

Highly Enantioselective and Recyclable Organocatalytic Michael Addition of Malonates to α,β -Unsaturated Aldehydes in Aqueous Media

Subrata K. Ghosh, Kritanjali Dhungana, Allan D. Headley,* and Bukuo Ni*

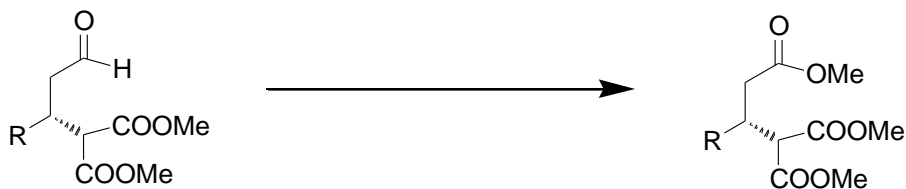
Department of Chemistry, Texas A&M University-Commerce, Commerce, TX 75429-3011,

USA

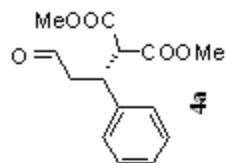
E-mail: Allan.Headley@tamuc.edu; Bukuo.Ni@tamuc.edu

Supporting Information

Oxidation of the aldehydes to the carboxylic esters for HPLC analysis:



Representative procedure: 0.10 mmol of aldehydes (addition products) were diluted with 3.0 mL *t*-BuOH, and then 3.0 mL of 1 M NaH₂PO₄ (aq.) and 3.0 mL of 1 M KMnO₄ were added successively. After 5 min of vigorous stirring 5.0 mL saturated NaHSO₃ was added and the pH was adjusted to approximately 3 with 1 M HCl. The resulting mixture was extracted 3 times with 10 mL EtOAc, the combined organic layers were washed with 10 mL of water and 10 mL brine, and dried over MgSO₄. The organic layer was concentrated in vacuum and the residual acid was dissolved in 2.0 mL toluene and 5.0 mL MeOH. TMSCHN₂ (2.0 M in *n*-hexane) was added dropwise until the yellow colour persisted. The solution was stirred for an additional 10 min and quenched with a drop of concentrated AcOH. The solvents were evaporated under vacuum. The crude product was purified by column chromatography on silica gel to yield the corresponding carboxylic ester.



Automation directory: /export/home/bun/vmr/sys/data/auts_2886_85.22
File: /export/home/bun/vmr/sys/data/auts_2886_85.22
Sample: sp-288-1

Pulse Sequence: zgpg30

Solvent: cdcl3

Operator: bun

File: sp-288-1

VNMR5-400 "nmr483"

