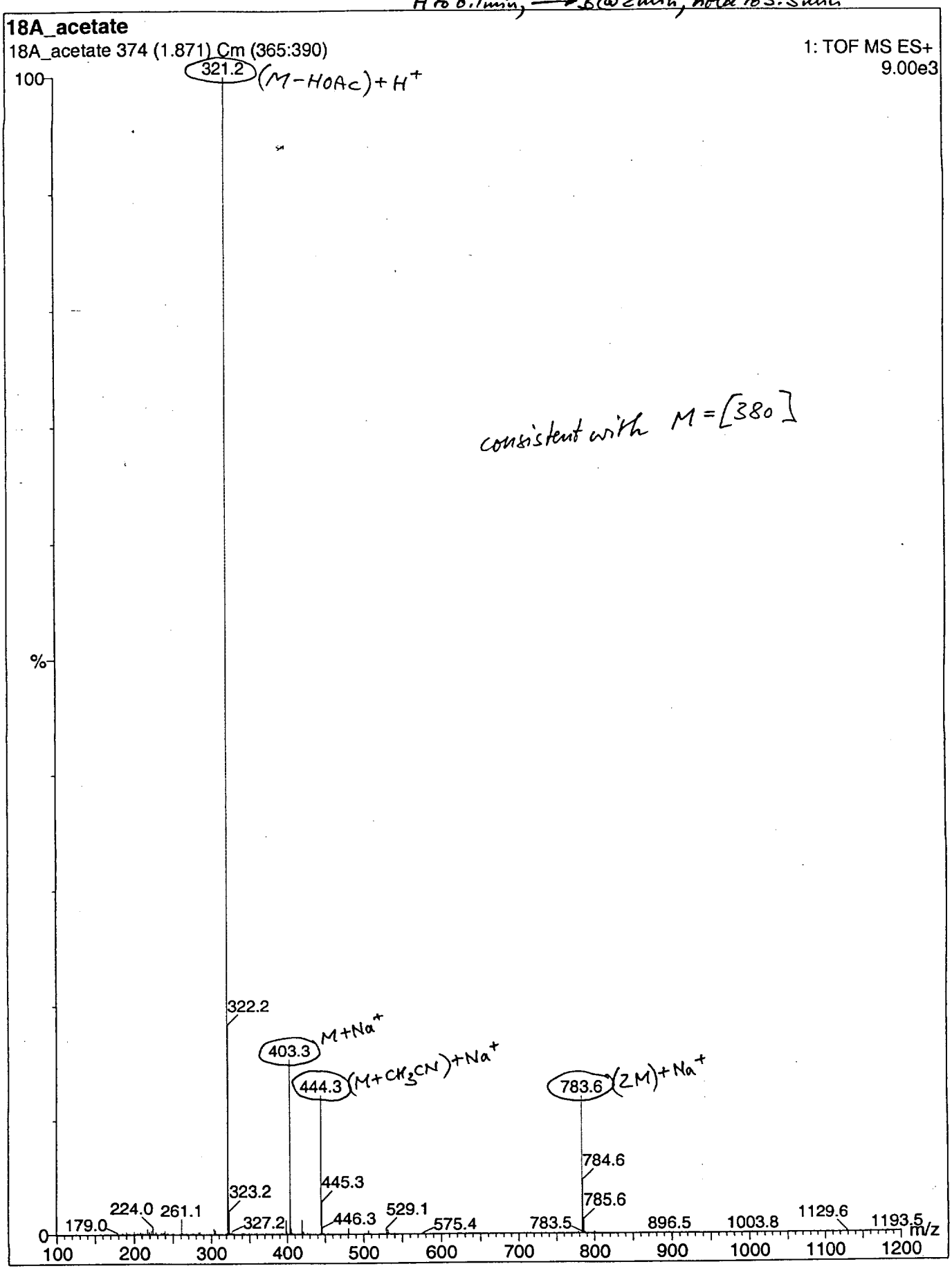
18a_acetate_ci#5-7 RT: 0.08-0.11 AV: 3 NL: 7.65E6
T: + c CI ms [64.99-899.98]



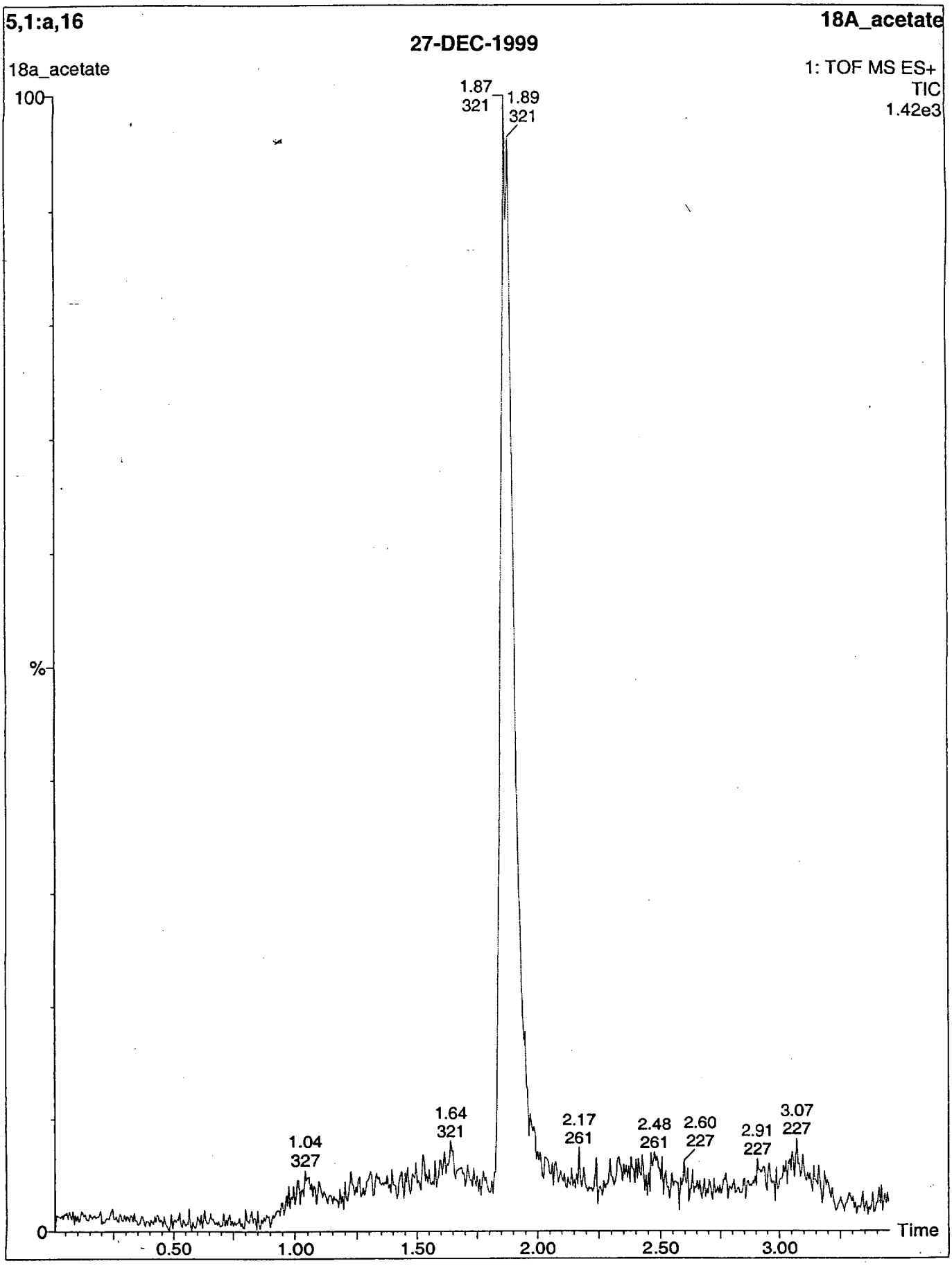
18a_acetate_ci#5-7 RT: 0.08-0.11 AV: 3 NL: 1.78E6
T: + c CI ms [64.99-899.98]



