

Supporting Information

Phase-vanishing halolactonization of neat substrates

Nicole Windmon¹ and Veljko Dragojlovic^{2,*}

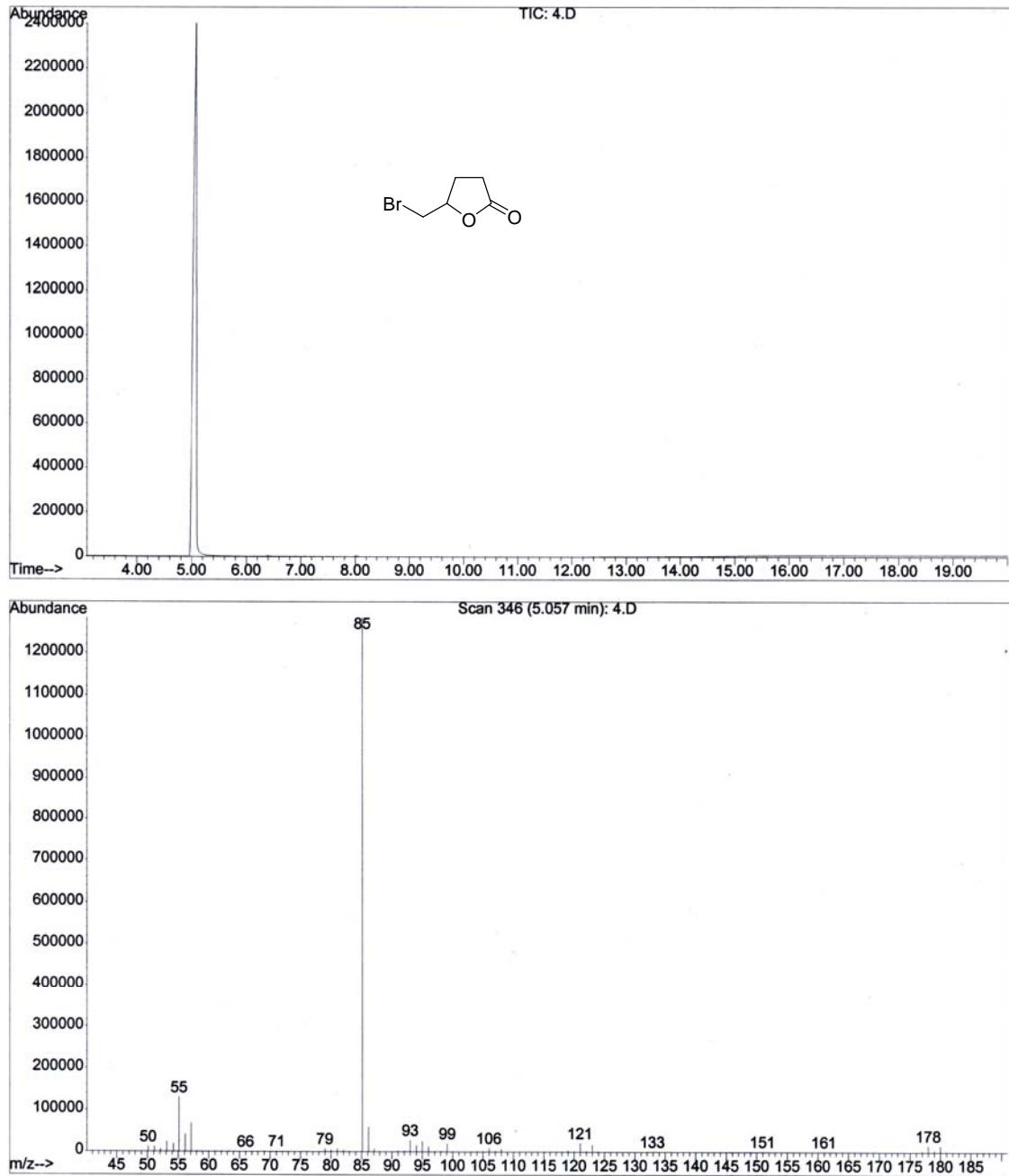
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GC-MS spectra of the compounds **2**, **3**, **4**, **5**, **8**, **13**, **18**, **19**, **21**, **22** and ^1H NMR spectra of the compounds **2**, **4**, **5**, **8**, **13**, **18**, **19**, **21**, **22**.

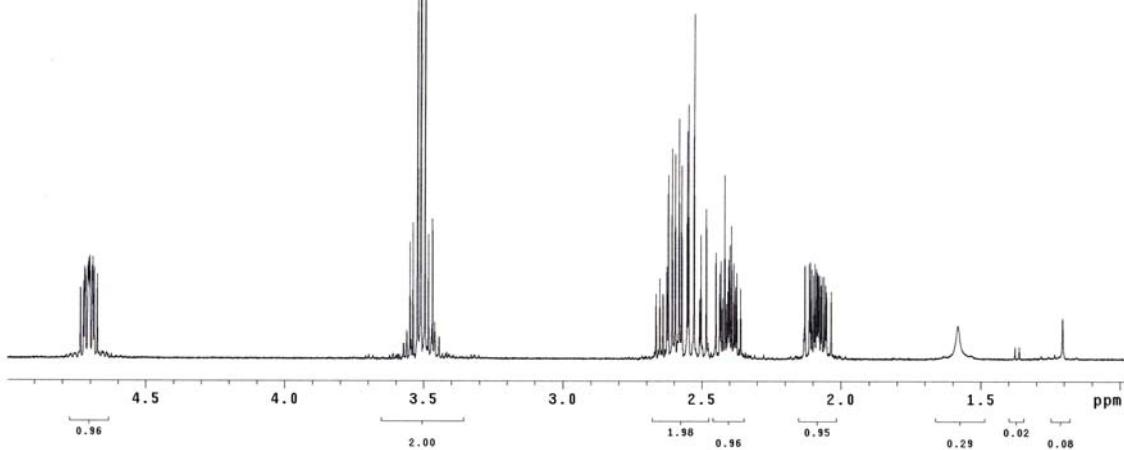
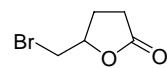


GC-MS of 5-(bromomethyl)-dihydrofuran-2(3H)-one (**2**).