Pulse delay: 1.000 sec

Pulse width: 2.000 sec

Acq. time: 2.619 sec

Width: 6410.3 Hz

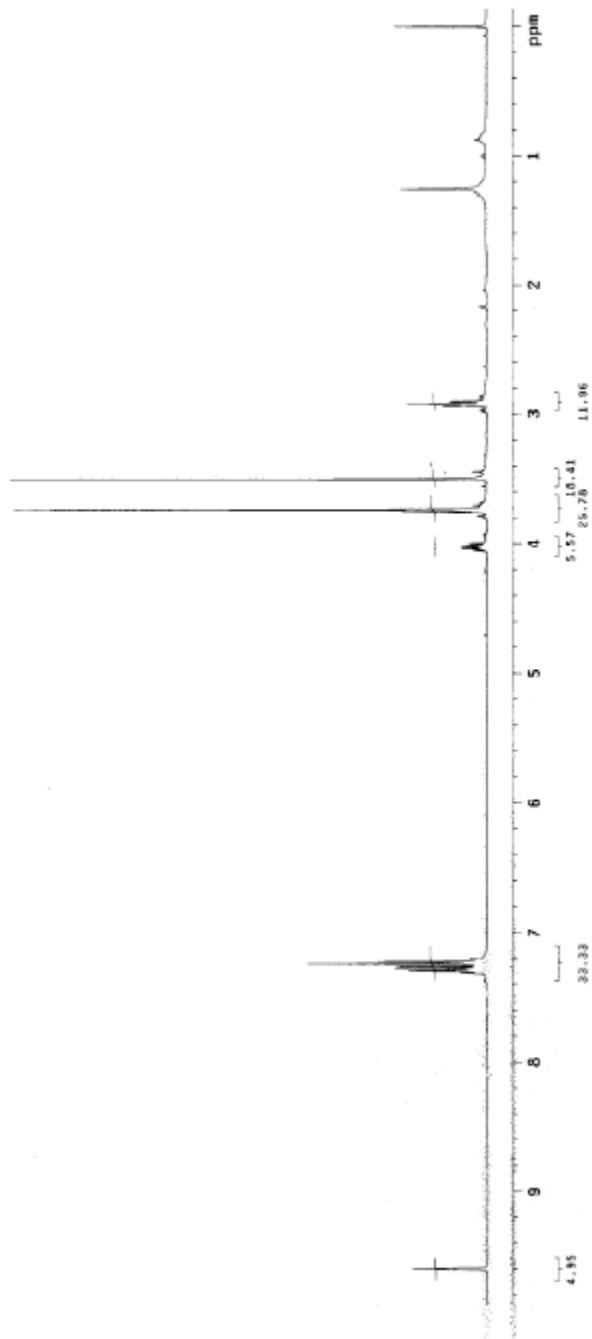
Observed frequency: 399.7309068 MHz

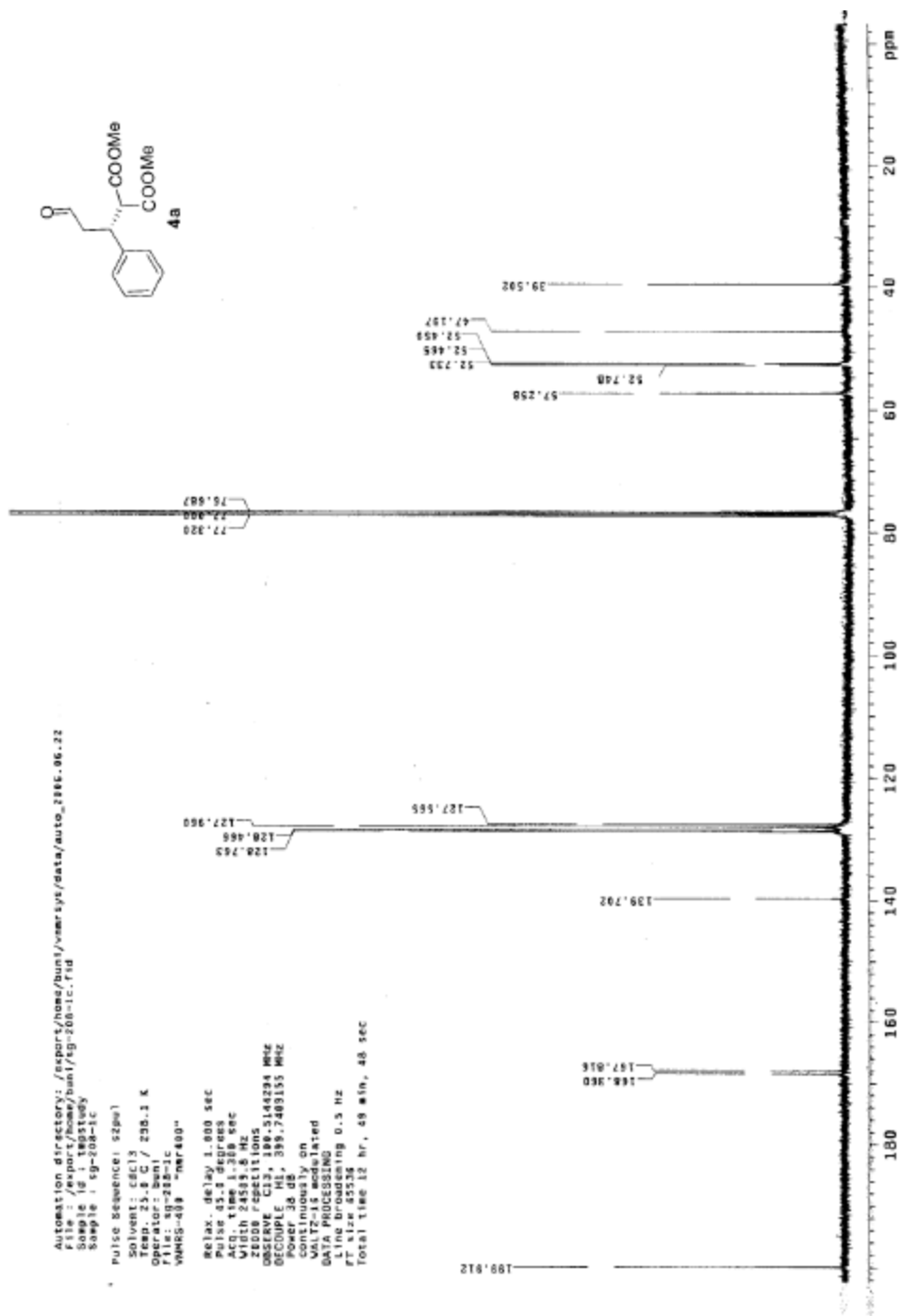
DATA PROCESSING

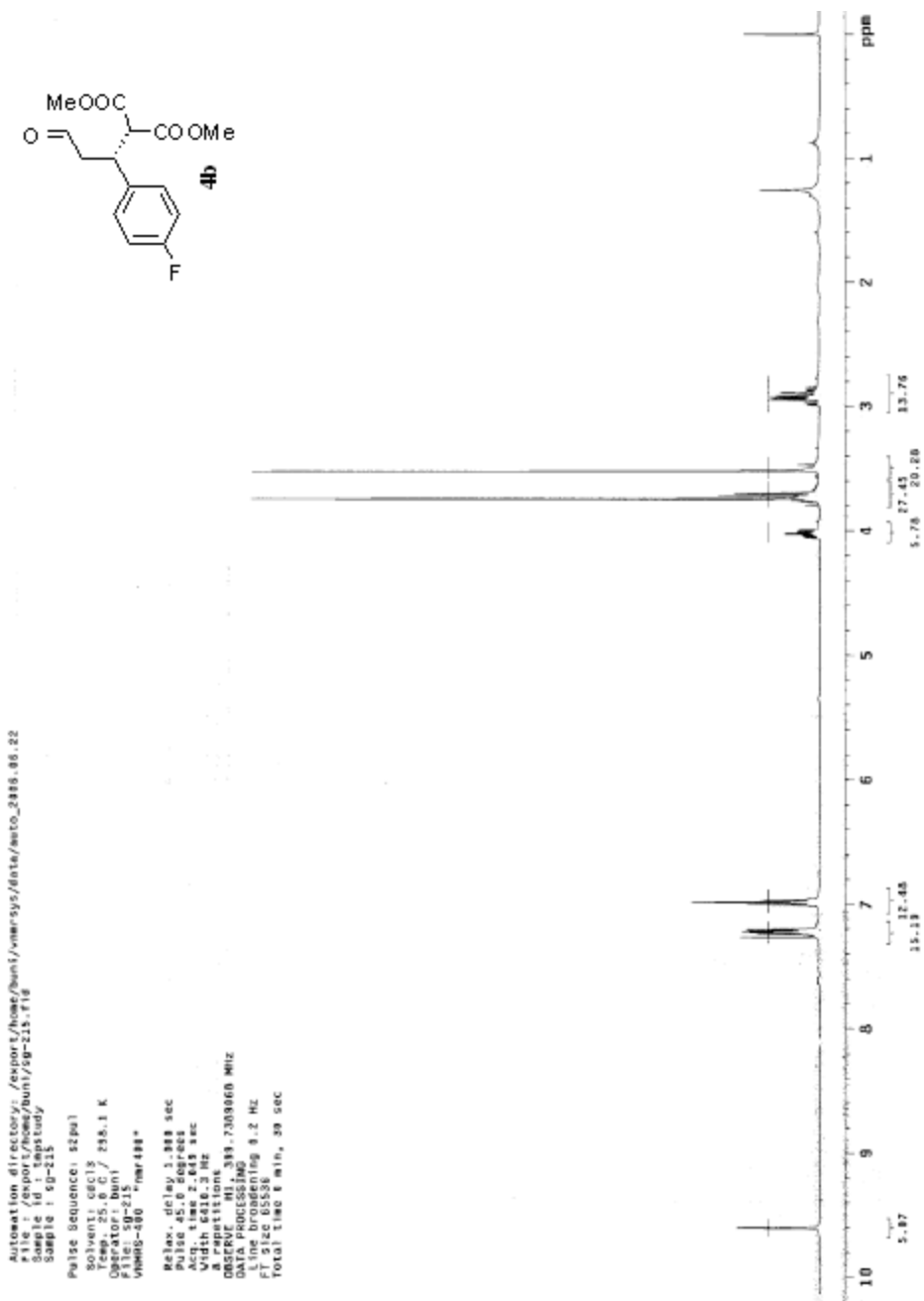
Line broadening: 0.2 Hz

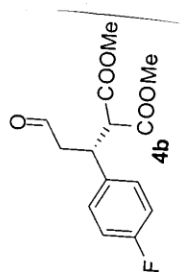
FT size: 65536

Total time: 9 min, 30 sec





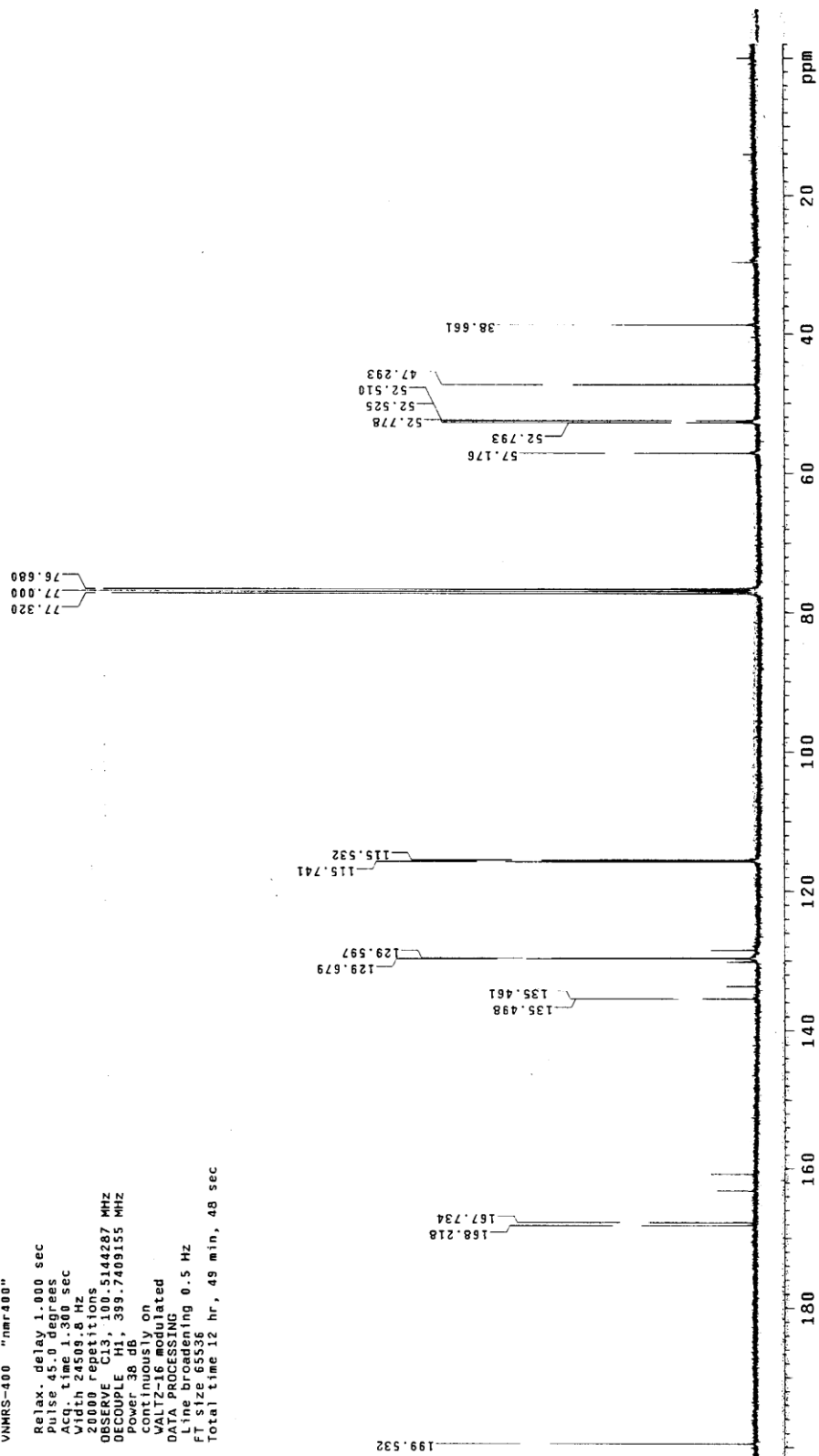


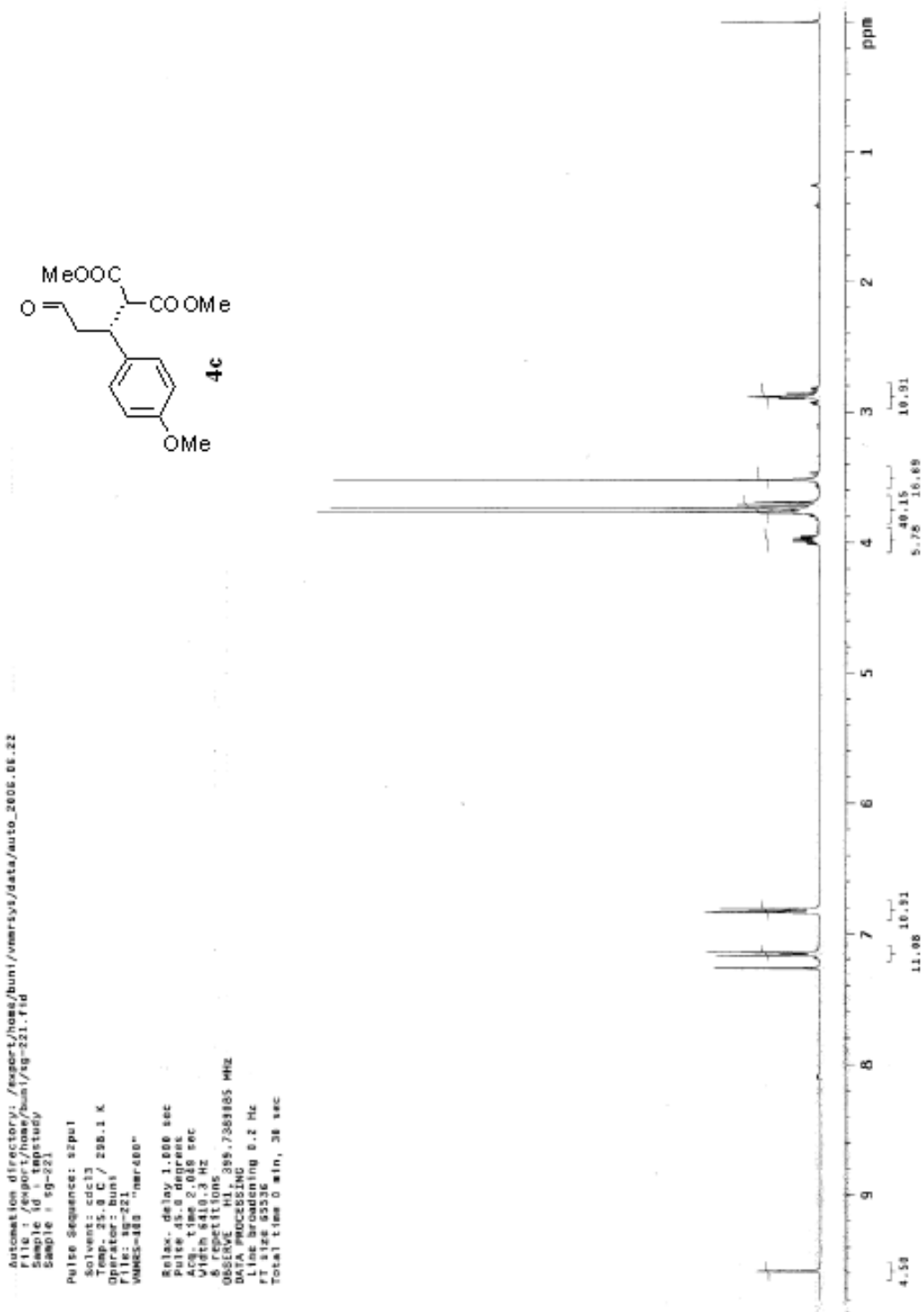


Automation directory: /export/home/bun1/vnmrSYS/data/aut0_2006.06.22
File: /export/home/bun1/sg-215c.fid
Sample id: tmpstudy
File: sg-215c
Sample: sg-215c

Pulse Sequence: s2pu1
Solvent: cdcl3
Temp: 25.0 C / 298.1 K
Operator: bun1
File: sg-215c
VNMRS-400 "nmr400"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
10 h 24509.6 Hz
260 h 24509.6 Hz
OBSERVE C13, 300.5144287 MHz
DECUPLE H1, 399.7409155 MHz
Power 38 dB,
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 12 hr, 49 min, 48 sec





Automation directory: /export/home/buni/vnmrsys/data/auto_2006_06.22
File : /export/home/buni/sg-221c.fid
Sample id : tnpstudy
Sample : sg-221

Pulse Sequence: s2pu1

Solvent: cdcl3
Temp: 300.2 K
P1: 1.00 sec
File: sg-221c
VNMRS-400 "nmr400"

Relax. delay: 1.000 sec

Pulse: 45.0 degrees

Acq. time: 1.300 sec

Width: 24509.8 Hz

20000 repetitions

OBSERVE: C13, 100.5144287 MHz

DECOUPLE: H1, 399.7409155 MHz

Power: 38 dB

continuously on

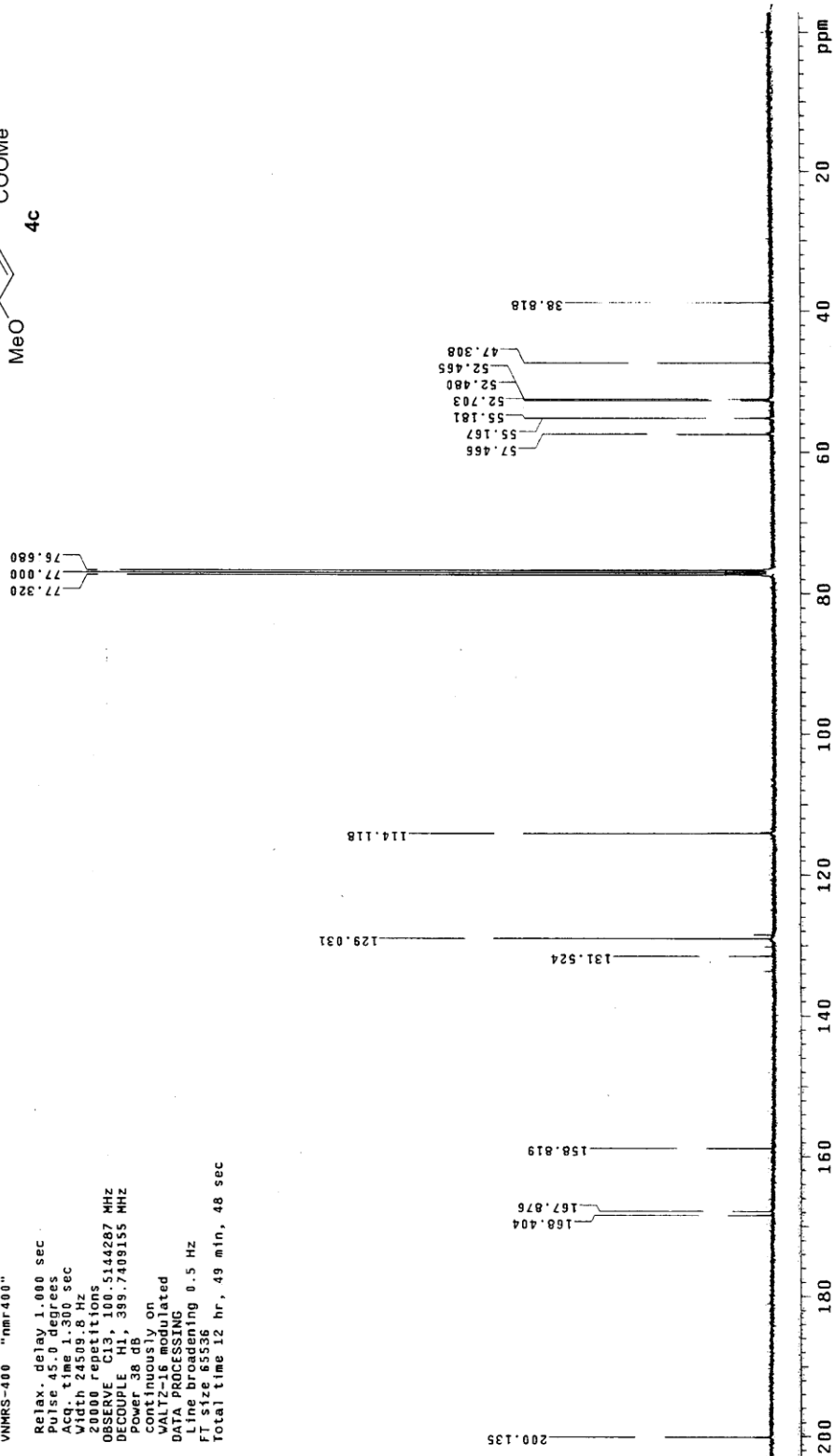
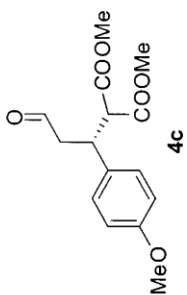
WALTZ-16 modulated

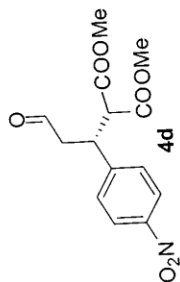
DATA PROCESSING

Line broadening: 0.5 Hz

FT size: 85336

Total time: 12 hr, 49 min, 48 sec

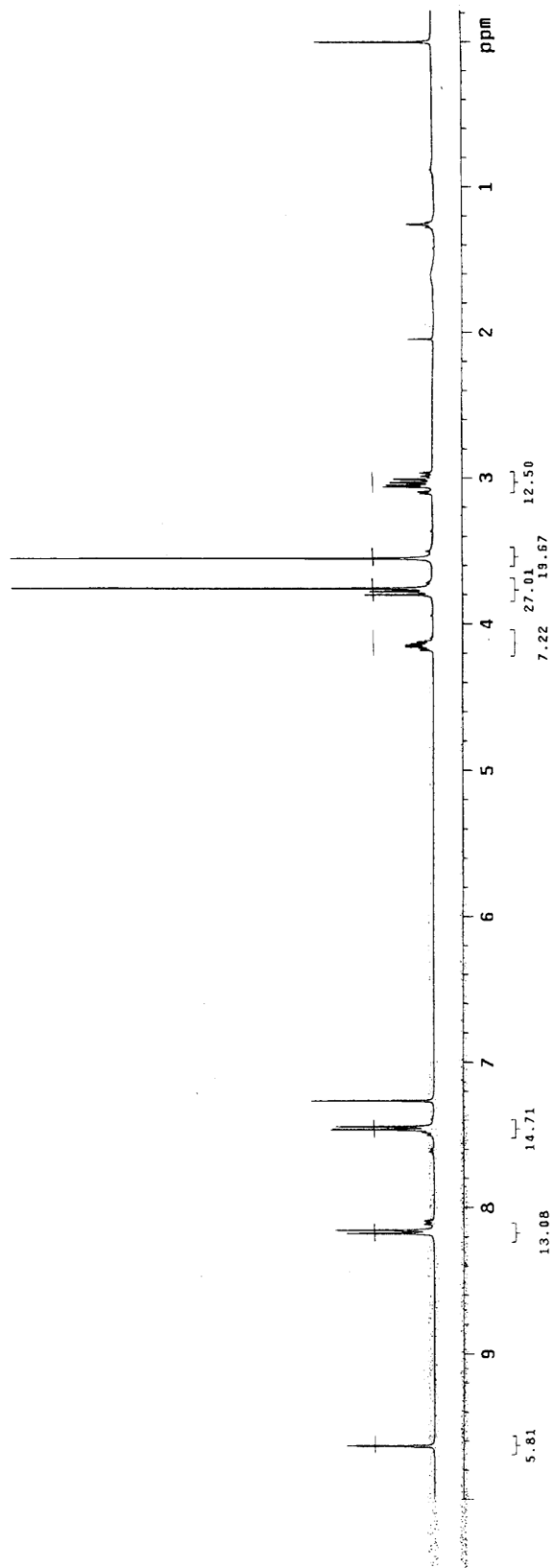


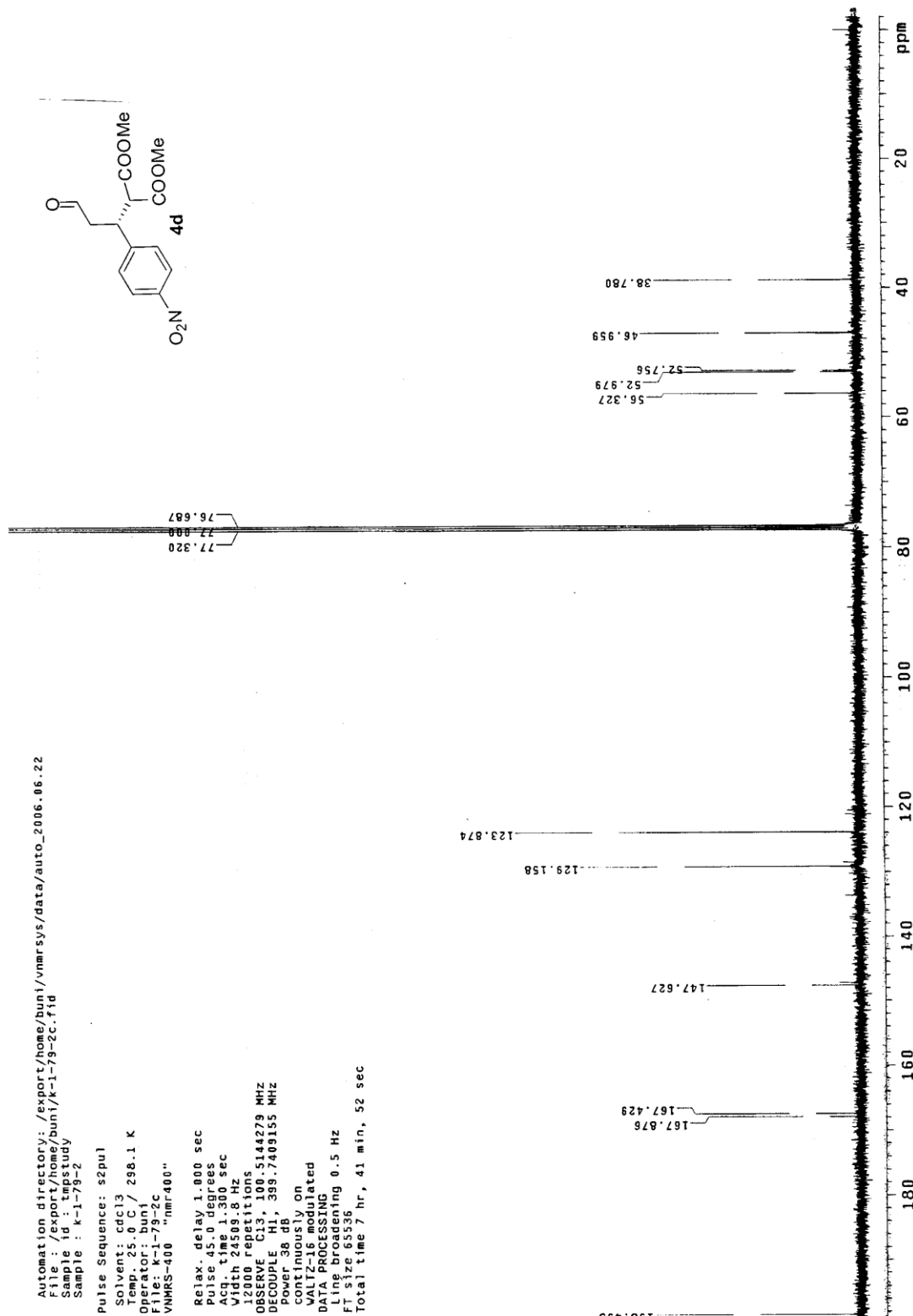


Automation directory: /export/home/buni/vnmrsys/data/auto_2006.06.22
File: /export/home/buni/k-1-77.fid
Sample id: tmpstudy
Sample: k-1-77

Pulse Sequence: s2pu1
Solvent: cdcl3
Temp: 25.0 C / 298.1 K
Operator: buni
File: k-1-77
VNMRS-400 "nmr400"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.049 sec
Width 840.3 Hz
OBSERVE 1Homs 399.7389077 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FI size 65536
Total time 0 min, 30 sec





Automation directory: /export/home/bunt/vnmr/sv/sets/astro_2006_06.22
File: /export/home/bunt/vnmr/sv/sets/astro_2006_06.22
Sample: 50-214

Pulse sequence: zgpg30

Solvent: CDCl3
Operator: bunt
File: 50-214

VNAME=489 "nmr400"

Pulse delay: 1.000 sec
Pulse: 400 degrees

Acq. time: 2.045 sec

Width: 410.3 Hz

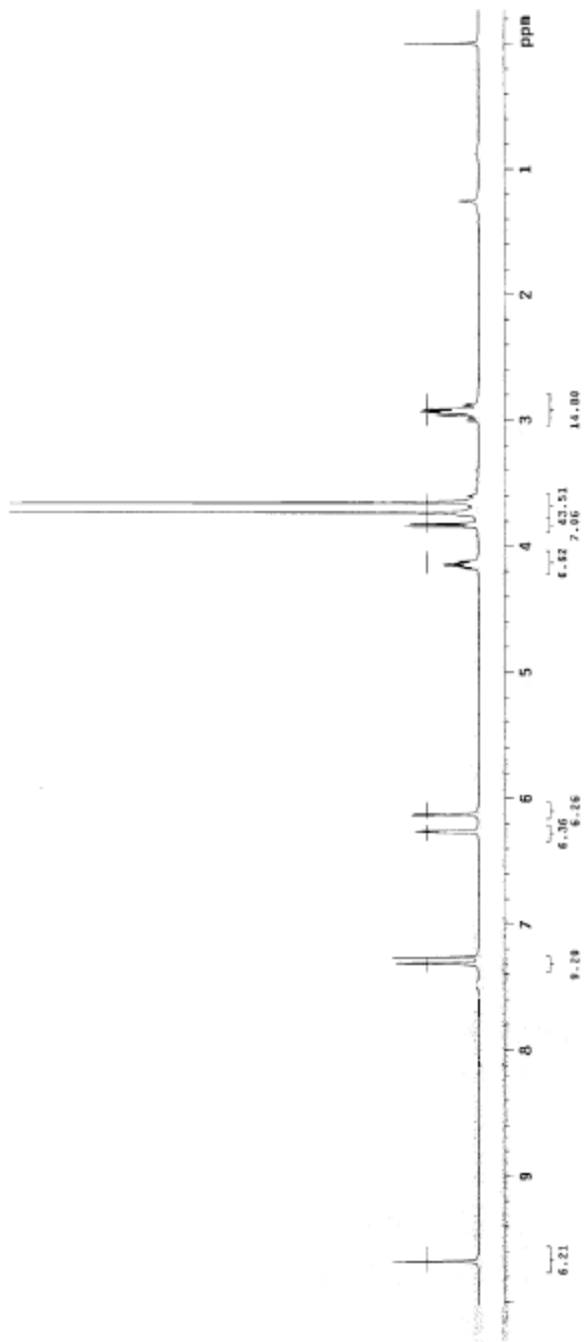
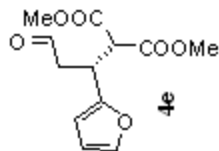
Observed: 309.7305805 MHz