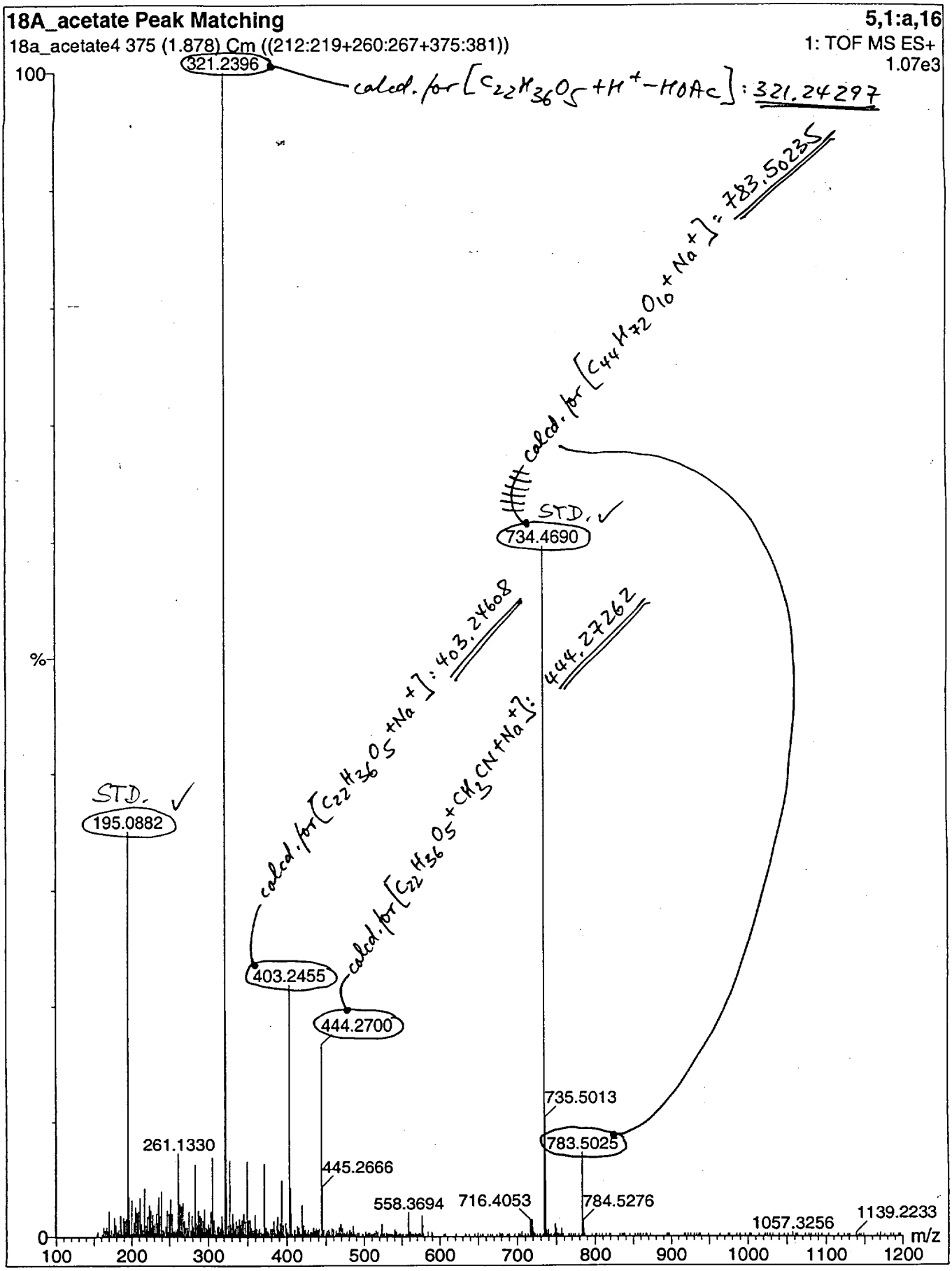
LC-MS (ESI/TOF) [ACN/H2O/HCO2H linear grad.] B = 5%H2O/75%ACN/0.1%HCO2H
A to 0.1min, → B@2min, hold to 3.5min

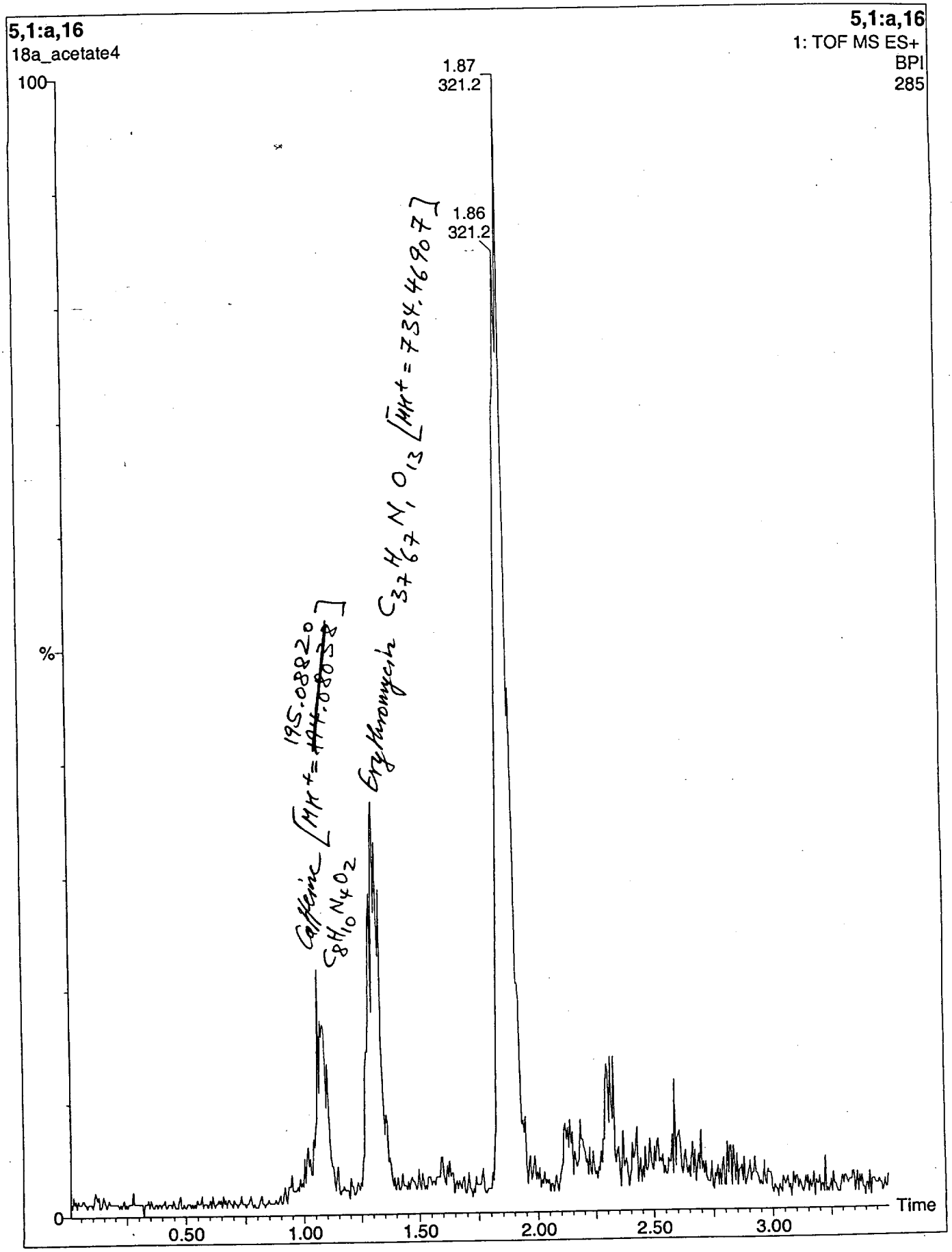


consistent with M = [380]

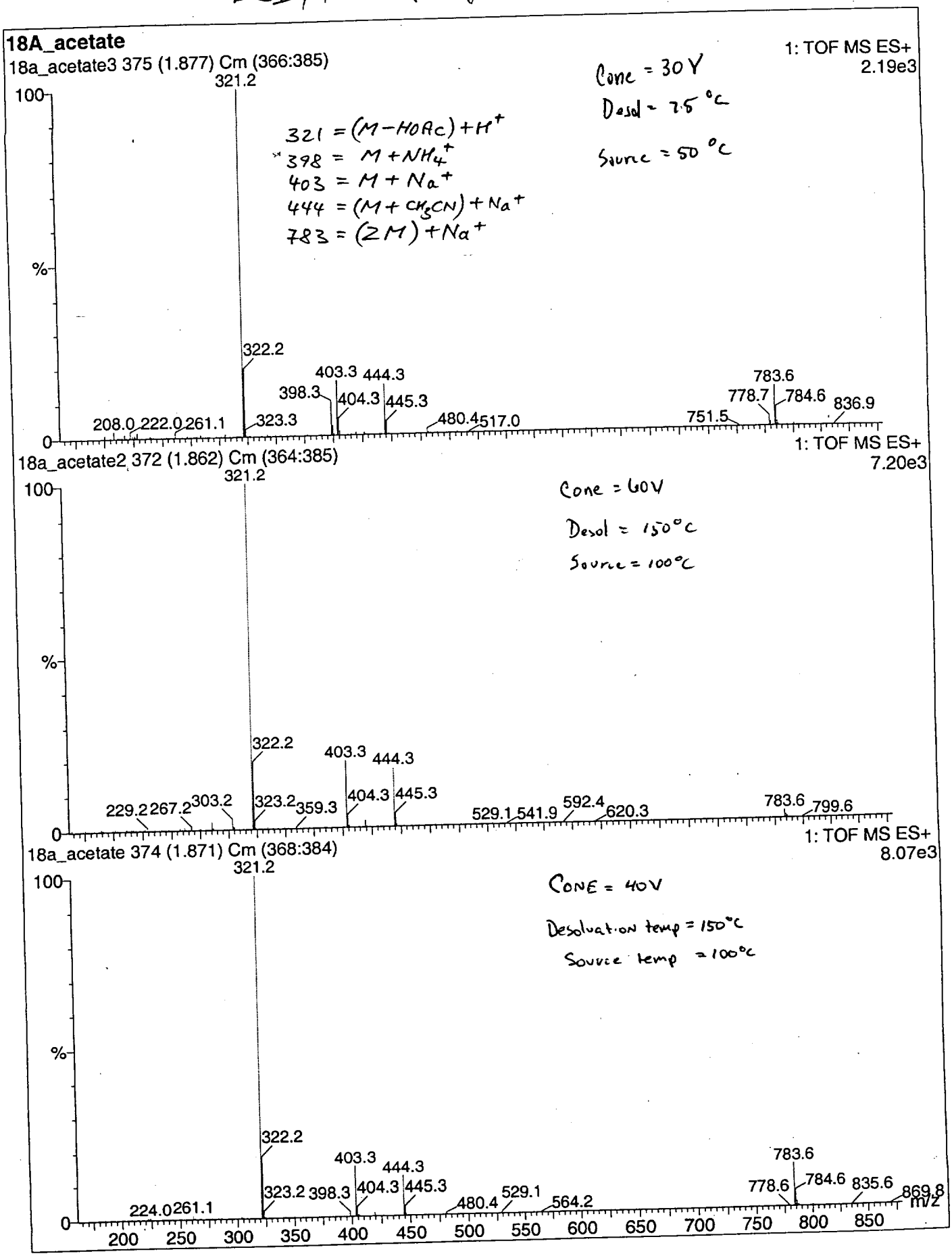

5,1:a,16
18A_acetate