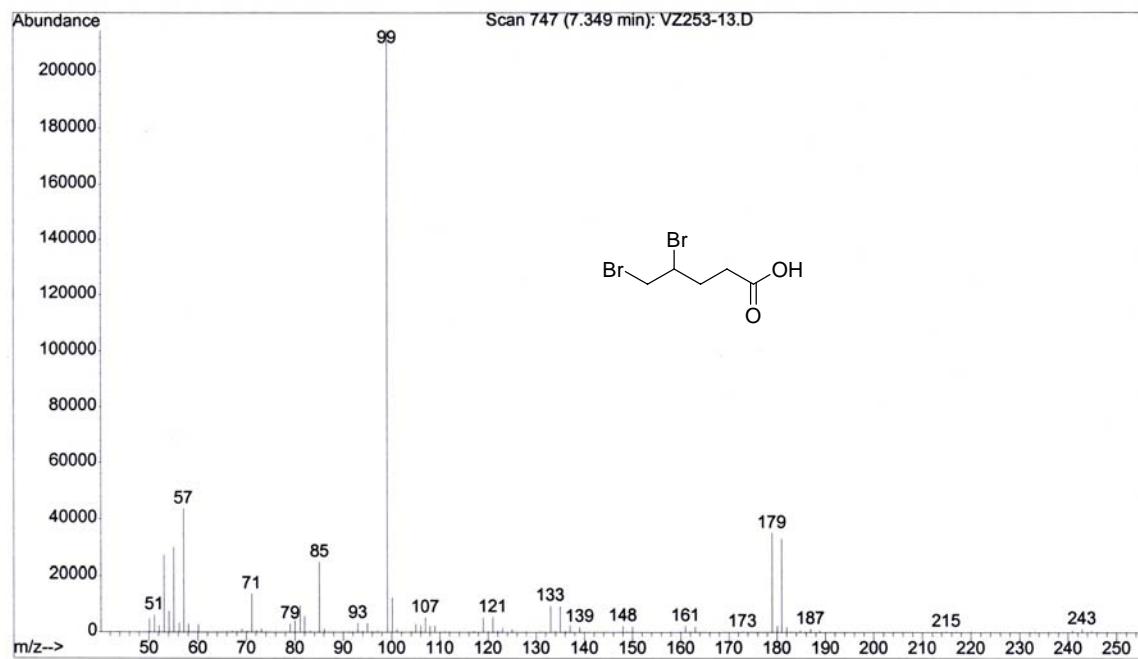
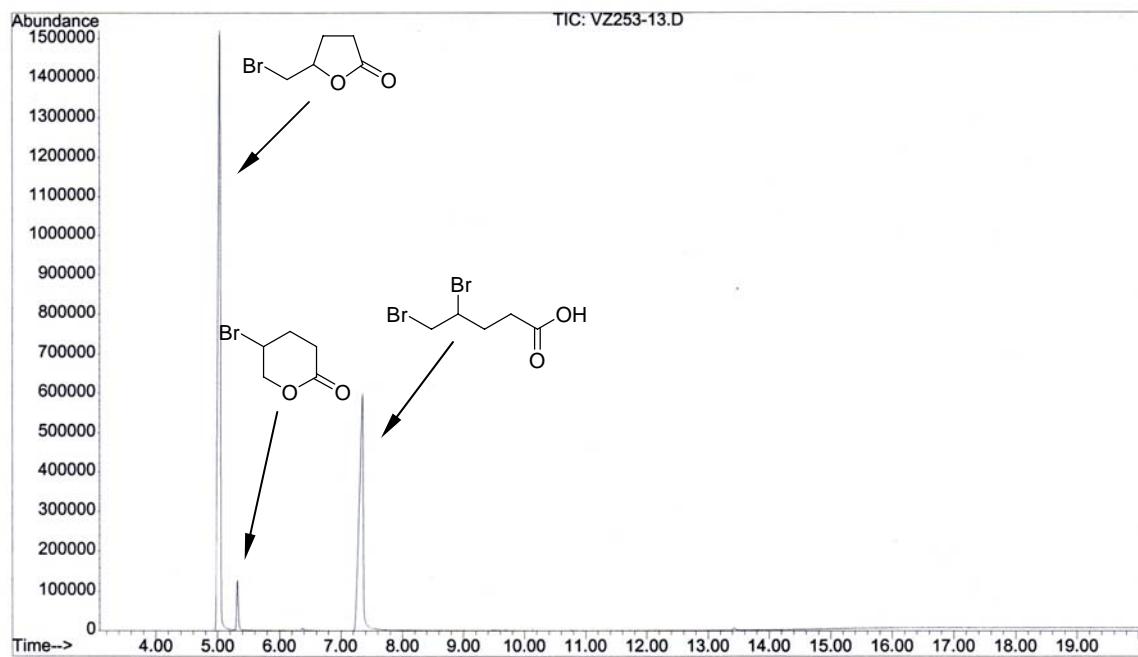
05_06_08_E
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Sample directory: trans-4-L-0_16Jan2004
File: PROTON

Pulse Sequence: s2pul
Solvent: CDCl₃
Ambient temperature
Mercury-400BB "Indian"