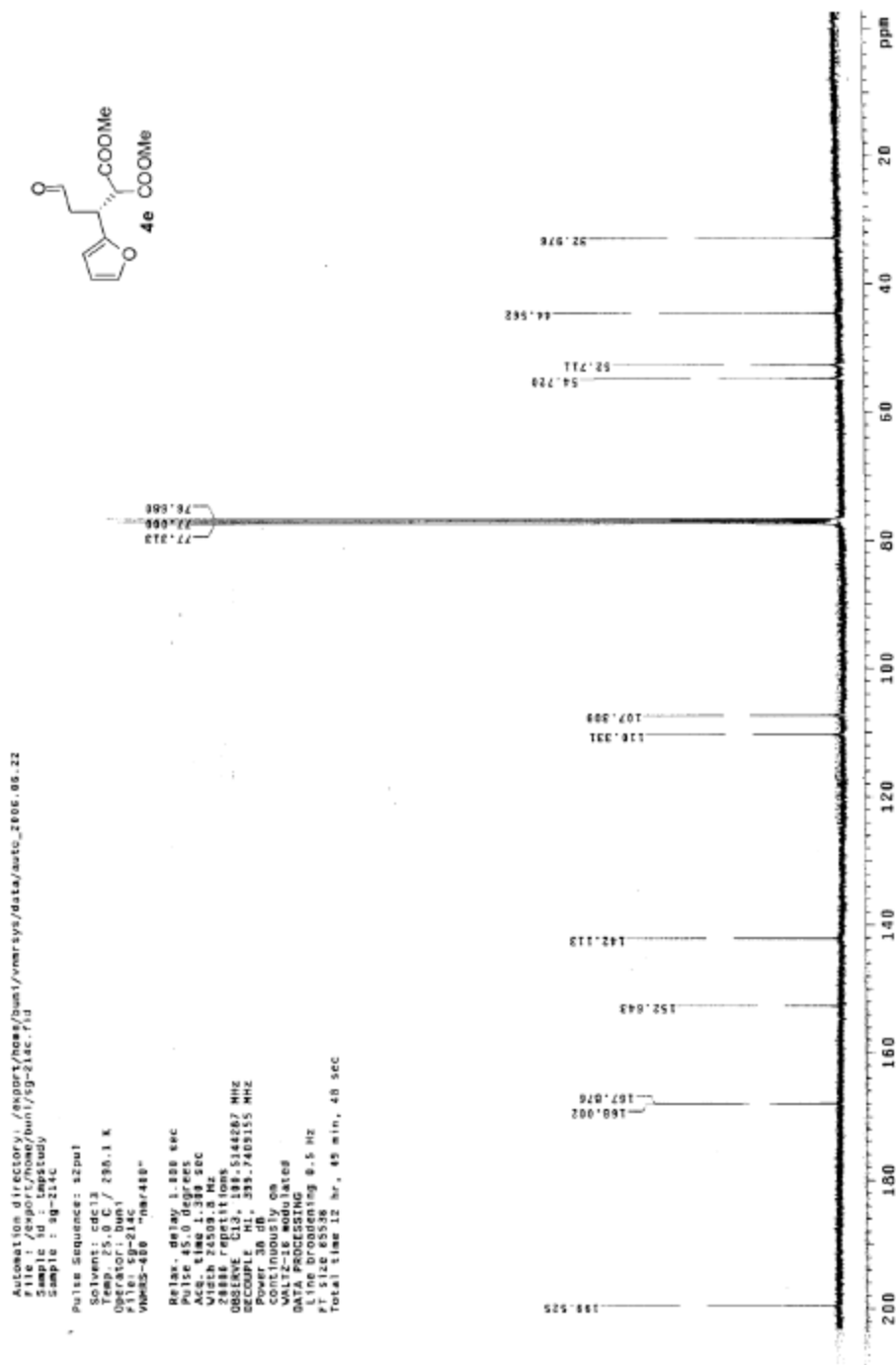
Processing: 0.2 Hz

Line broadening: 0.2 Hz

FT size: 65536

Total time: 9 min, 30 sec

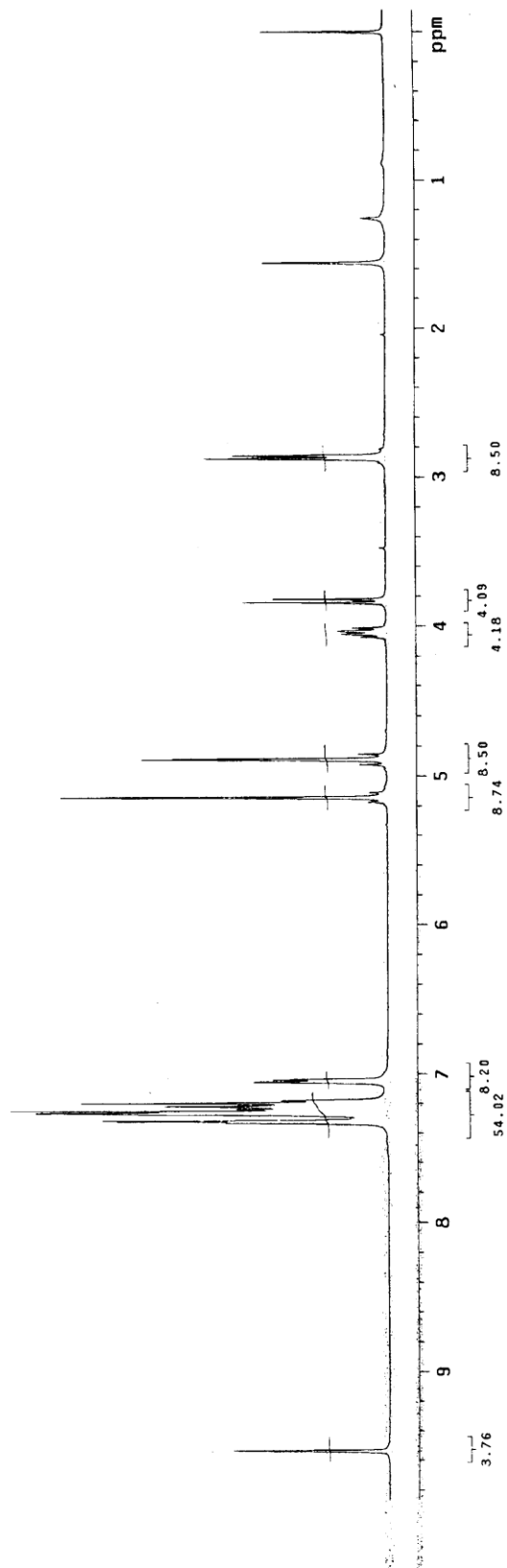
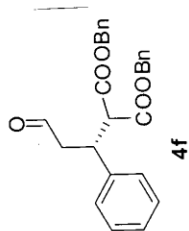


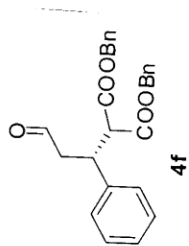


Automation directory: /export/home/buni/vnmrsys/data/auto_2006.06.22
File: /export/home/buni/sg-227.fid
Sample id: tmostudy
Sample: sg-227

Pulse Sequence: szpul
Solvent: cdcl3
Temp: 25.0 C / 298.1 K
Operator: buni
File: sg-227
VNMR5-400 "nmr400"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.049 sec
Width 6410.3 Hz
8 repetitions
OBSERVED F1 399.7389128 MHz
DATA PROCESSING
File processing 0.2 Hz
FT size 65536
Total time 0 min, 30 sec





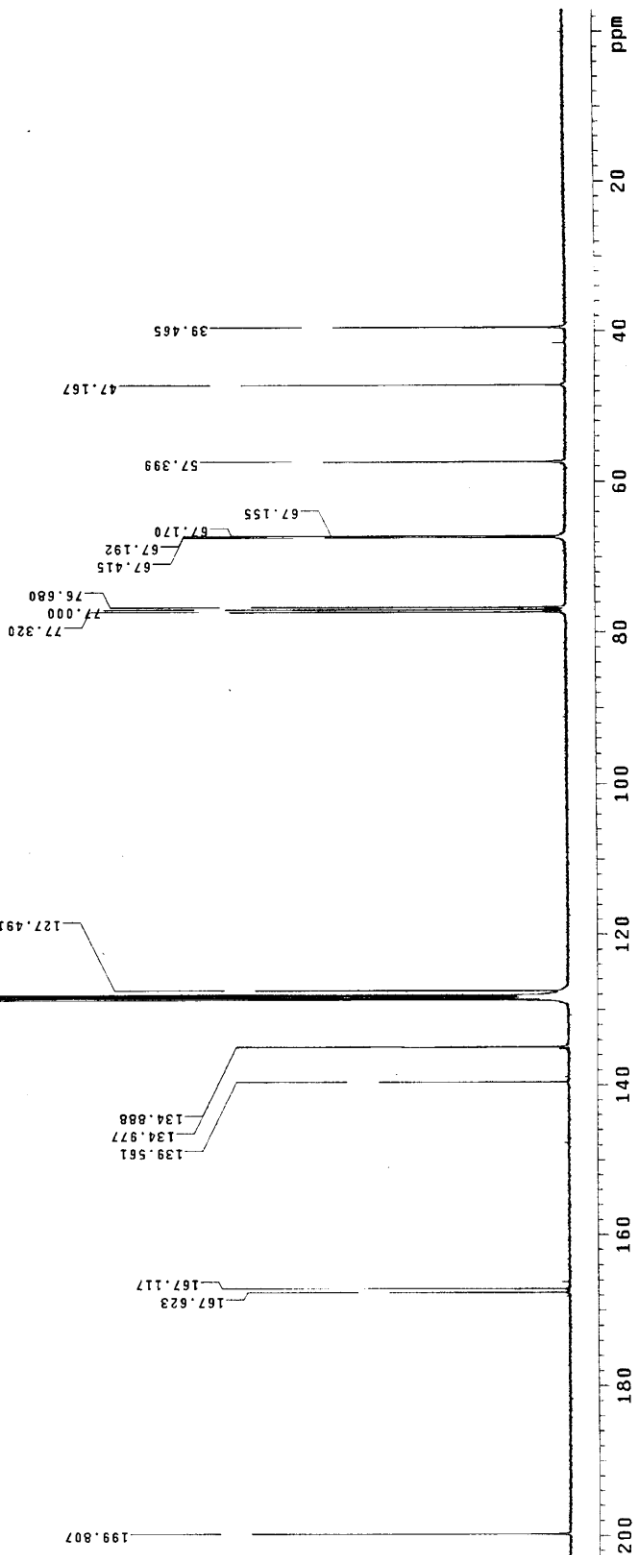
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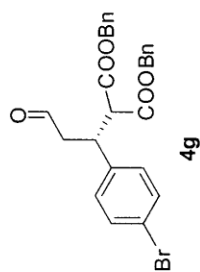
Sample id: tmpstudy
Sample: sg-227c

Pulse Sequence: s2pul

Solvent: cdcl3
Temp: 25.0 C / 298.1 K
Operator: buni
File: sg-227c
VNMR-400 "nmr400"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 24509.8 Hz
Spectral width 31000.000 Hz
OBSERVE CH1, 399.740155 MHZ
DECUPLE CH1, 399.740155 MHZ
Power 38 dB,
continuously on
VATZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 12 hr, 49 min, 48 sec

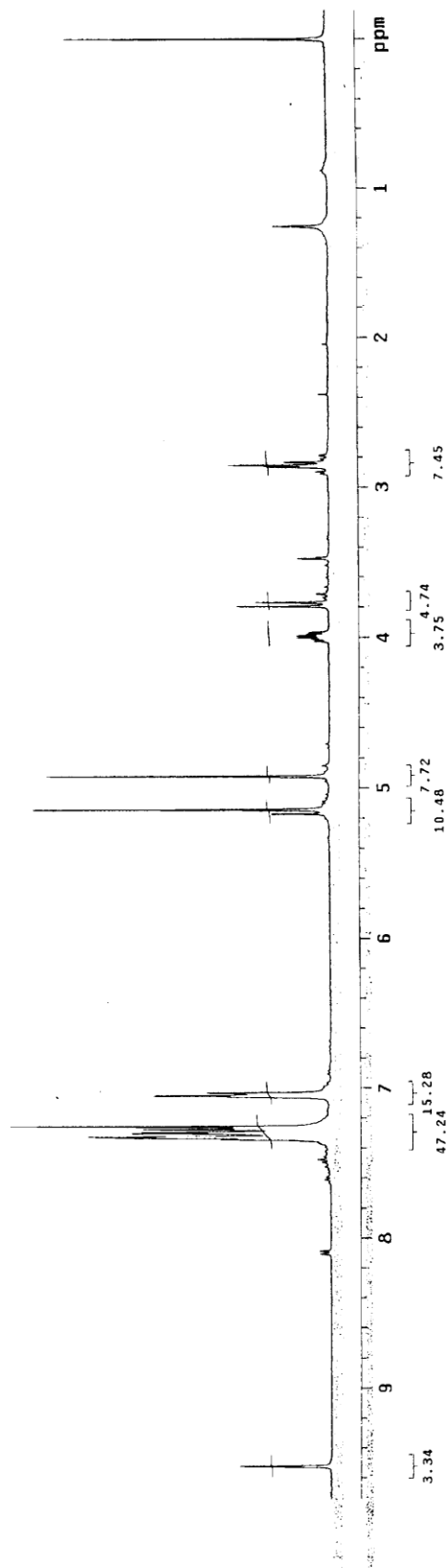


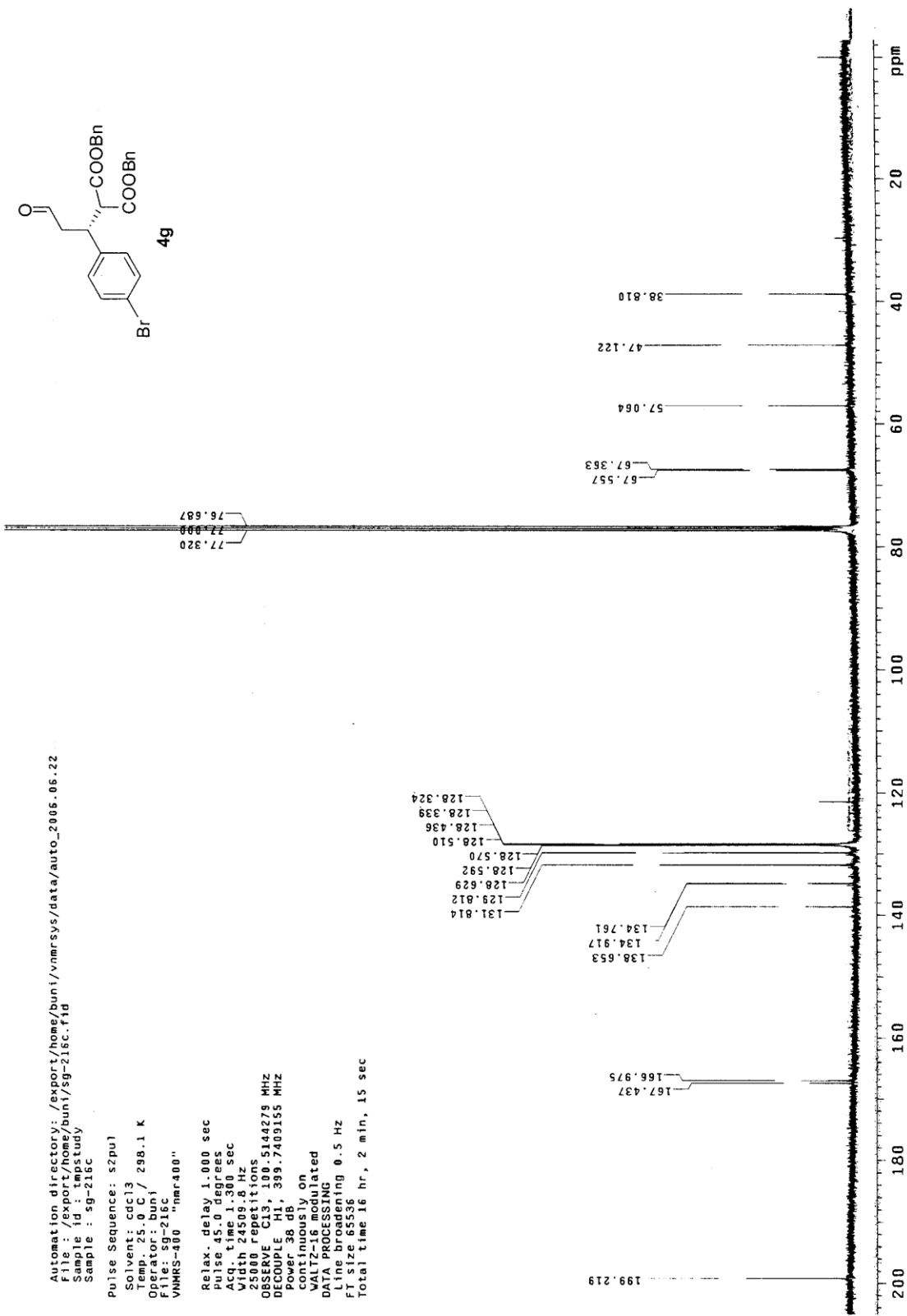


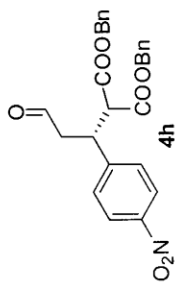
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File: /export/home/bunif/vnmrSYS/data/auto_2006.06.22
Sample id: tmpstudy
Sample: sg-216