27-DEC-1999
18a_acetate
1: TOF MS ES+
TIC
1.42e3
100
%
0
1.87
321
1.89
321
1.04
327
1.64
321
2.17
261
2.48
261
2.60
227
2.91
227
3.07
227
0.50
1.00
1.50
2.00
2.50
3.00
Time

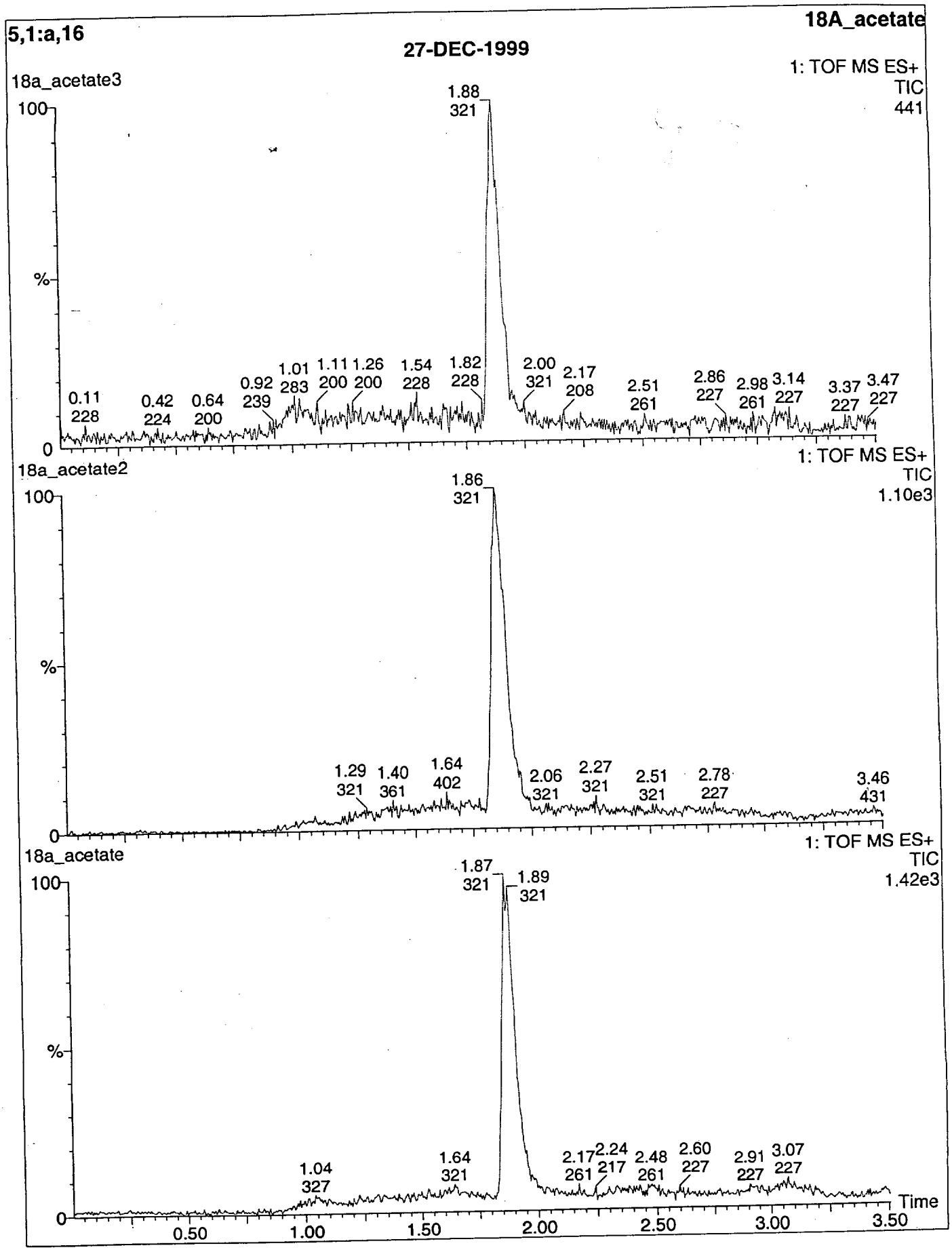
ESI HOT (exact mass by peak matching w/ int. standard)