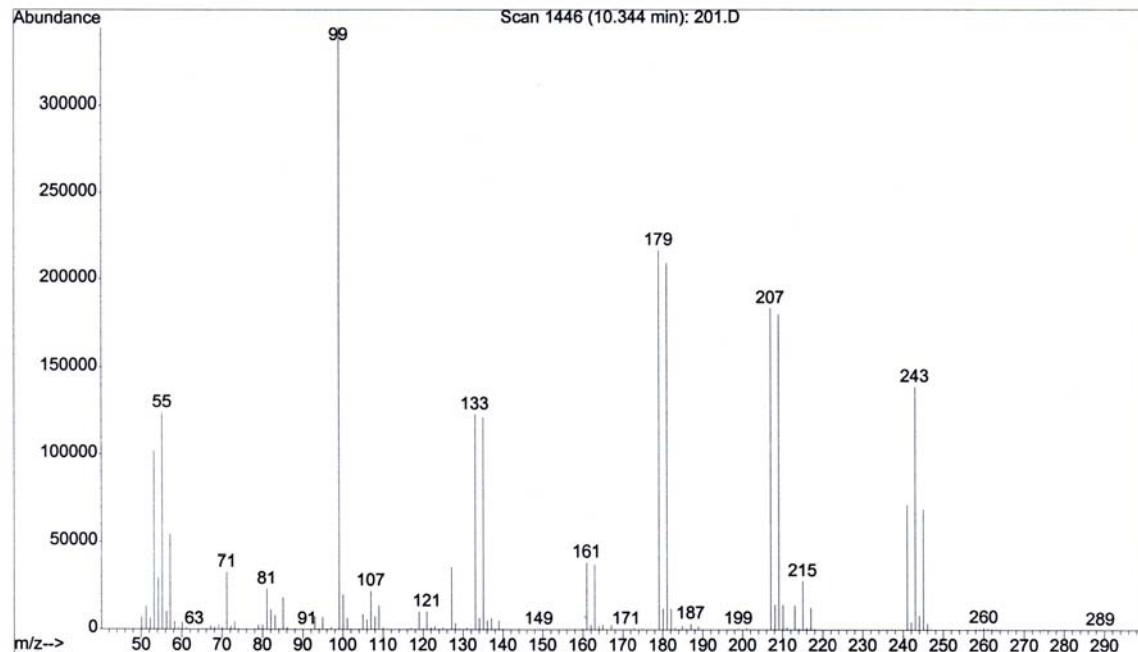
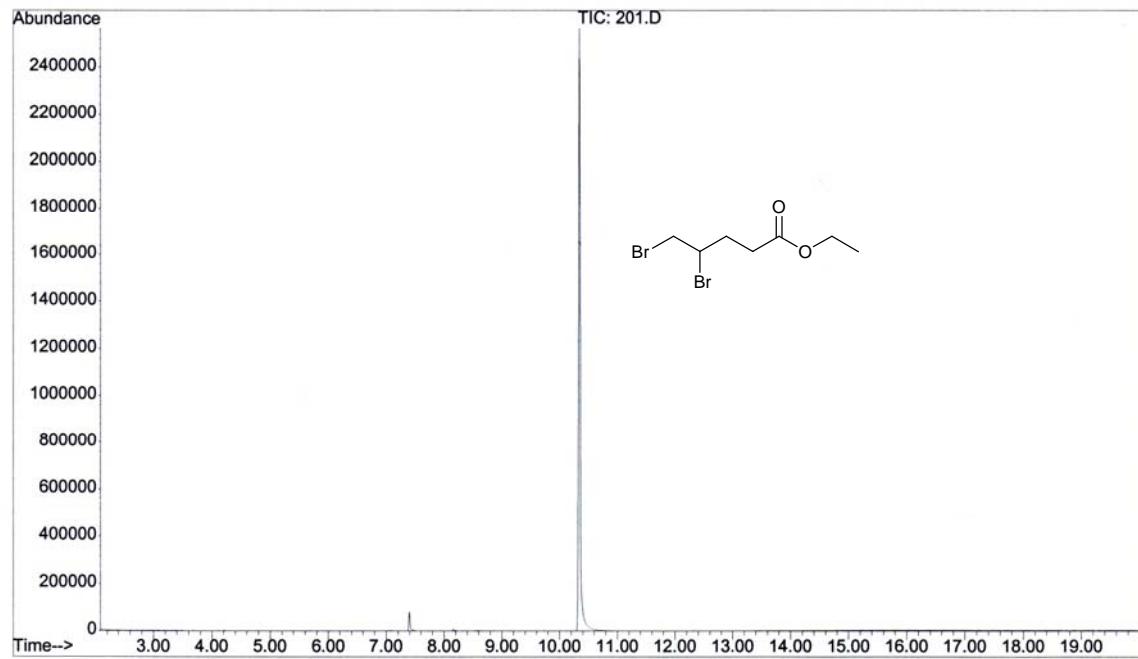
Relax. delay 16.000 sec
Pulse 51.8 degrees
Acq. time 8.000 sec
Width 6410.3 Hz
4 Averaging
OBSERVE H1, 400.6260530 MHz
DATA PROCESSING
FT size 131072
Total time 2 min, 42 sec



¹H NMR of 5-(bromomethyl)-dihydrofuran-2(3H)-one (2).



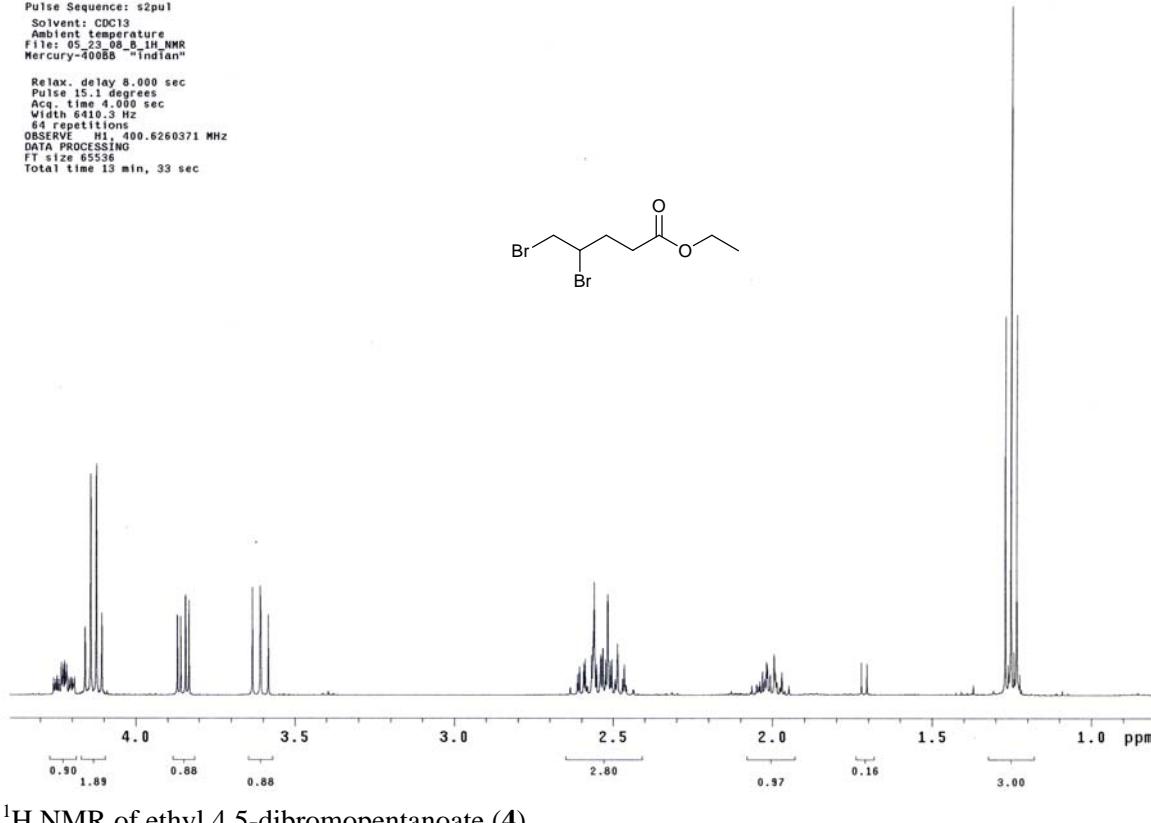
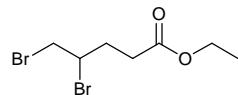
GC-MS of solvent-free reaction of bromine with 4-pentenoic acid. The reaction mixture contains 4,5-dibromopentanoic acid (**3**).