Pulse Sequence: szpul
Solvent: cdcl3
Temp: 25.0 C / 298.1 K
Operator: bunif
File: sg-216
VNMR-400 "nmr400"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.049 sec
Width 5410.3 Hz
8 repetitions
OBSERVE F1, 399.7369085 MHz
DATA PROCESSING
F1 size 65536
Total time 0 min, 30 sec



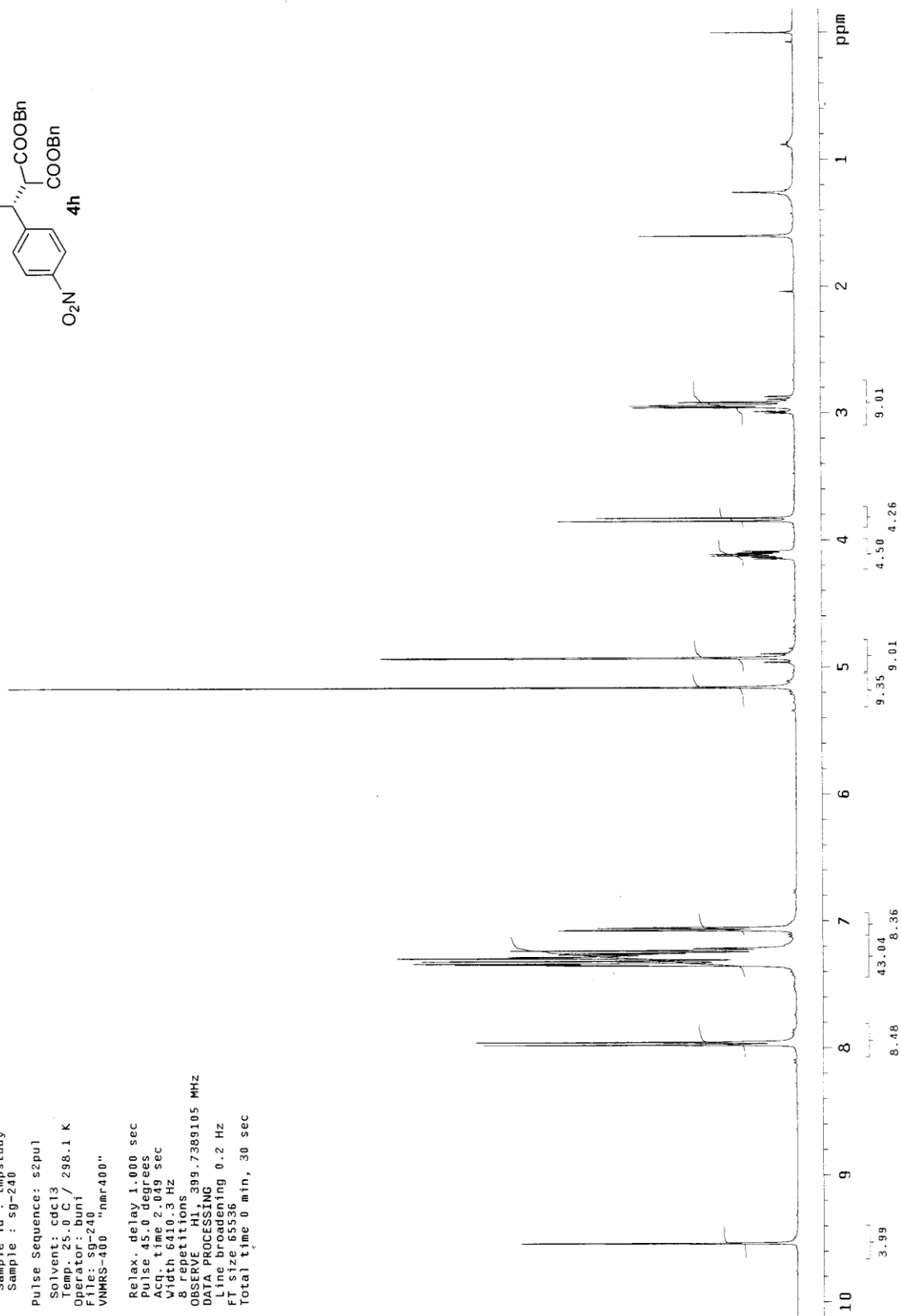




Automation directory: /export/home/walkup/auto_2012_06.20
File: /export/home/bun1/sg-240.fid
Sample id: tmpstudy
Sample: sg-240

Pulse Sequence: s2pu1
Solvent: cdcl3
Temp: 25.0 C / 298.1 K
Operator: bun1
File: sg-240
VNMRS-400 "nmr400"

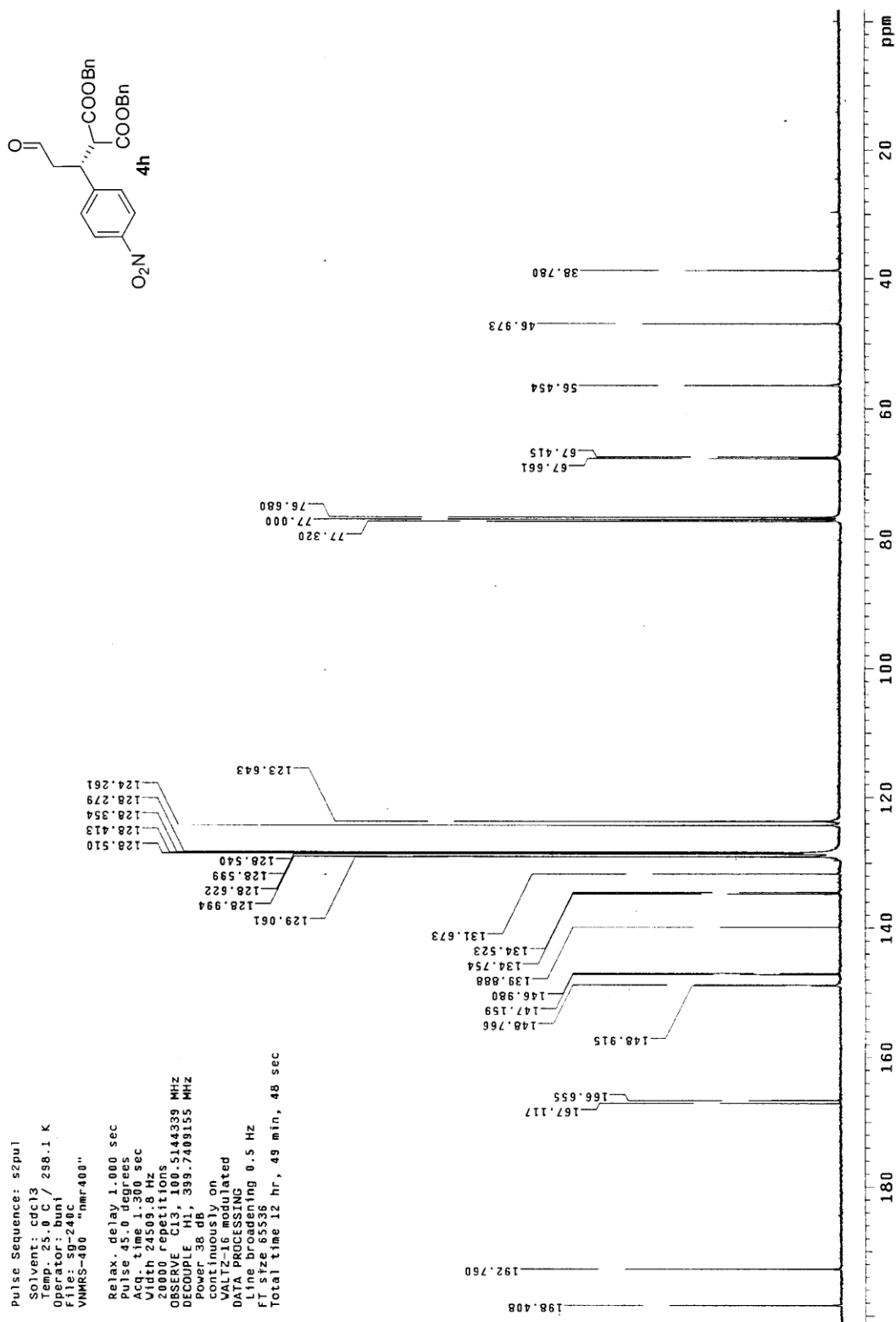
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.049 sec
Width 6410.3 Hz
8 repetitions
OBSERVE H1, 399.7389105 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min, 30 sec

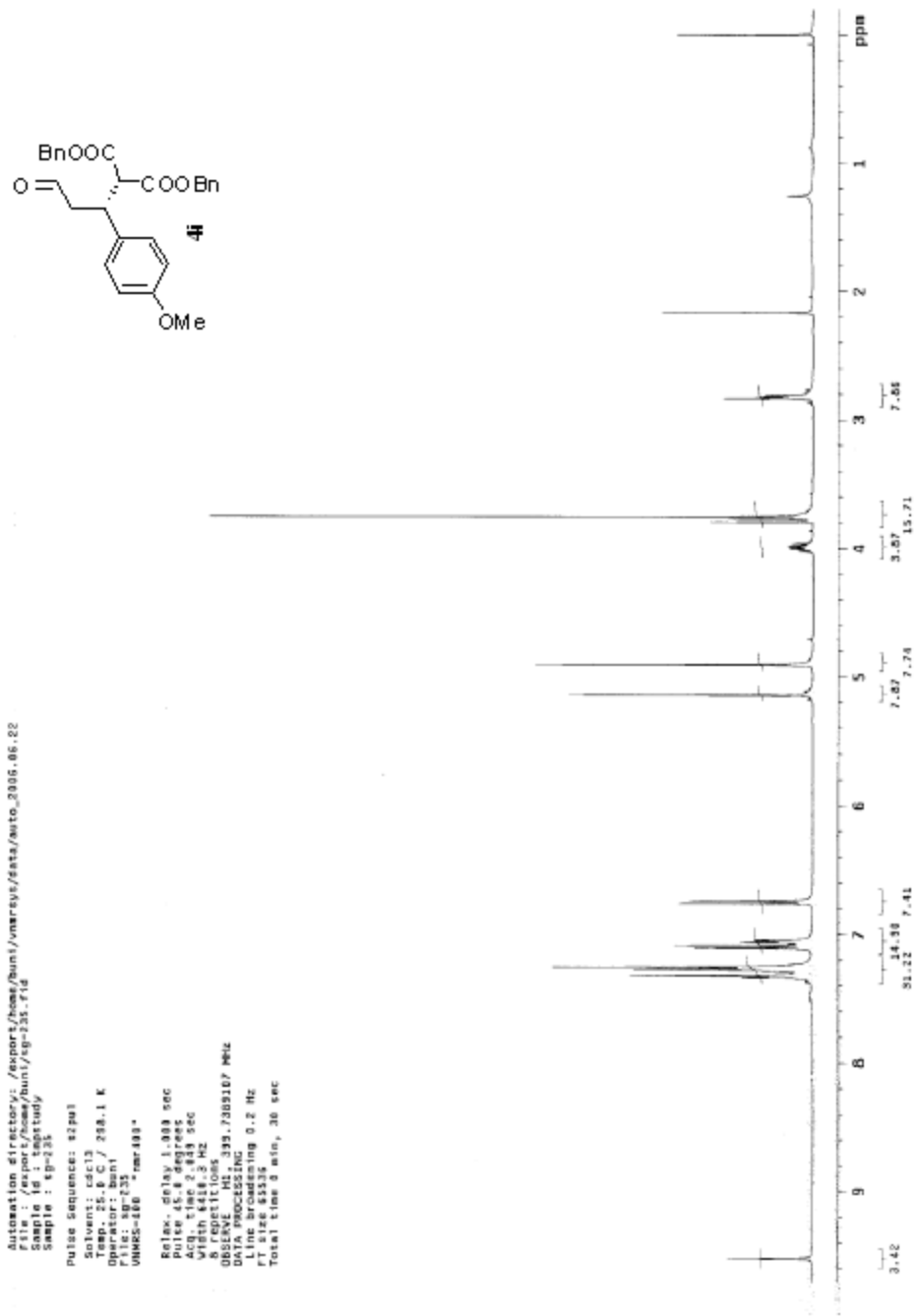


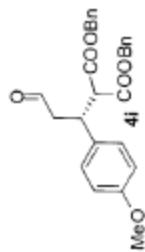
Automation directory: /export/home/bunl/vnmrsvs/data/auto_2006_06.22
File: /export/home/bunl/vnmrsvs/data/sg-240c.fid
Sample id: tmpstudy
Sample: sg-240c

Pulse Sequence: s2pul
Solvent: cdcl3
Temp: 25.00 C / 298.1 K
Operator: bunl
File: sg-240c
VNMR-400 "nmr-400"

Relax. delay: 1.000 sec
Pulse: 45.0 degrees
Acq. time: 1.300 sec
Width: 24509.8 Hz
20000 repetitions
OBSERVE C13, 100.5144339 MHz
DECOUPLE H1, 399.7409155 MHz
Power: 38 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening: 0.5 Hz
F1 size: 83509
Total time: 12 hr, 49 min, 48 sec



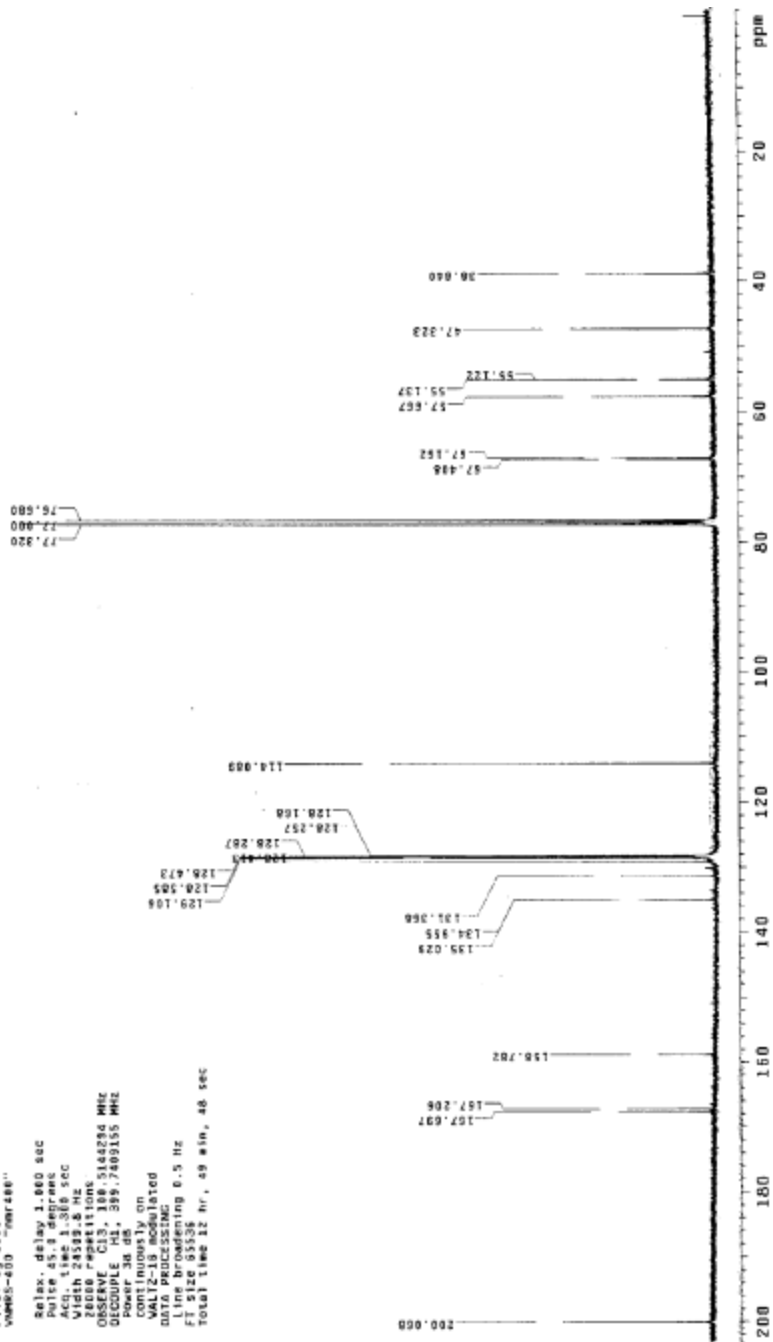




Automation directory: /export/home/sumi/vnmrsys/data/aut_2006_04_22
 File: /export/home/sumi/vnmrsys/data/aut_2006_04_22
 Sample ID: 4j-233c
 Sample: 4j-233c

Pulse Sequence: zgpg30
 Solvent: dms-d6
 Temp: 25.3 C / 250.1 K
 Observed: 300
 File: 4j-233c
 VNAME: 4j-233c "nmr466"

Relax. delay: 1.000 sec
 Pulse: 45.0 degrees
 C13: 100.625 MHz
 C13: 100.625 MHz
 Width: 24548.8 Hz
 26000 Repetitions
 OBSERVE: C13, 100.625 MHz
 DECOUPLE: H1, 500.136 MHz
 Acquisition on
 CONTINUOUSLY on
 VOLTAGE: 19.000 V
 DATA PROCESSING
 Line broadening: 6.5 Hz
 Frequency offset: 0.000 Hz
 Total time: 22 hr, 09 min, 48 sec