5,1:a,16
18a_acetate4
100
%
0
1.87
321.2
1.86
321.2
Caffeine [MH+ = 195.08820 ~~194.08038~~]
C8H10N4O2
Erythromycin
C37H67N1O13 [MH+ = 734.46907]
0.50
1.00
1.50
2.00
2.50
3.00
Time
5,1:a,16
1: TOF MS ES+
BPI
285

ESI / TOF (voltage & temperature variation)




5,1:a,16
27-DEC-1999
18A_acetate
18a_acetate3
1: TOF MS ES+
TIC
441
100
%
0
1.88
321
0.11
228
0.42
224
0.64
200
0.92
239
1.01
283
1.11
200
1.26
200
1.54
228
1.82
228
2.00
321
2.17
208
2.51
261
2.86
227
2.98
261
3.14
227
3.37
227
3.47
227
18a_acetate2
1: TOF MS ES+
TIC
1.10e3
1.86
321
1.29
321
1.40
361
1.64
402
2.06
321
2.27
321
2.51
321
2.78
227
3.46
431
18a_acetate
1: TOF MS ES+
TIC
1.42e3
1.87
321
1.89
321
1.04
327
1.64
321
2.17
261
2.24
217
2.48
261
2.60
227
2.91
227
3.07
227
Time
0.50
1.00
1.50
2.00
2.50
3.00
3.50