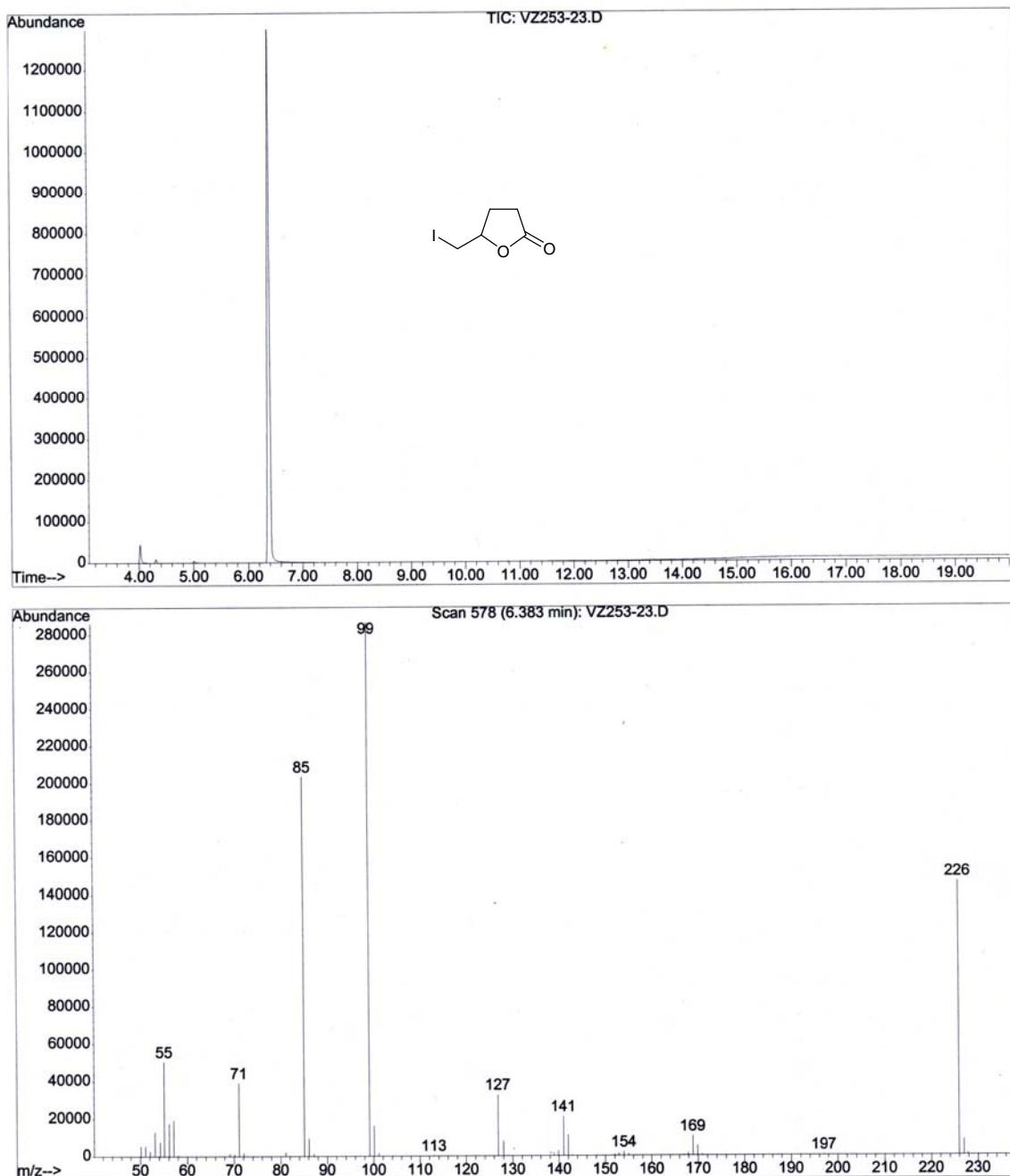
GC-MS of ethyl 4,5-dibromopentanoate (**4**).

05_23_08_B_1H_NMR
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Pulse Sequence: s2pul
Solvent: CDCl₃
Ambient temperature
File: 05_23_08_B_1H_NMR
Mercury-400BB "indian"

Relax, delay 8.000 sec
Pulse 15.1 degrees
Acq. time 4.000 sec
Width 6410.3 Hz
448 repetitions
OBSERVE H-1, 400.6260371 MHz
DATA PROCESSING
FT size 65536
Total time 13 min, 33 sec