Automation directory: /export/home/bunt/vnmrpy/data/auto_2012.06.22

File: /export/home/bunt/vnmrpy/data/auto_2012.06.22

Sample id: 1: teststudy

Sample: 1: sp-234

Pulse sequence: zgpg30

Solvent: cdcl3

Temp: 25.0 C / 296.1 K

Operator: bunt

File: /export/home/bunt/vnmrpy/data/auto_2012.06.22

VNMR-2D: vnmr43g*

Relax: delay 1.000 sec

Pulse: 45.0 degrees

Acq: time 2.043 sec

RG: 655.360 Hz

8: FIDRES11.000 Hz

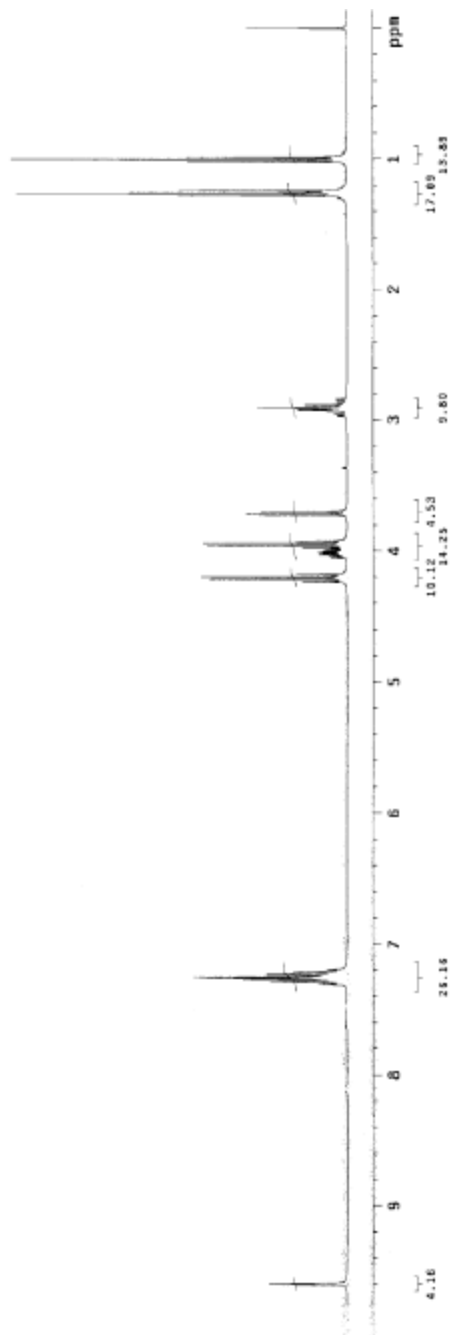
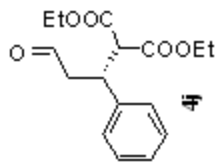
DDSERVE: H1: 333.7349505 MHz

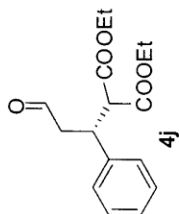
DATA PROCESSING

FT size: 65536

FT size: 65536

Total time: 8 min, 30 sec

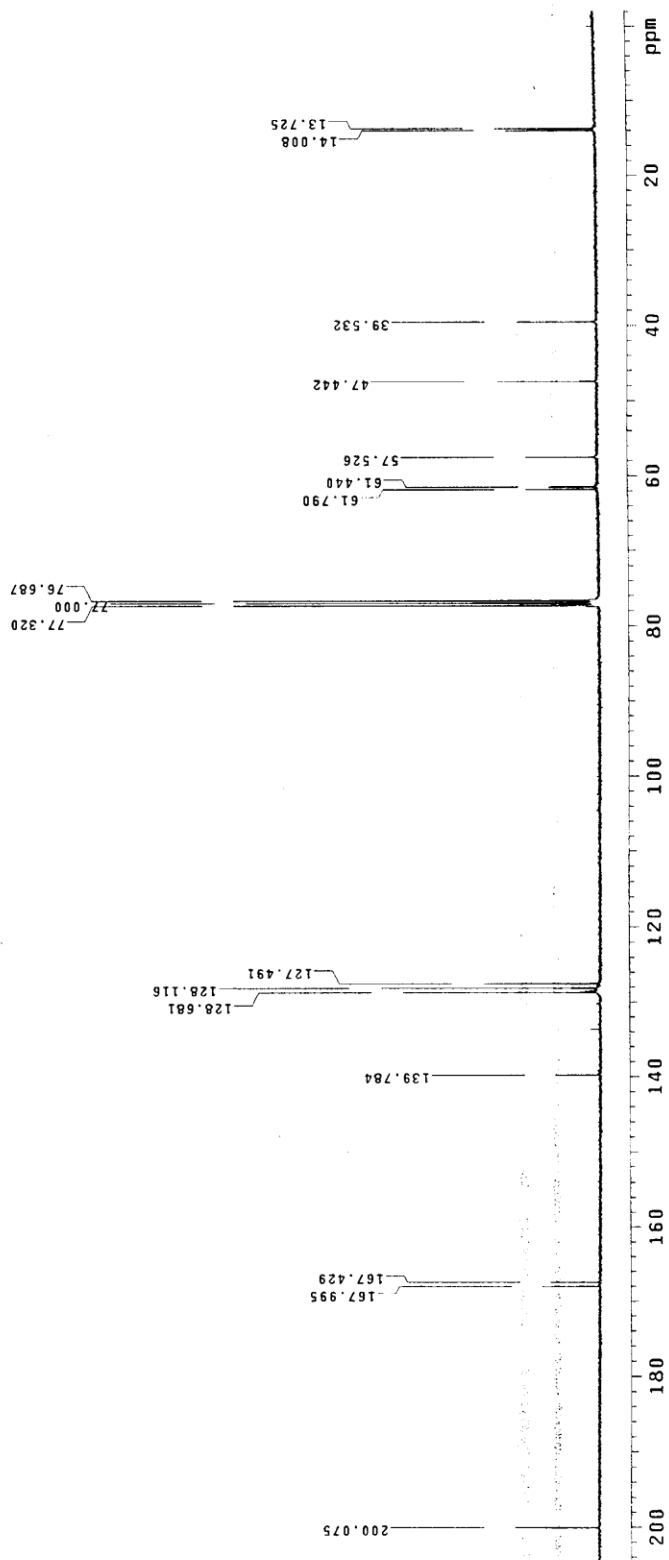


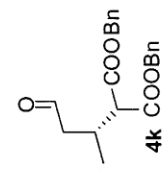


Automation directory: /export/home/buni/vnmrsys/data/auto_2006.0c.22
File: /export/home/buni/sg-234c.fid
Sample id: tmpstudy
Sample : sg-234c

Pulse Sequence: s2pul
Solvent: cdcl3
Temp: 25.0 C / 298.1 K
Operator: buni
File: sg-234c
VNMR-400 "nmr400"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 24500.0 Hz
OBSERVE CH 100.5144287 MHz
DECUPLE CH 1, 399.7409155 MHz
Power 38 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 12 hr, 49 min, 48 sec

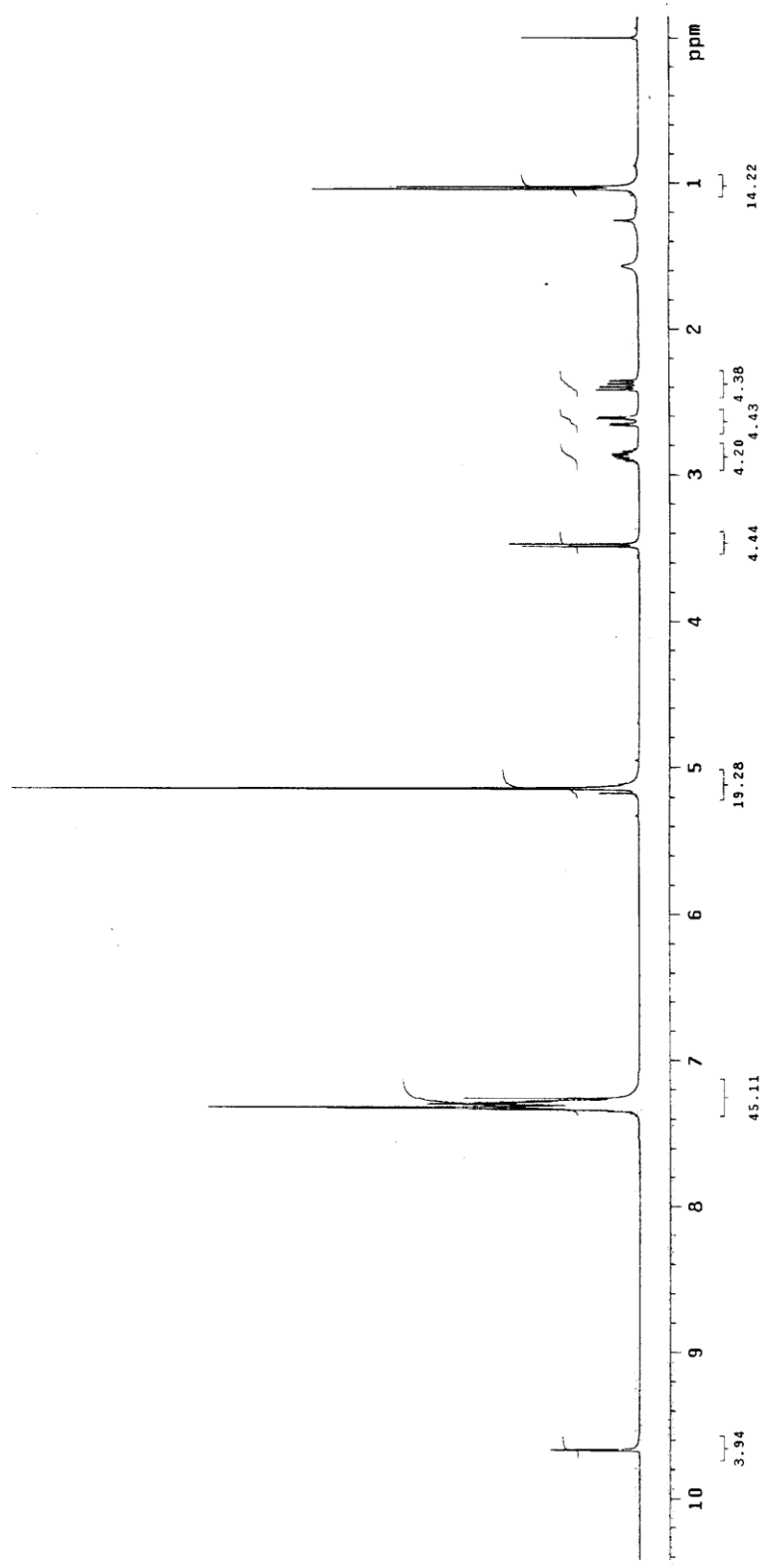


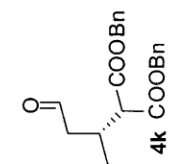


Automation directory: /export/home/bun1/vnmrsys/data/aut0_2006.06.22
File: /export/home/bun1/vnmrsys/data/aut0_2006.06.22
Sample id: tmpstudy
Sample: sg-239

Pulse Sequence: s2pu1
Solvent: cdcl3
Temp: 25.0 C / 298.1 K
Operator: bun1
File: sg-239
VNMRS-400 "nmr400"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.049 sec
F1 freq 99.626 MHz
& resolution 0.08 Hz
OBSERVE H1 399.7369099 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min, 30 sec

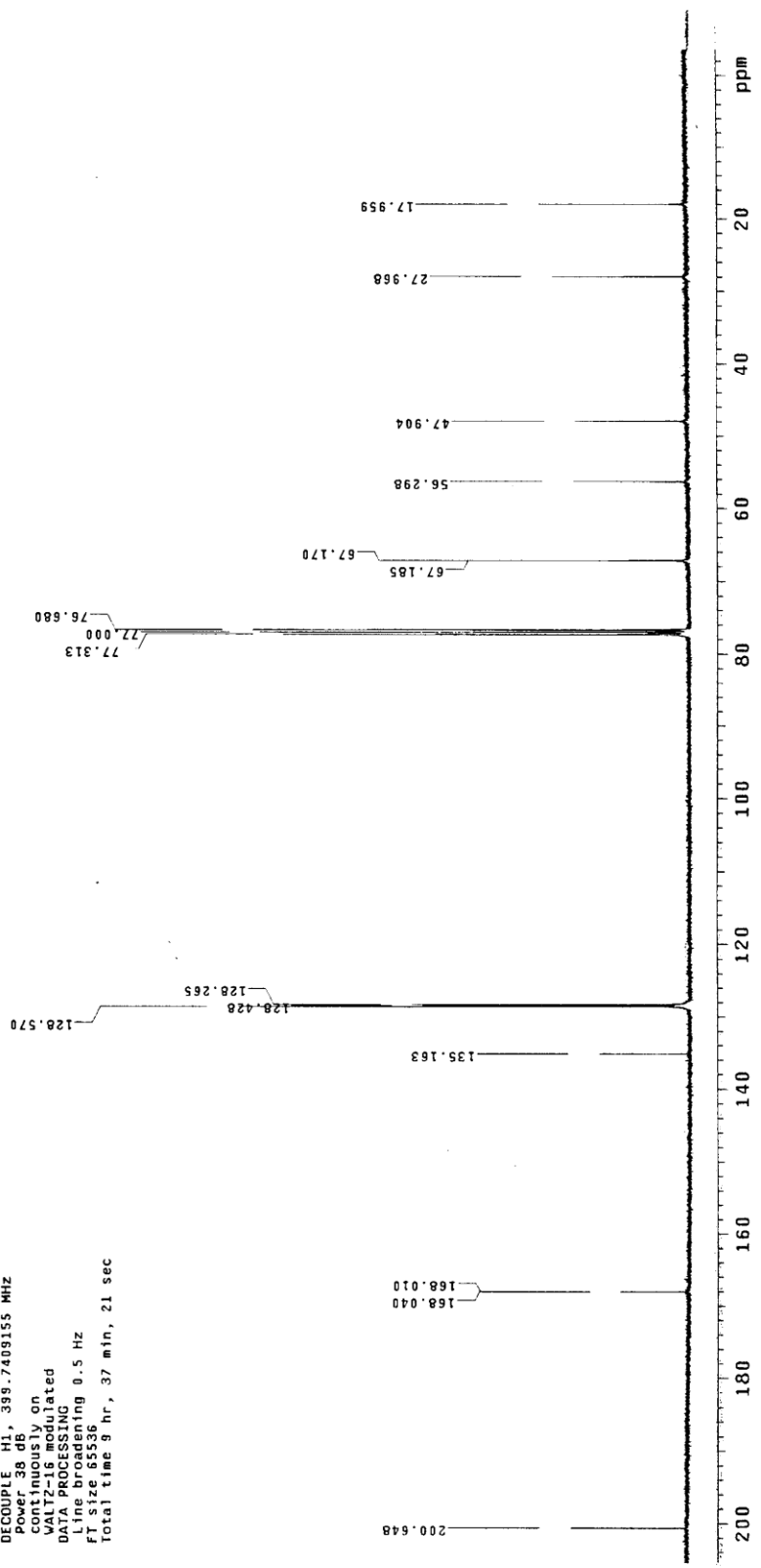




Automation directory: /export/home/bun1/vnmrSYS/data/au0_2006.06.22
File: /export/home/bun1/sg-239c.fid
Sample id: tmpstudy
Sample: sg-239c

Pulse Sequence: s2pu1
Solvent: cdCl3
Temp: 25.0 C / 298.1 K
Operator: bun1
File: sg-239c
VNMRS-400 "nmr400"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 24509.6 Hz
Virt. Recit: 1.05
OBSERVE C13, 100.5144294 MHz
DECUPLE H1, 399.7409155 MHz
Power 38 dB,
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 9 hr, 37 min, 21 sec

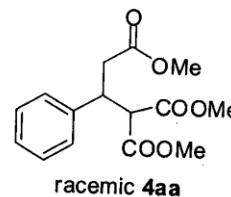


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==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\subrata\k-2-4.lcd

Acquired by : Admin
Sample Name : k-2-4
Sample ID : k-2-4
Vial # :
Injection Volume : 5 uL
Data File Name : k-2-4.lcd
Method File Name : 80%-Hexane-20%-isopropanol-35min (0.5 mL, 254nm).lcm
Batch File Name :
Report File Name : Default.lcr
Data Acquired : 12/21/2009 7:59:28 PM
Data Processed : 4/5/2010 5:09:13 PM

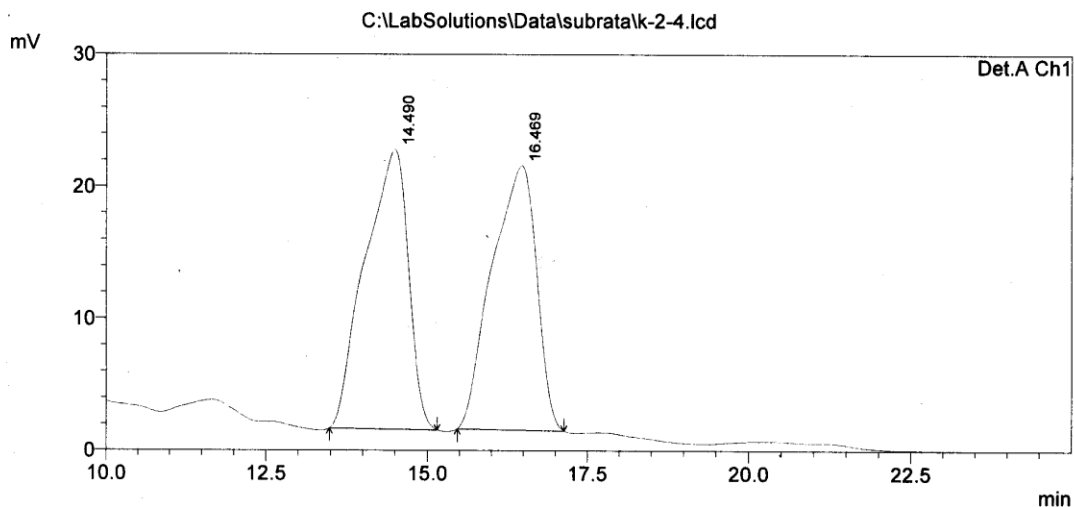


<Chromatogram>

PeakTable

Detector A Ch1 254nm

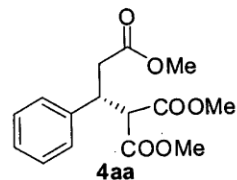
Peak#	Ret. Time	Area	Height	Area %	Height %
1	14.490	988607	21203	50.420	51.424
2	16.469	972126	20029	49.580	48.576
Total		1960733	41232	100.000	100.000