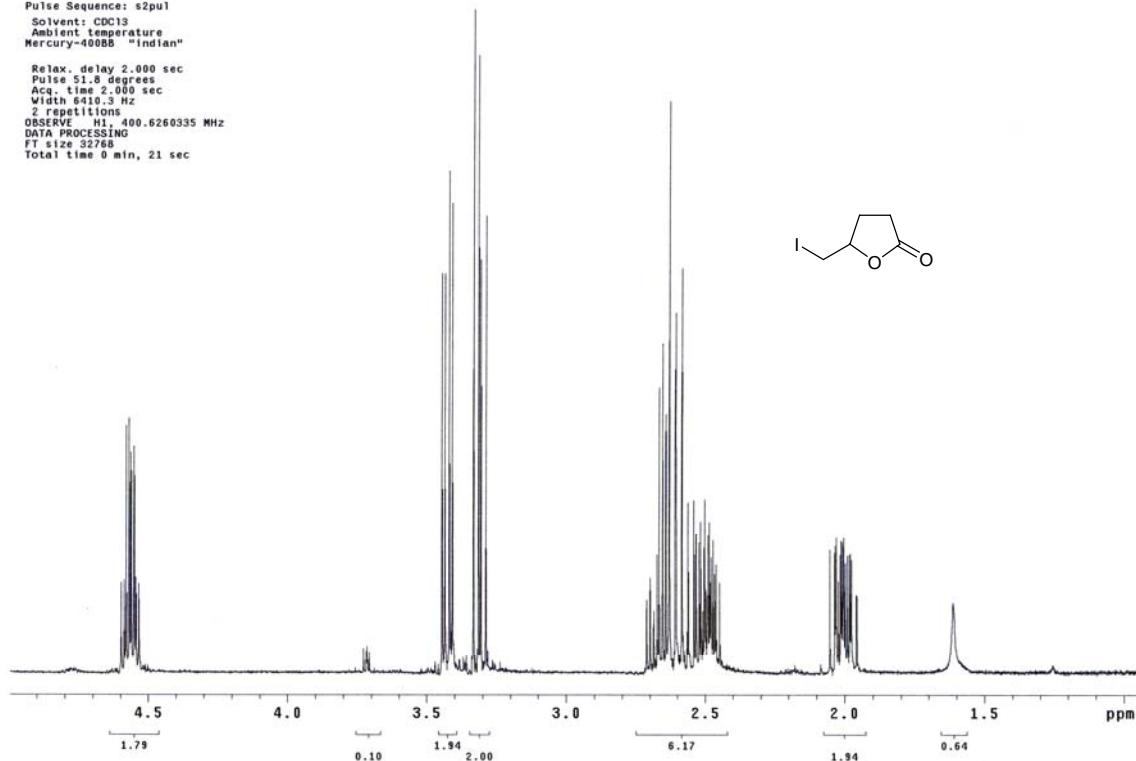


¹H NMR of ethyl 4,5-dibromopentanoate (**4**).

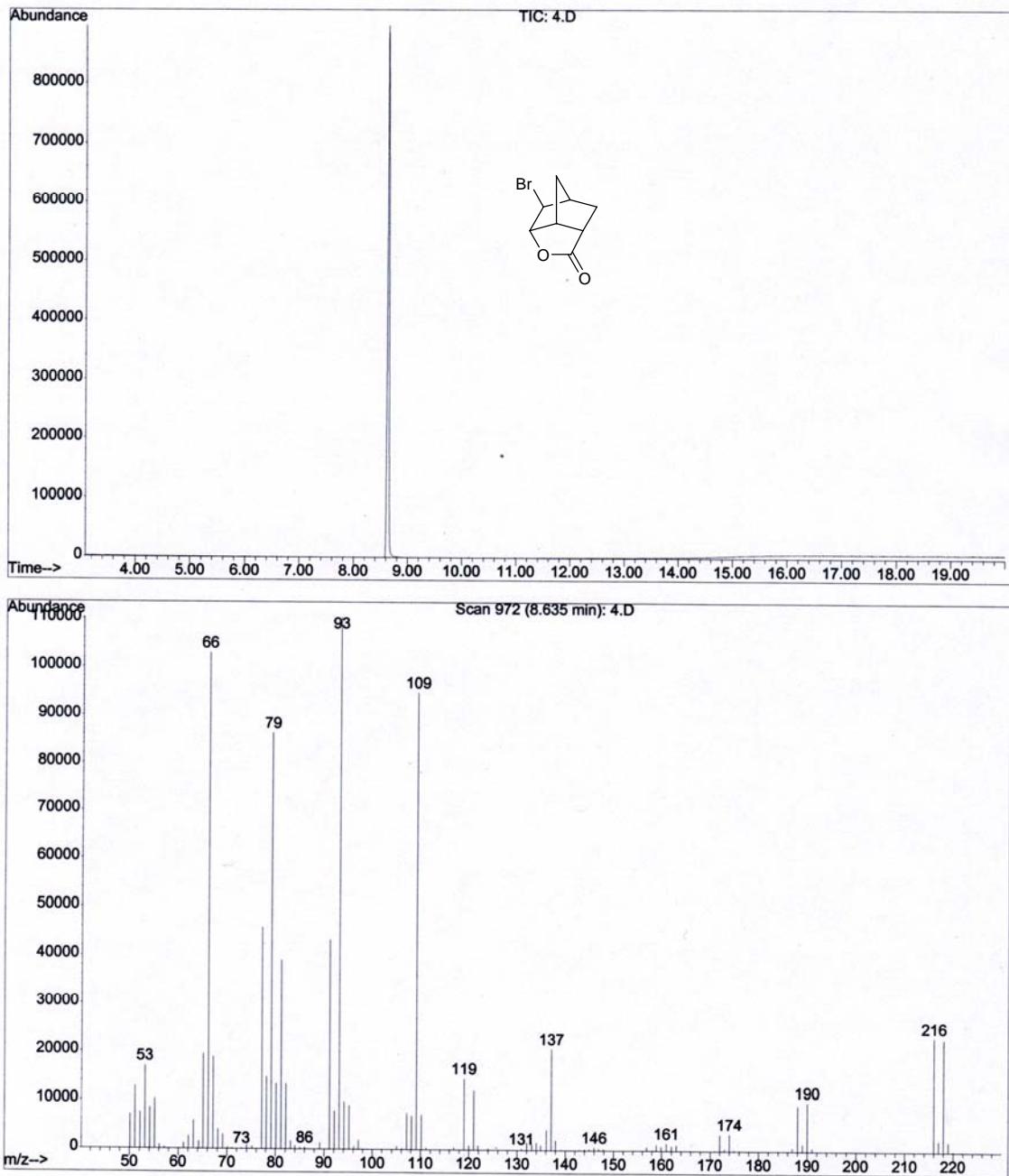


GC-MS of 5-(iodomethyl)-dihydrofuran-2(3H)-one (**5**).

05_06_08_C
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Sample directory: trans-4-L-0_16Jan2004
File: PROTON
Pulse Sequence: s2pul
Solvent: CDCl₃
Ambient temperature
Mercury-400BB "Indian"
Relax. delay 2.000 sec
Pulse 51.8 degrees
Acq. time 2.000 sec
Sweep 10.000 sec
2 repetitions
OBSERVE H1, 400.6260335 MHz
DATA PROCESSING
FT size 32768
Total time 8 min, 21 sec

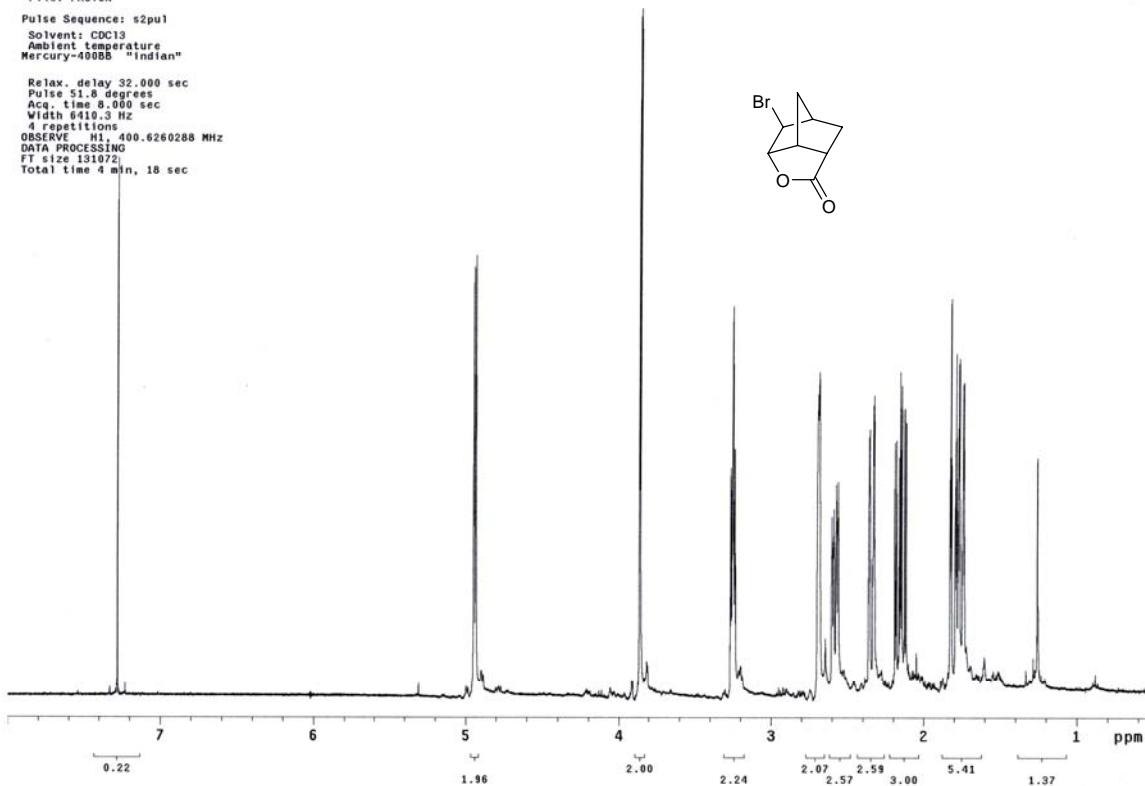


¹H NMR of 5-(iodomethyl)-dihydrofuran-2(3H)-one (**5**).

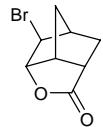


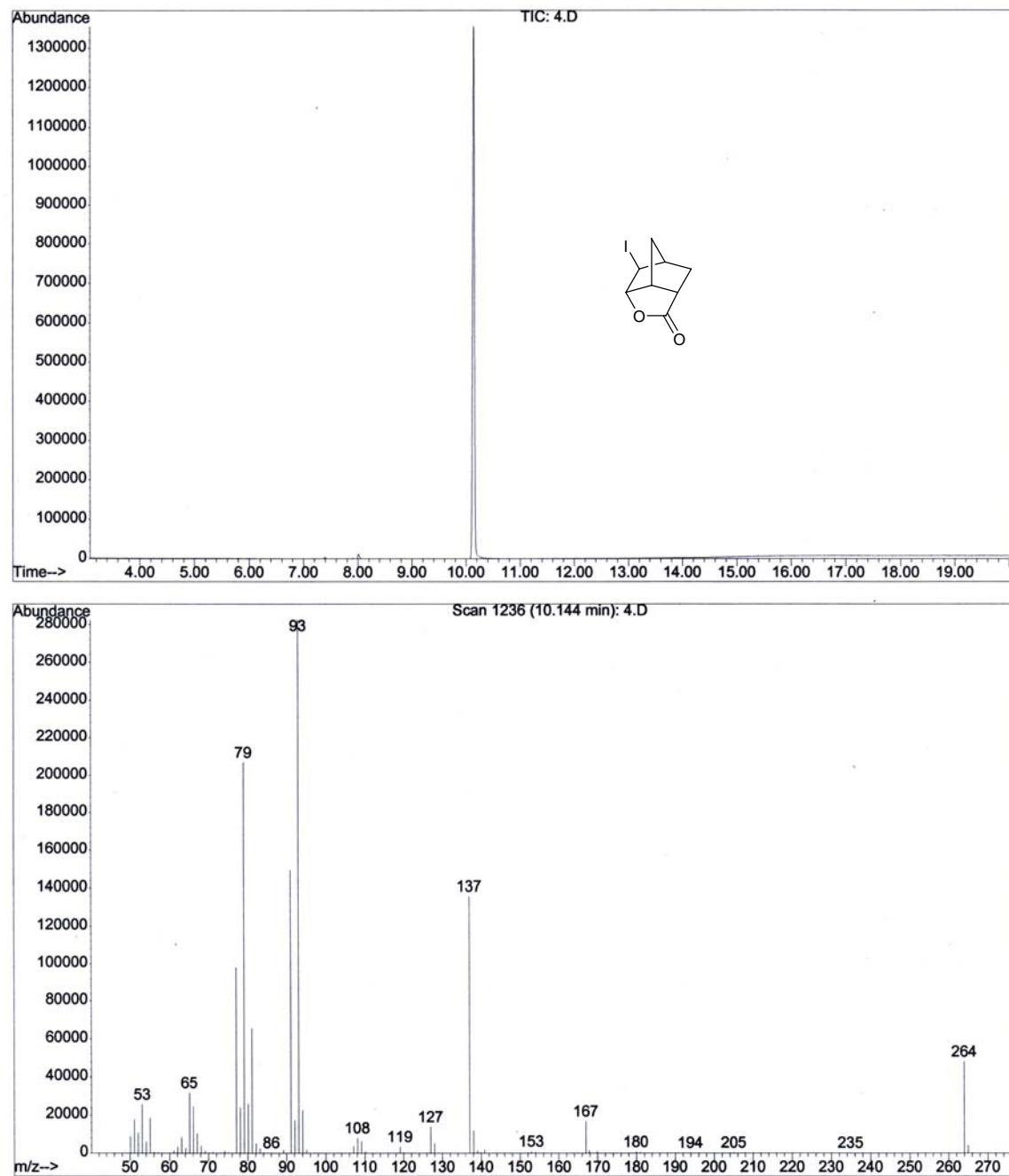
GC-MS of bromolactone 8.