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==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\subrata\sg-208-1E.lcd
Acquired by : Admin
Sample Name : sg-208-1E
Sample ID : sg-208-1E
Vail # :
Injection Volume : 5 uL
Data File Name : sg-208-1E.lcd
Method File Name : 80%-Hexane-20%-isopropanol-35min (0.5 mL, 254nm).lcm
Batch File Name :
Report File Name : Default.lcr
Data Acquired : 12/29/2009 8:04:58 PM
Data Processed : 12/29/2009 9:07:25 PM

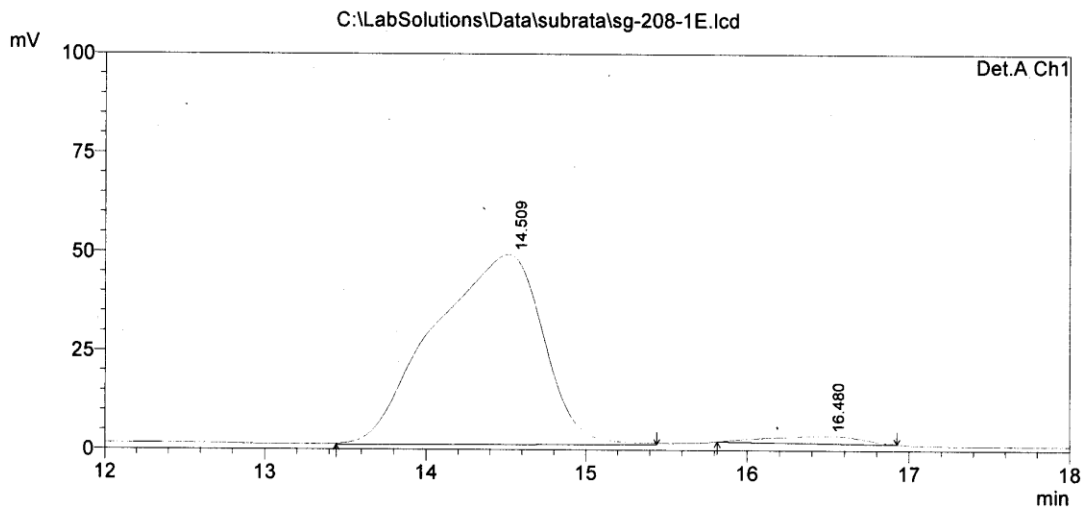


<Chromatogram>

PeakTable

Detector A Ch1 254nm

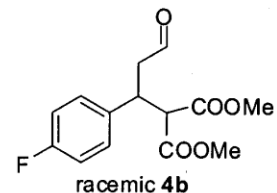
Peak#	Ret. Time	Area	Height	Area %	Height %
1	14.509	2275501	48098	96.831	96.216
2	16.480	74461	1891	3.169	3.784
Total		2349962	49990	100.000	100.000



==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\subrata\k-2-13-3c.lcd

Acquired by : Admin
Sample Name : k-2-13-3c
Sample ID : k-2-13-3c
Vial # :
Injection Volume : 5 uL
Data File Name : k-2-13-3c.lcd
Method File Name : 70%-Hexane-30%-isopropanol-30min (1.0mL, 220nm).lcm
Batch File Name :
Report File Name : Default.lcr
Data Acquired : 1/31/2010 3:34:49 PM
Data Processed : 4/5/2010 4:07:17 PM

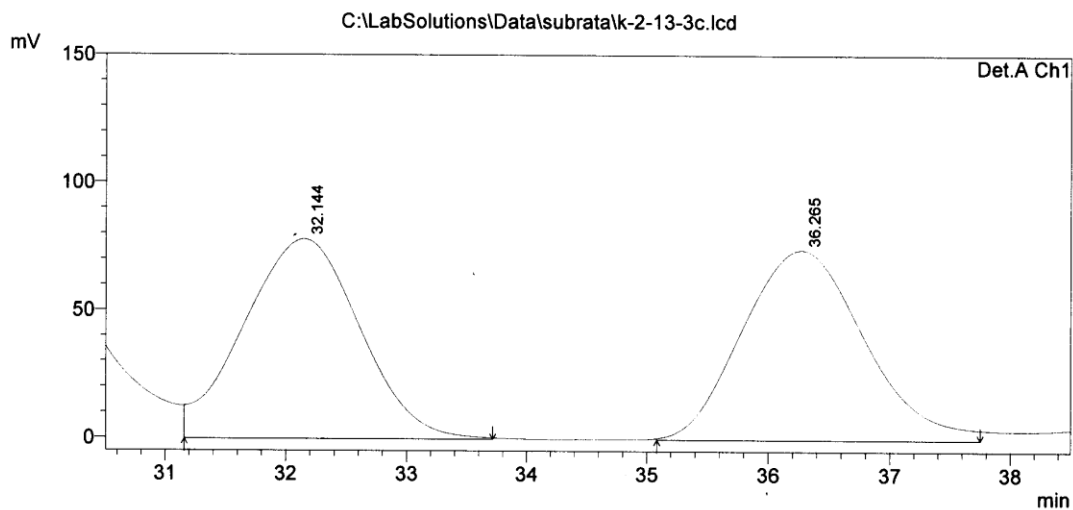


<Chromatogram>

PeakTable

Detector A Ch1 220nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	32.144	5189746	78038	50.113	51.279
2	36.265	5166438	74146	49.887	48.721
Total		10356184	152184	100.000	100.000

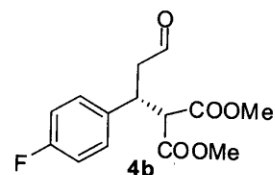


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==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\subrata\sg-215a.lcd

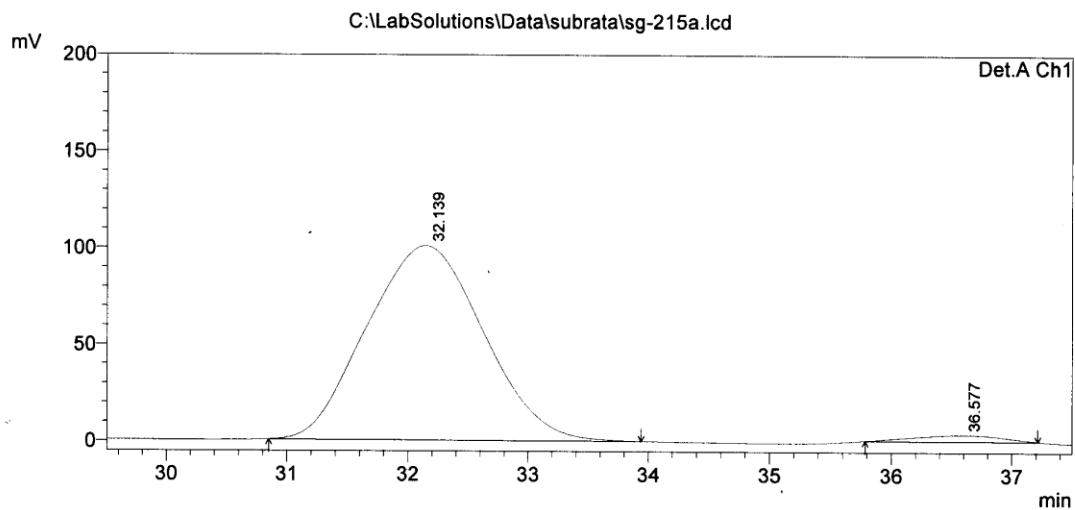
Acquired by : Admin
Sample Name : sg-215a
Sample ID : sg-215a
Vial # :
Injection Volume : 5 uL
Data File Name : sg-215a.lcd
Method File Name : 70%-Hexane-30%-isopropanol-30min (1.0mL, 220nm).lcm
Batch File Name :
Report File Name : Default.lcr
Data Acquired : 1/31/2010 4:22:20 PM
Data Processed : 1/31/2010 5:17:37 PM



<Chromatogram>

PeakTable

Peak#	Ret. Time	Area	Height	Area %	Height %
1	32.139	6750767	100576	97.561	96.781
2	36.577	168739	3345	2.439	3.219
Total		6919507	103921	100.000	100.000

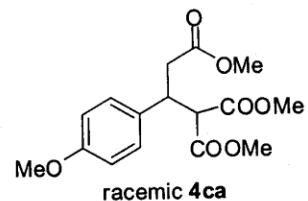


6/28/2012 16:33:14 1 /

==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\subrata\k-2-12.lcd

Acquired by : Admin
Sample Name : k-2-12
Sample ID : k-2-12
Vail # :
Injection Volume : 5 uL
Data File Name : k-2-12.lcd
Method File Name : 80%-Hexane-20%-isopropanol-35min (0.5 mL, 254nm).lcm
Batch File Name :
Report File Name : Default.lcr
Data Acquired : 1/20/2010 9:04:13 PM
Data Processed : 6/9/2010 5:45:31 PM

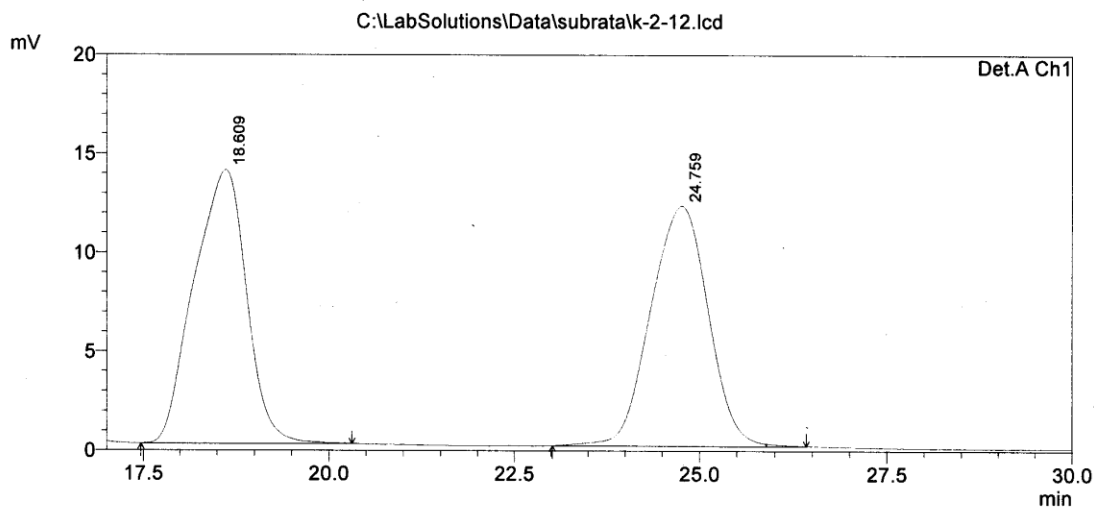


<Chromatogram>

PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	18.609	679119	13826	51.006	53.289
2	24.759	652329	12119	48.994	46.711
Total		1331448	25946	100.000	100.000

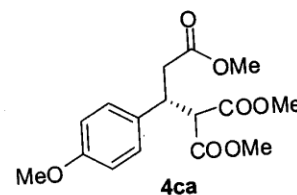


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==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\subrata\sg-221.lcd

Acquired by : Admin
Sample Name : sg-221
Sample ID : sg-221
Vial # :
Injection Volume : 5 uL
Data File Name : sg-221.lcd
Method File Name : 80%-Hexane-20%-isopropanol-35min (0.5 mL, 254nm).lcm
Batch File Name :
Report File Name : Default.lcr
Data Acquired : 1/20/2010 8:06:50 PM
Data Processed : 4/24/2010 4:34:57 PM

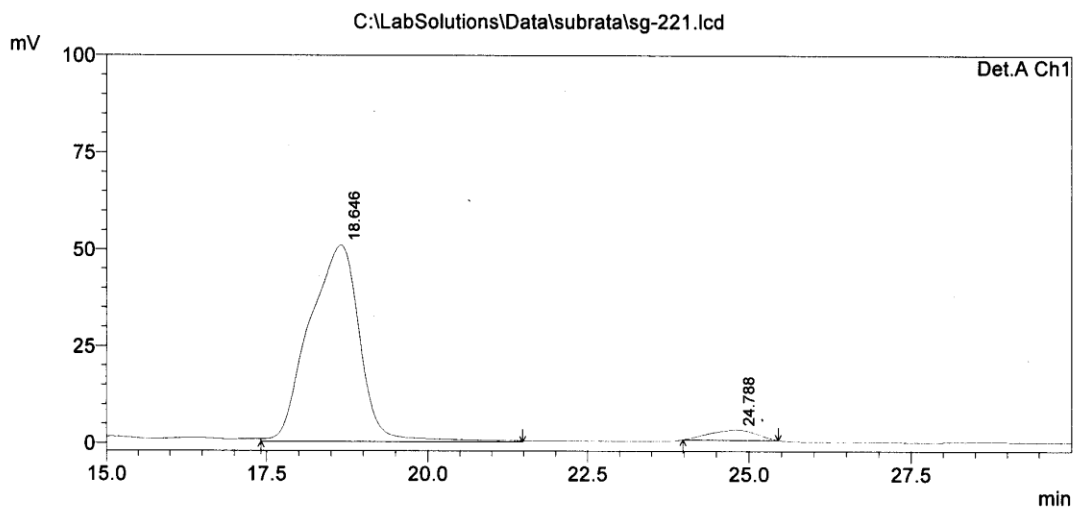


<Chromatogram>

Detector A Ch1 254nm

PeakTable

Peak#	Ret. Time	Area	Height	Area %	Height %
1	18.646	2750045	50757	95.473	95.150
2	24.788	130391	2587	4.527	4.850
Total		2880436	53344	100.000	100.000

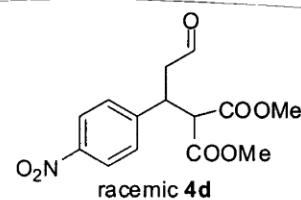


6/28/2012 16:32:17 1 / 2

==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\subrata\k-1-91.lcd

Acquired by : Admin
Sample Name : k-1-91
Sample ID : k-1-91
Vail # :
Injection Volume : 5 uL
Data File Name : k-1-91.lcd
Method File Name : hexane-isopropanol(70-30)-30min (254nm, 0.7 mL).lcm
Batch File Name :
Report File Name : Default.lcr
Data Acquired : 11/17/2009 9:33:12 PM
Data Processed : 6/10/2010 4:25:36 PM

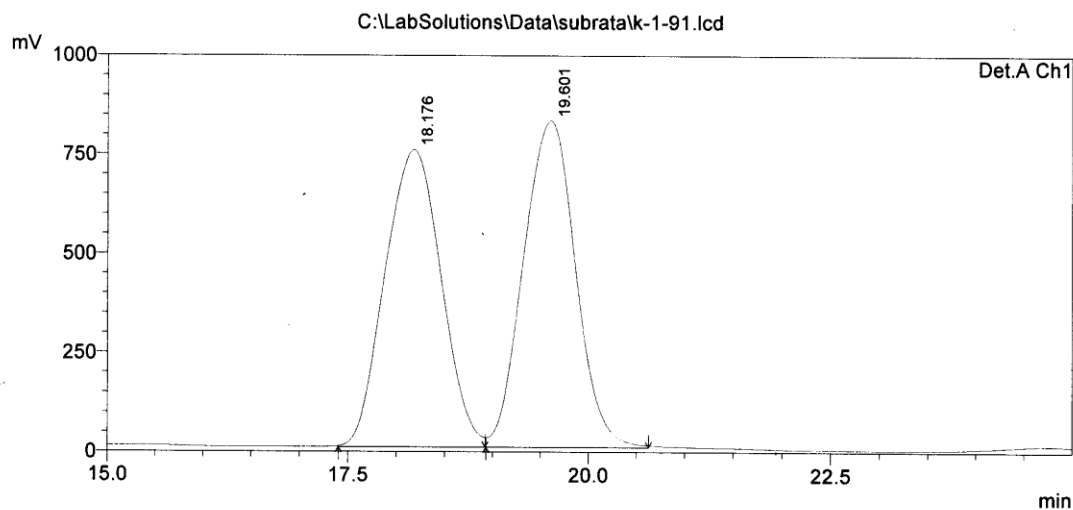


<Chromatogram>

PeakTable

Detector A Ch1 254nm

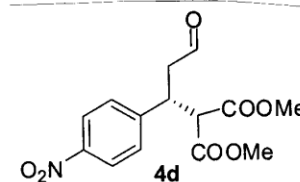
Peak#	Ret. Time	Area	Height	Area %	Height %
1	18.176	30371976	749794	49.400	47.645
2	19.601	31109325	823925	50.600	52.355
Total		61481301	1573719	100.000	100.000



==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\subrata\k-1-77.lcd

Acquired by : Admin
Sample Name : k-1-77
Sample ID : k-1-77
Vial # :
Injection Volume : 5 uL
Data File Name : k-1-77.lcd
Method File Name : hexane-isopropanol(70-30)-30min (254nm, 0.7 mL).lcm
Batch File Name :
Report File Name : Default.lcr
Data Acquired : 9/19/2009 3:00:45 AM
Data Processed : 4/24/2010 5:28:15 PM

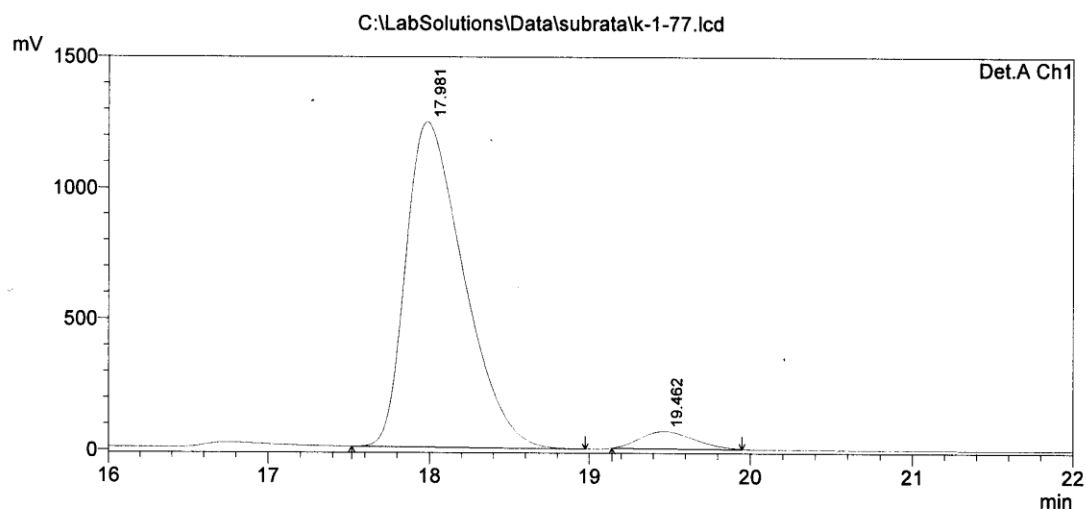


<Chromatogram>

PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	17.981	31109016	1242069	95.418	95.024
2	19.462	1493921	65037	4.582	4.976
Total		32602937	1307106	100.000	100.000

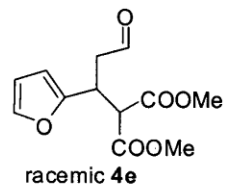


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==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\subrata\k-2-9.lcd

Acquired by : Admin
Sample Name : k-2-9
Sample ID : k-2-9
Vail # :
Injection Volume : 5 uL
Data File Name : k-2-9.lcd
Method File Name : 70%-Hexane-30%-isopropanol-35min (0.7mL, 254nm).lcm
Batch File Name :
Report File Name : Default.lcr
Data Acquired : 1/11/2010 9:39:01 PM
Data Processed : 4/5/2010 4:01:35 PM

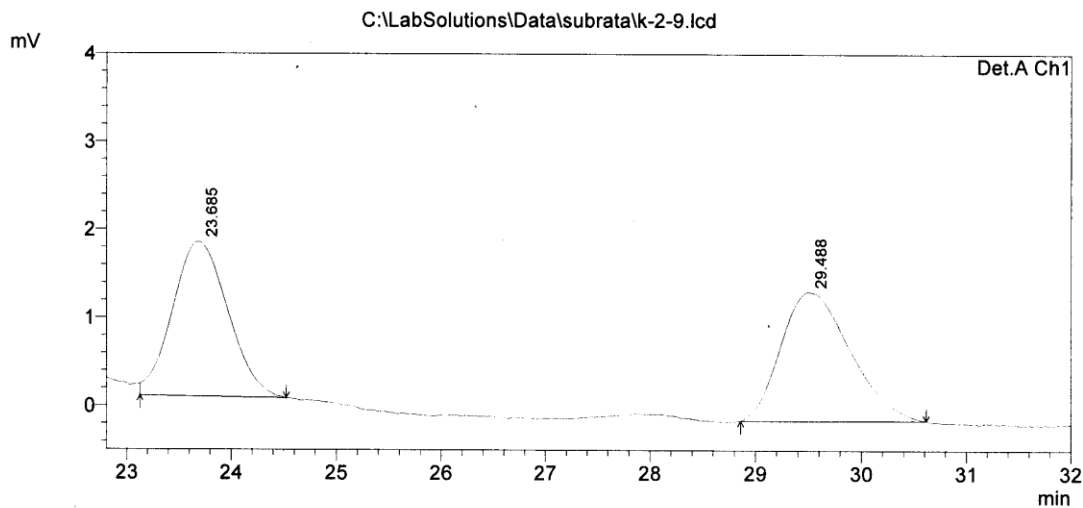


<Chromatogram>

Detector A Ch1 254nm

PeakTable

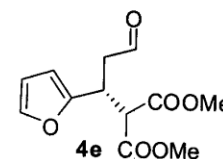
Peak#	Ret. Time	Area	Height	Area %	Height %
1	23.685	64548	1755	49.191	54.538
2	29.488	66672	1463	50.809	45.462
Total		131220	3217	100.000	100.000



==== Shimadzu LCsolution Analysis Report ====

Acquired by : Admin
Sample Name : sg-214a
Sample ID : sg-214a
Vial # :
Injection Volume : 5 uL
Data File Name : sg-214a.lcd
Method File Name : 70%-Hexane-30%-isopropanol-35min (0.7mL, 254nm).lcm
Batch File Name :
Report File Name : Default.lcr
Data Acquired : 1/11/2010 10:15:33 PM
Data Processed : 4/24/2010 4:33:59 PM

C:\LabSolutions\Data\subrata\sg-214a.lcd

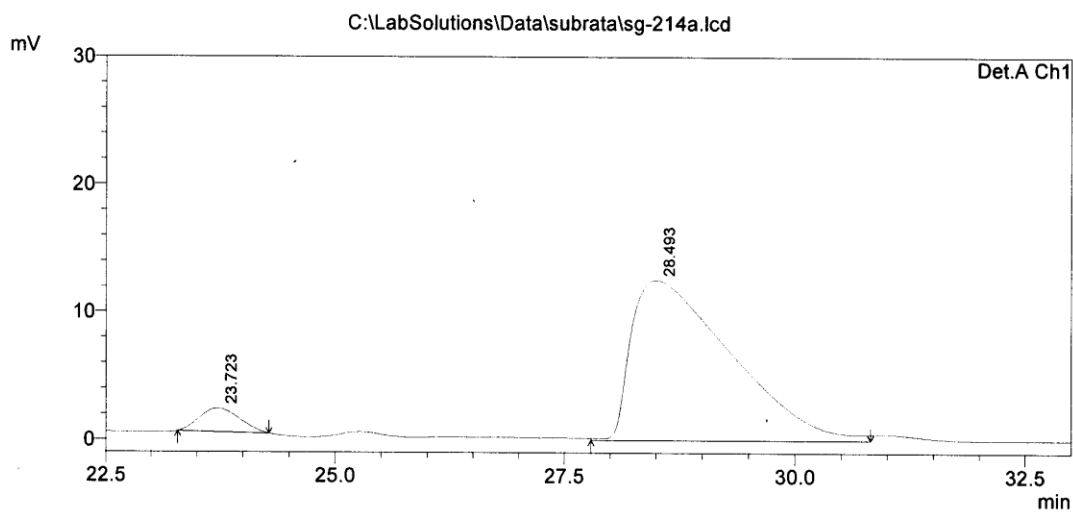


<Chromatogram>

Detector A Ch1 254nm

PeakTable

Peak#	Ret. Time	Area	Height	Area %	Height %
1	23.723	55264	1840	5.573	12.822
2	28.493	936326	12510	94.427	87.178
Total		991590	14350	100.000	100.000

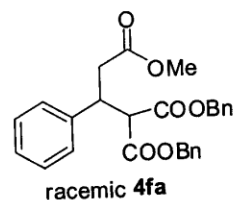


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==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\subrata\k-2-10E.lcd

Acquired by : Admin
Sample Name : k-2-10E
Sample ID : k-2-10E
Vial # :
Injection Volume : 5 uL
Data File Name : k-2-10E.lcd
Method File Name : 80%-Hexane-20%-isopropanol-30min (1.0 mL, 254nm).lcm
Batch File Name :
Report File Name : Default.lcr
Data Acquired : 1/21/2010 11:22:55 AM
Data Processed : 4/5/2010 4:03:12 PM

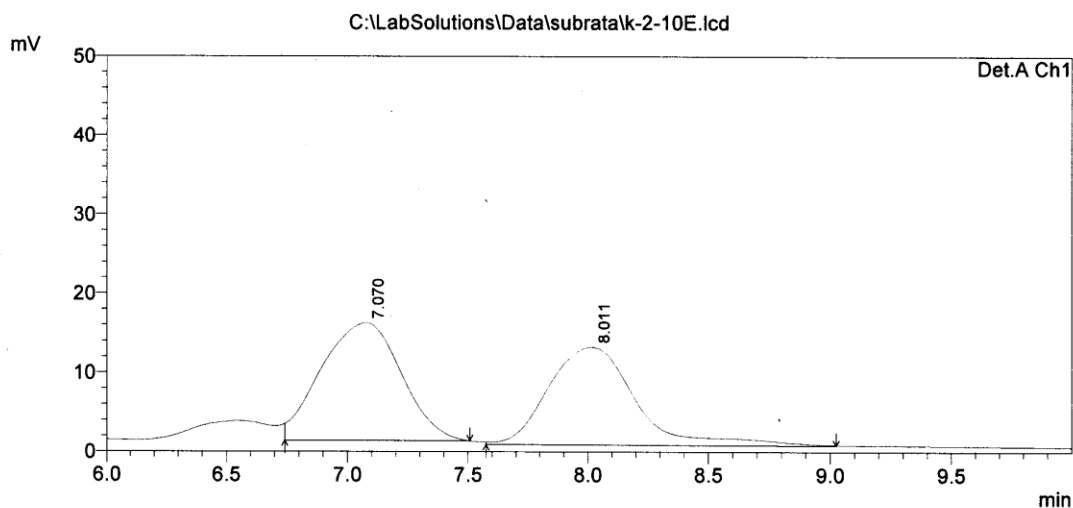


<Chromatogram>

PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	7.070	340435	14821	52.281	54.648
2	8.011	310733	12300	47.719	45.352
Total		651167	27122	100.000	100.000

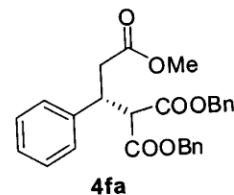


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==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\subrata\sg-227E.lcd

Acquired by : Admin
Sample Name : sg-227E
Sample ID : sg-227E
Vial # :
Injection Volume : 5 uL
Data File Name : sg-227E.lcd
Method File Name : 80%-Hexane-20%-isopropanol-30min (1.0 mL, 254nm).lcm
Batch File Name :
Report File Name : Default.lcr
Data Acquired : 1/21/2010 1:24:16 PM
Data Processed : 4/24/2010 4:48:07 PM

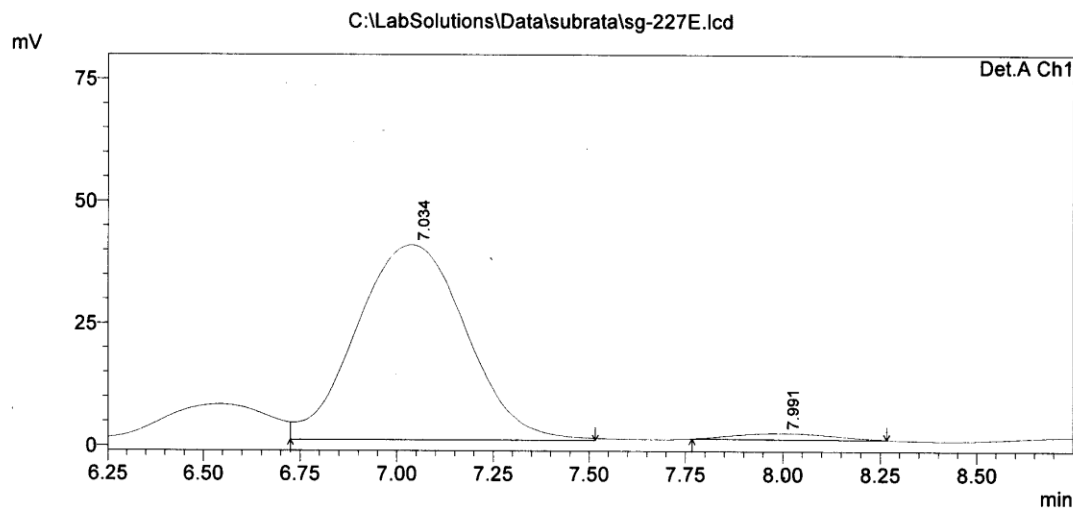


<Chromatogram>

PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	7.034	784186	39828	97.308	96.918
2	7.991	21692	1266	2.692	3.082
Total		805878	41095	100.000	100.000

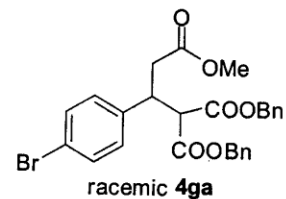


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==== Shimadzu LCsolution Analysis Report ====

Acquired by : Admin
Sample Name : k-2-14
Sample ID : k-2-14
Vail # :
Injection Volume : 5 uL
Data File Name : k-2-14.lcd
Method File Name : 80%-Hexane-20%-isopropanol-60min (0.7 mL, 254nm).lcm
Batch File Name :
Report File Name : Default.lcr
Data Acquired : 1/10/2010 7:59:21 PM
Data Processed : 6/9/2010 5:46:24 PM

C:\LabSolutions\Data\subrata\k-2-14.lcd

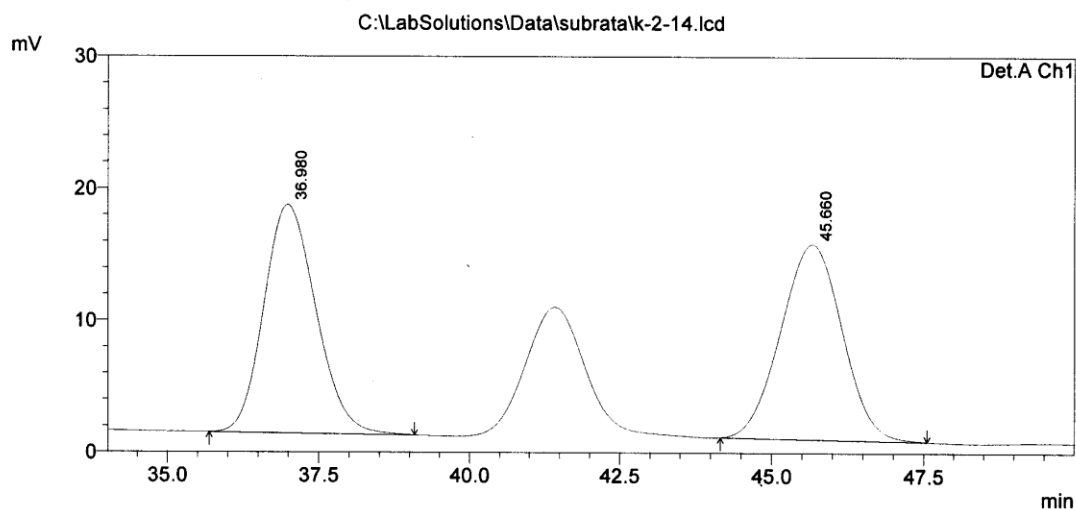


<Chromatogram>

PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	36.980	1033883	17315	49.915	53.934
2	45.660	1037412	14789	50.085	46.066
Total		2071295	32104	100.000	100.000

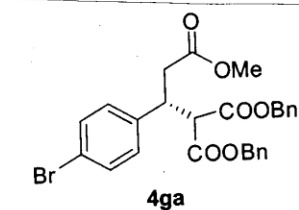


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==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\subrata\sg-216b.lcd

Acquired by : Admin
Sample Name : sg-216b
Sample ID : sg-216b
Vial # :
Injection Volume : 5 uL
Data File Name : sg-216b.lcd
Method File Name : 80%-Hexane-20%-isopropanol-60min (0.7 mL, 254nm).lcm
Batch File Name :
Report File Name : Default.lcr
Data Acquired : 1/10/2010 9:01:21 PM
Data Processed : 1/12/2010 8:03:39 PM

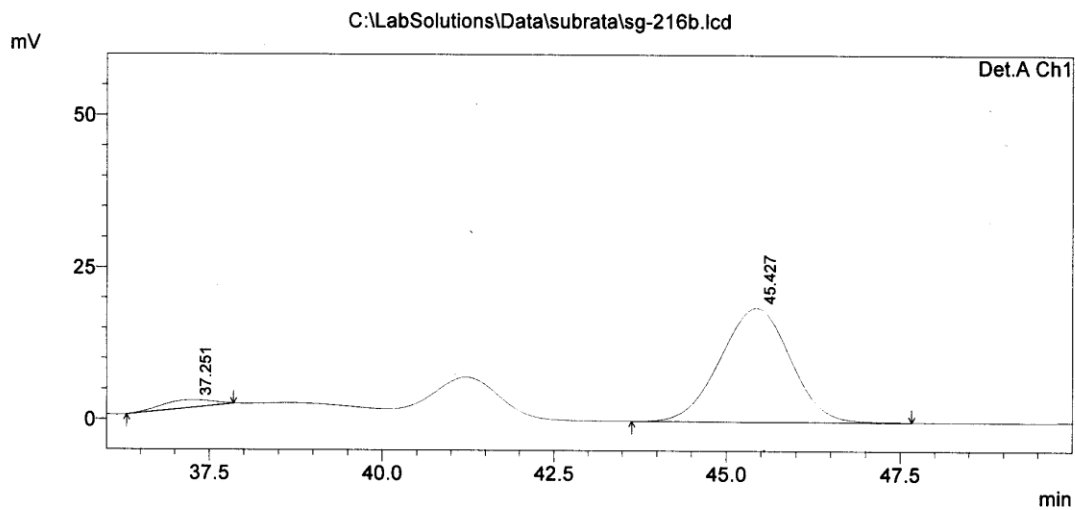


<Chromatogram>

Detector A Ch1 254nm

PeakTable

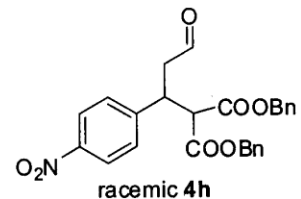
Peak#	Ret. Time	Area	Height	Area %	Height %
1	37.251	63050	1212	4.584	6.087
2	45.427	1312518	18700	95.416	93.913
Total		1375568	19912	100.000	100.000