05_13_08_C
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Sample directory: trans-4-L-0_16Jan2004
File: PROTON
Pulse Sequence: s2pul
Solvent: CDCl₃
Ambient temperature
Mercury-400B
"Indian"
Relax. delay 32.000 sec
Pulse 51.8 degrees
Acq. time 8.000 sec
Width 6410.3 Hz
4096 timesteps
OBSERVE H1 400.6260288 MHz
DATA PROCESSING
FT size 131072
Total time 4 min, 18 sec



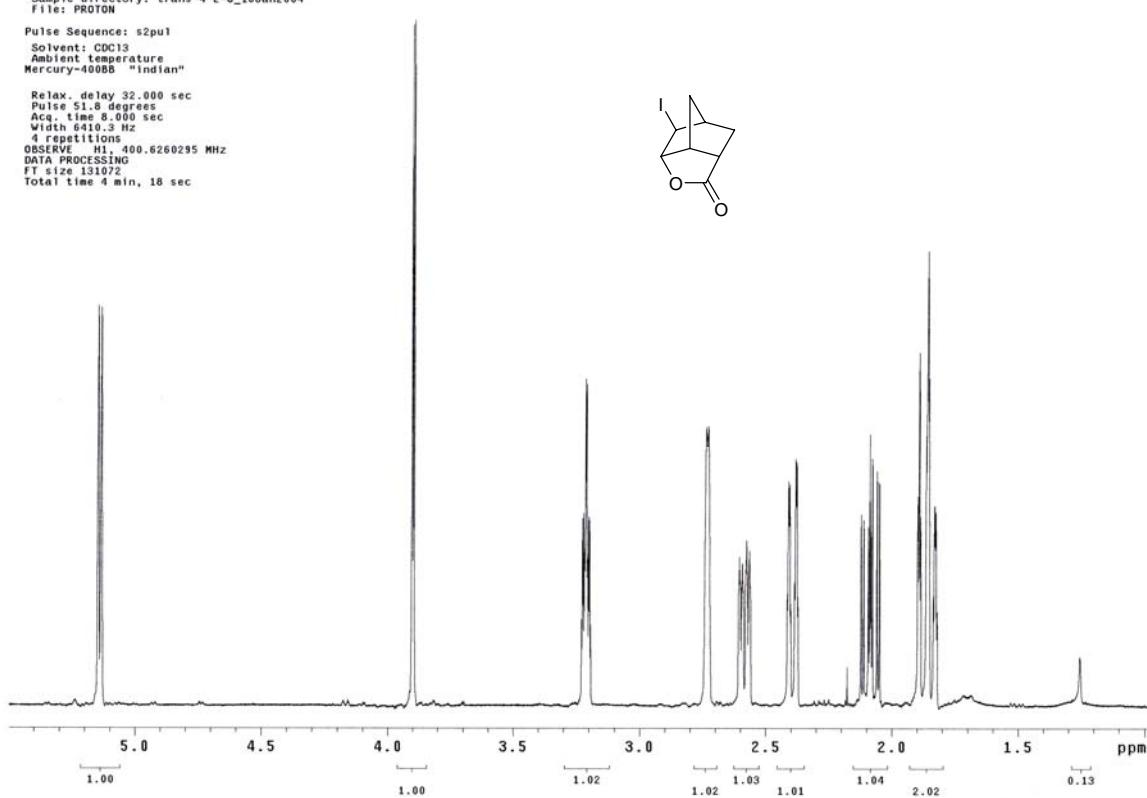
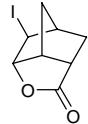
¹H NMR of bromolactone **8**.