==== Shimadzu LCsolution Analysis Report ====

Acquired by : Admin
Sample Name : k-2-22
Sample ID : k-2-22
Vial # :
Injection Volume : 5 uL
Data File Name : k-2-22.lcd
Method File Name : 70%-Hexane-30%-isopropanol-120min (1.0mL, 220nm).lcm
Batch File Name :
Report File Name : Default.lcr
Data Acquired : 2/10/2010 4:29:36 PM
Data Processed : 4/5/2010 4:17:39 PM

C:\LabSolutions\Data\subrata\k-2-22.lcd

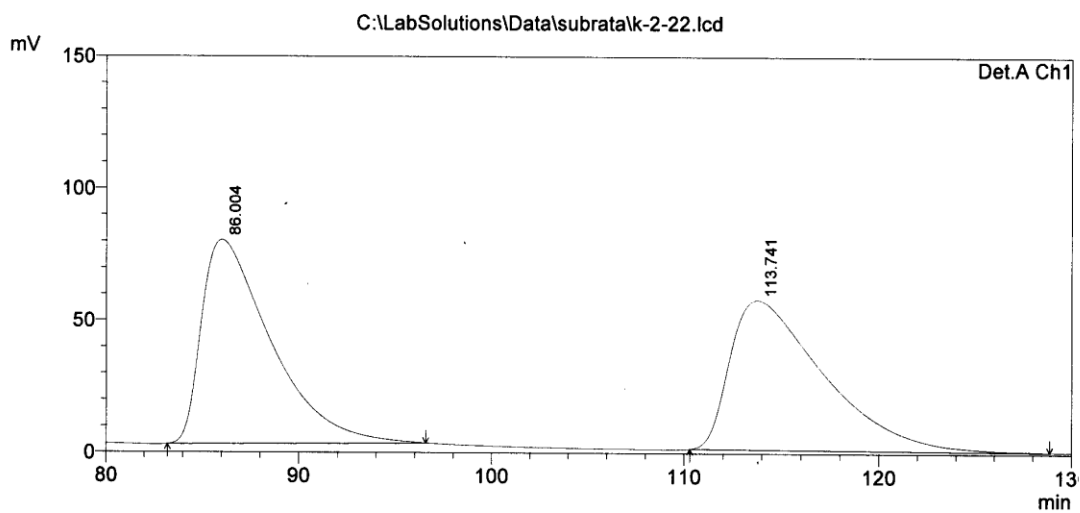


<Chromatogram>

PeakTable

Detector A Ch1 220nm

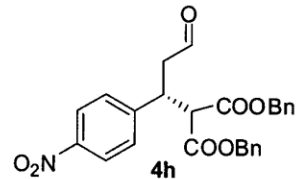
Peak#	Ret. Time	Area	Height	Area %	Height %
1	86.004	19214343	77300	50.374	57.740
2	113.741	18928807	56576	49.626	42.260
Total		38143150	133876	100.000	100.000



==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\subrata\sg-240-1.lcd

Acquired by : Admin
Sample Name : sg-240-1
Sample ID : sg-240-1
Vial # :
Injection Volume : 5 uL
Data File Name : sg-240-1.lcd
Method File Name : 70%-Hexane-30%-isopropanol-120min (1.0mL, 220nm).lcm
Batch File Name :
Report File Name : Default.lcr
Data Acquired : 2/3/2010 9:25:12 PM
Data Processed : 2/4/2010 8:26:08 PM

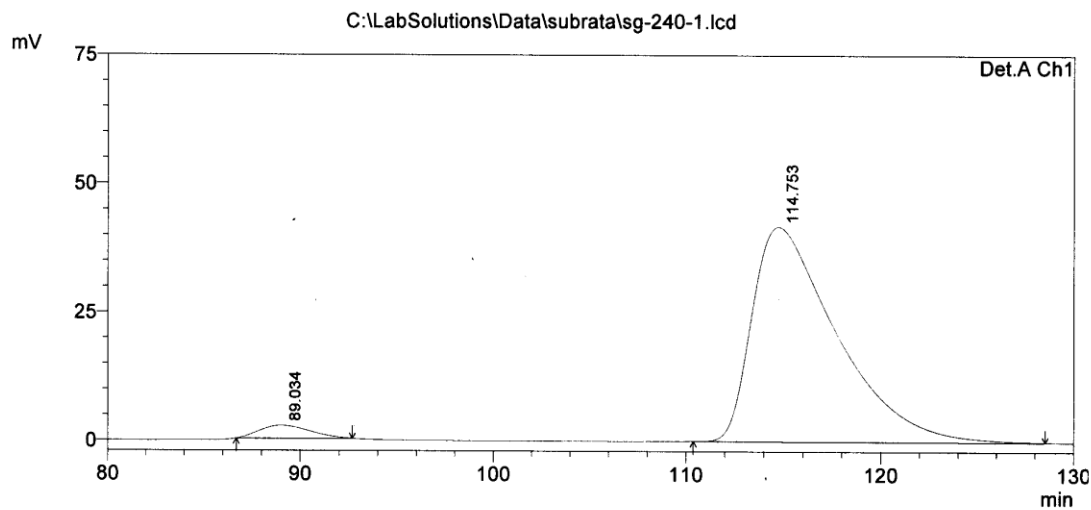


<Chromatogram>

PeakTable

Detector A Ch1 220nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	89.034	449563	2507	3.444	5.665
2	114.753	12604779	41747	96.556	94.335
Total		13054342	44253	100.000	100.000

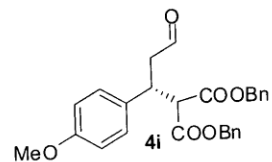


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==== Shimadzu LCsolution Analysis Report ====

Acquired by : Admin
Sample Name : sg-235a
Sample ID : sg-235a
Vial # :
Injection Volume : 5 uL
Data File Name : sg-235a.lcd
Method File Name : 70%-Hexane-30%-isopropanol-70min (1.0mL, 220nm).lcm
Batch File Name :
Report File Name : Default.lcr
Data Acquired : 2/1/2010 10:42:22 PM
Data Processed : 4/24/2010 4:53:08 PM

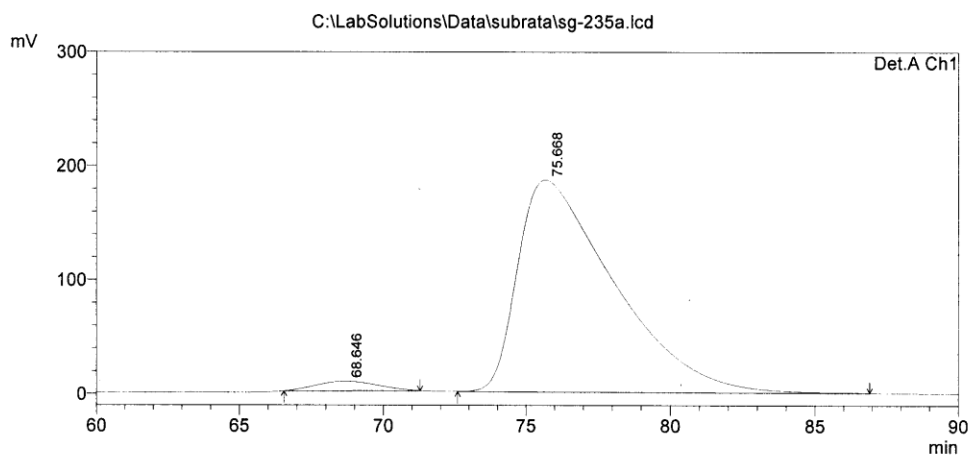
C:\LabSolutions\Data\subrata\sg-235a.lcd



<Chromatogram>

PeakTable

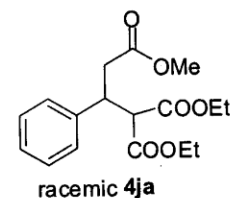
Peak#	Ret. Time	Area	Height	Area %	Height %
1	68.646	1218553	8307	2.737	4.275
2	75.668	43306329	186002	97.263	95.725
Total		44524882	194309	100.000	100.000



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==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\subrata\k-2-20a.lcd
Acquired by : Admin
Sample Name : k-2-20a
Sample ID : k-2-20a
Vail # :
Injection Volume : 5 uL
Data File Name : k-2-20a.lcd
Method File Name : 70%-Hexane-30%-isopropanol-45min (0.5mL, 220nm).lcm
Batch File Name :
Report File Name : Default.lcr
Data Acquired : 2/9/2010 2:57:46 PM
Data Processed : 4/5/2010 4:14:09 PM

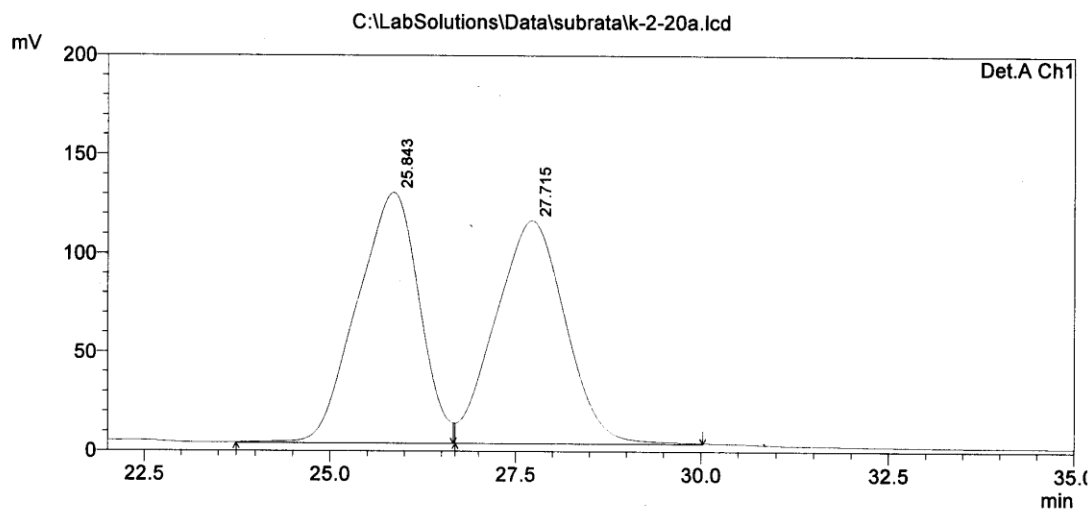


<Chromatogram>

Detector A Ch1 220nm

PeakTable

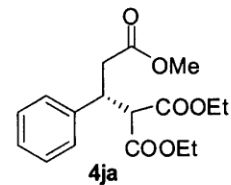
Peak#	Ret. Time	Area	Height	Area %	Height %
1	25.843	7332105	126815	49.805	52.917
2	27.715	7389465	112836	50.195	47.083
Total		14721570	239651	100.000	100.000



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==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\subrata\sg-234a.lcd
Acquired by : Admin
Sample Name : sg-234a
Sample ID : sg-234a
Vial # :
Injection Volume : 5 uL
Data File Name : sg-234a.lcd
Method File Name : 70%-Hexane-30%-isopropanol-45min (0.5mL, 220nm).lcm
Batch File Name :
Report File Name : Default.lcr
Data Acquired : 2/2/2010 11:11:54 AM
Data Processed : 2/9/2010 2:56:05 PM

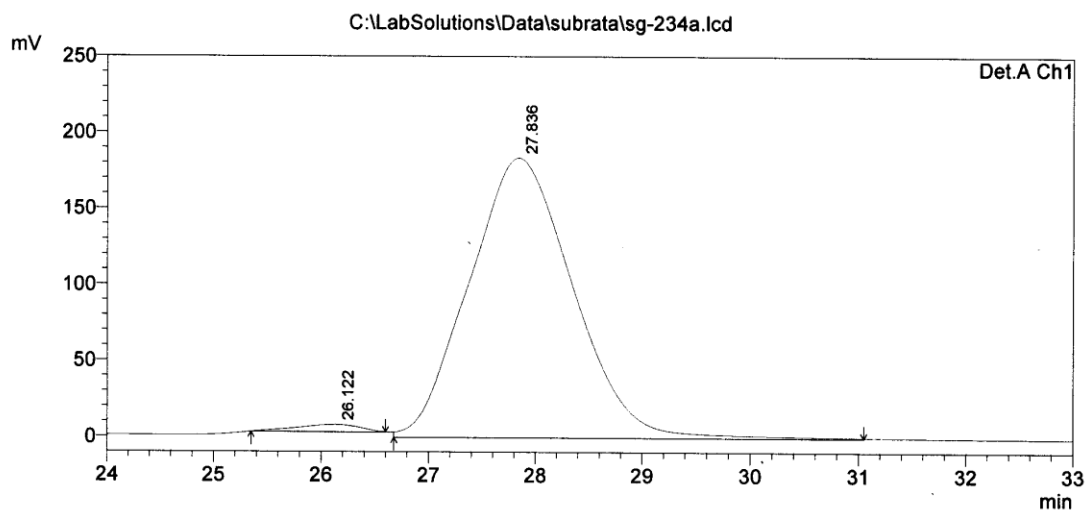


<Chromatogram>

PeakTable

Detector A Ch1 220nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	26.122	191730	4768	1.518	2.529
2	27.836	12441608	183763	98.482	97.471
Total		12633338	188531	100.000	100.000

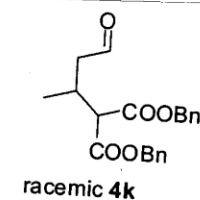


6/28/2012 16:39:25 1 / 2

==== Shimadzu LCsolution Analysis Report ====

Acquired by : Admin
Sample Name : k-2-15a
Sample ID : k-2-15a
Vail # :
Injection Volume : 5 uL
Data File Name : k-2-15a.lcd
Method File Name : 95%-Hexane-5%-isopropanol-50min (0.7mL, 254nm).lcm
Batch File Name :
Report File Name : Default.lcr
Data Acquired : 2/4/2010 8:45:42 PM
Data Processed : 4/5/2010 4:10:06 PM

C:\LabSolutions\Data\subrata\k-2-15a.lcd

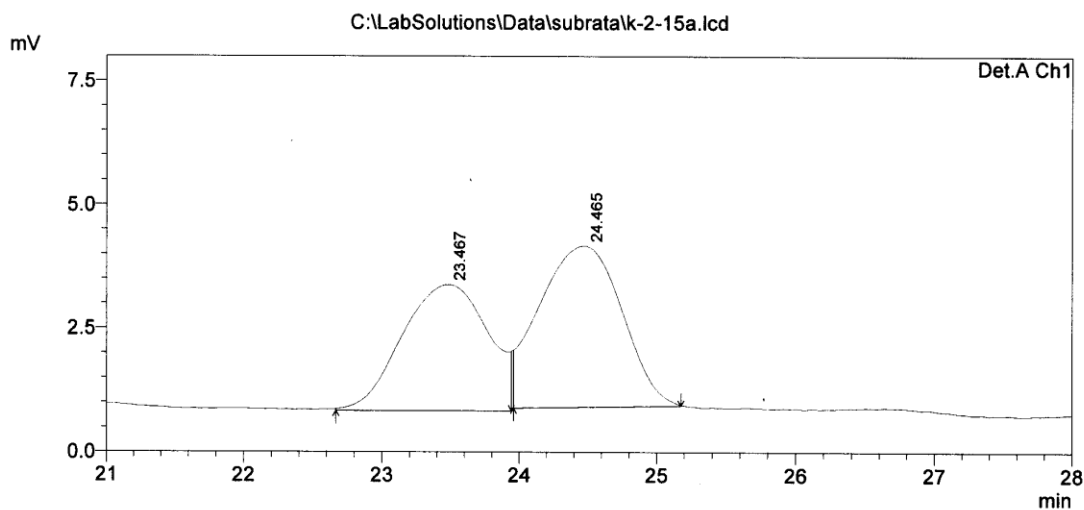


<Chromatogram>

PeakTable

Detector A Ch1 254nm

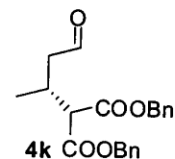
Peak#	Ret. Time	Area	Height	Area %	Height %
1	23.467	110264	2539	44.774	43.836
2	24.465	136005	3253	55.226	56.164
Total		246268	5791	100.000	100.000



==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\subrata\sg-239.lcd

Acquired by : Admin
Sample Name : sg-239
Sample ID : sg-239
Vial # :
Injection Volume : 5 uL
Data File Name : sg-239.lcd
Method File Name : 95%-Hexane-5%-isopropanol-50min (0.7mL, 254nm).lcm
Batch File Name :
Report File Name : Default.lcr
Data Acquired : 2/4/2010 7:00:03 PM
Data Processed : 4/24/2010 5:30:12 PM



<Chromatogram>

PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	23.703	326960	10200	7.354	8.898
2	24.733	4119148	104431	92.646	91.102
Total		4446108	114630	100.000	100.000