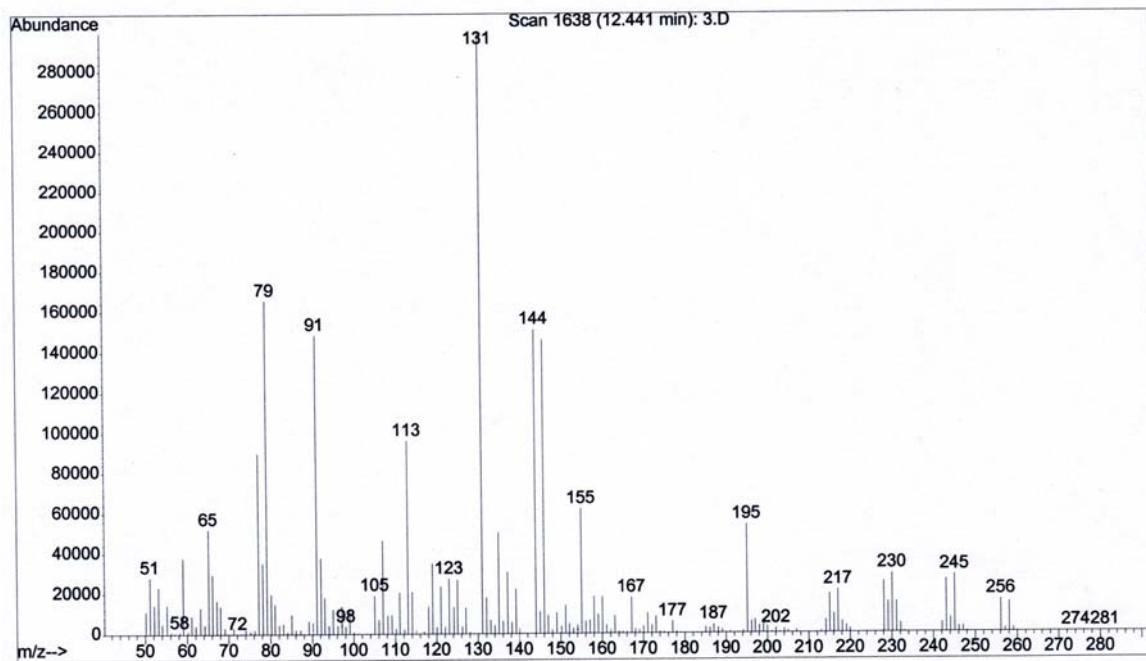
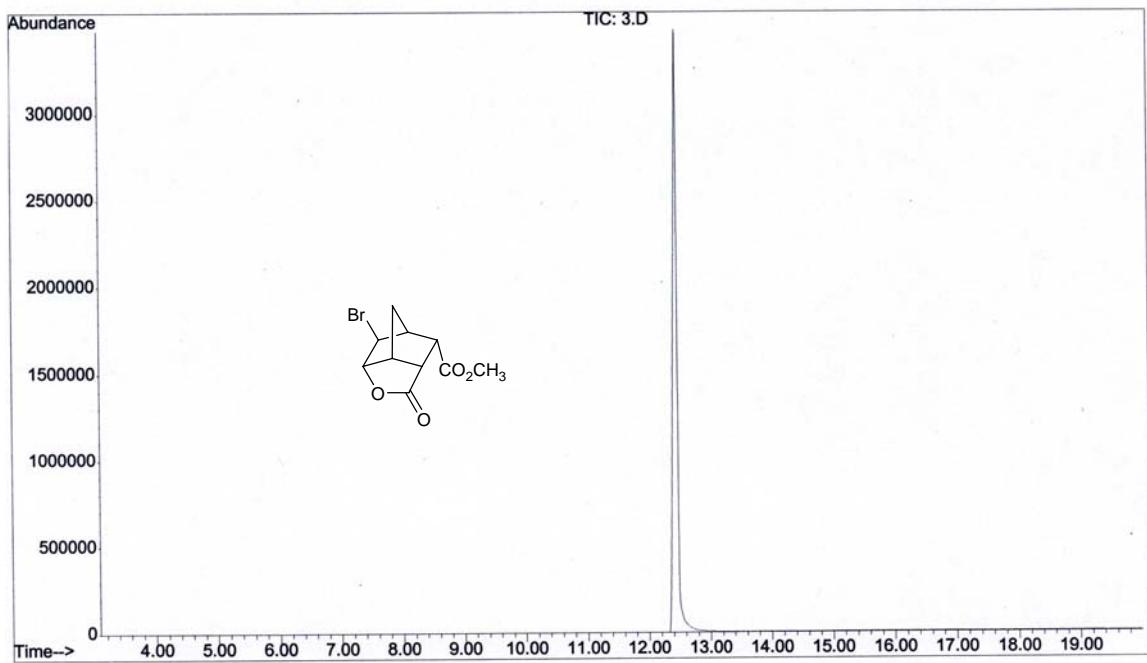


GC-MS of iodolactone 13.

05_14_08_D
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 Sample directory: trans-4-L-0_16Jan2004
 File: PROTON
 Pulse Sequence: s2pul
 Solvent: CDCl₃
 Ambient temperature
 Mercury-400B6 "Indian"
 Relax. delay 32.000 sec
 Pulse 51.8 degrees
 Acq. time 8.000 sec
 Width 6410.3 Hz
 4.096 tums
 OBSERVE H1 400.6260295 MHz
 DATA PROCESSING
 FT size 131072
 Total time 4 min, 18 sec



¹H NMR of iodolactone 13.

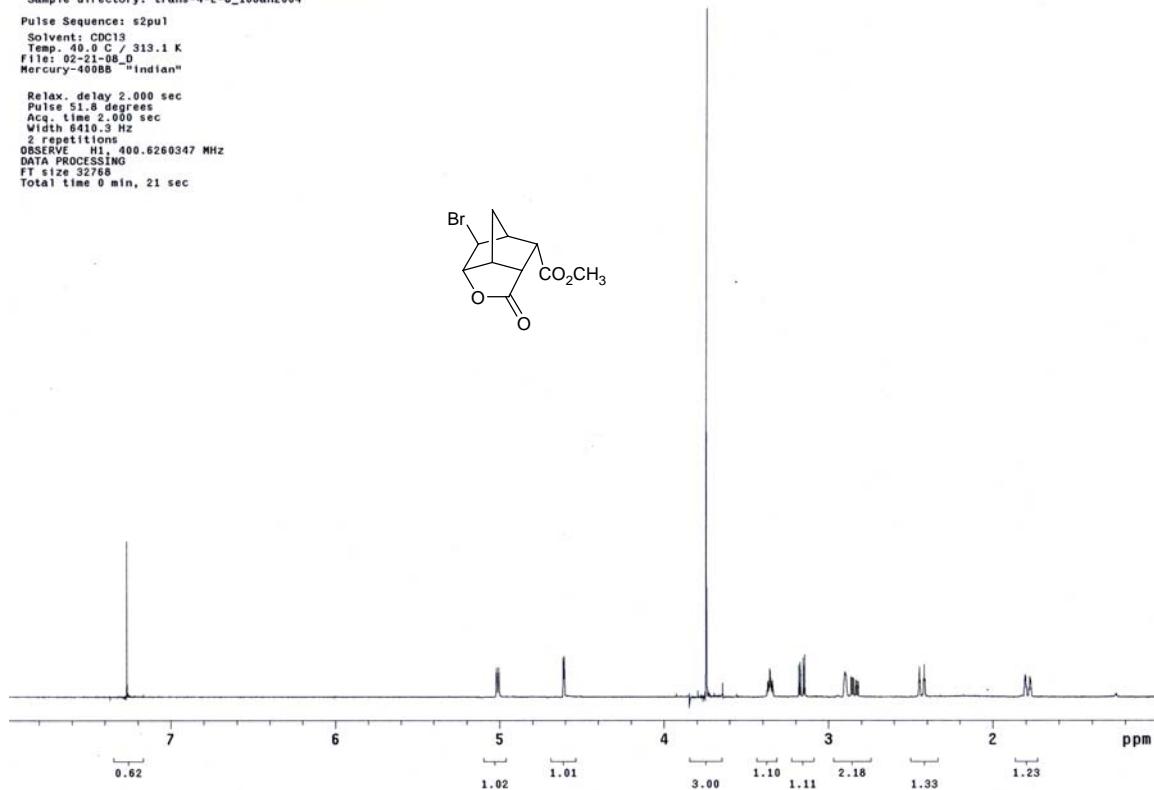
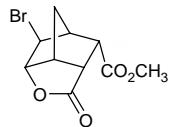


GC-MS of bromolactone ester 18.

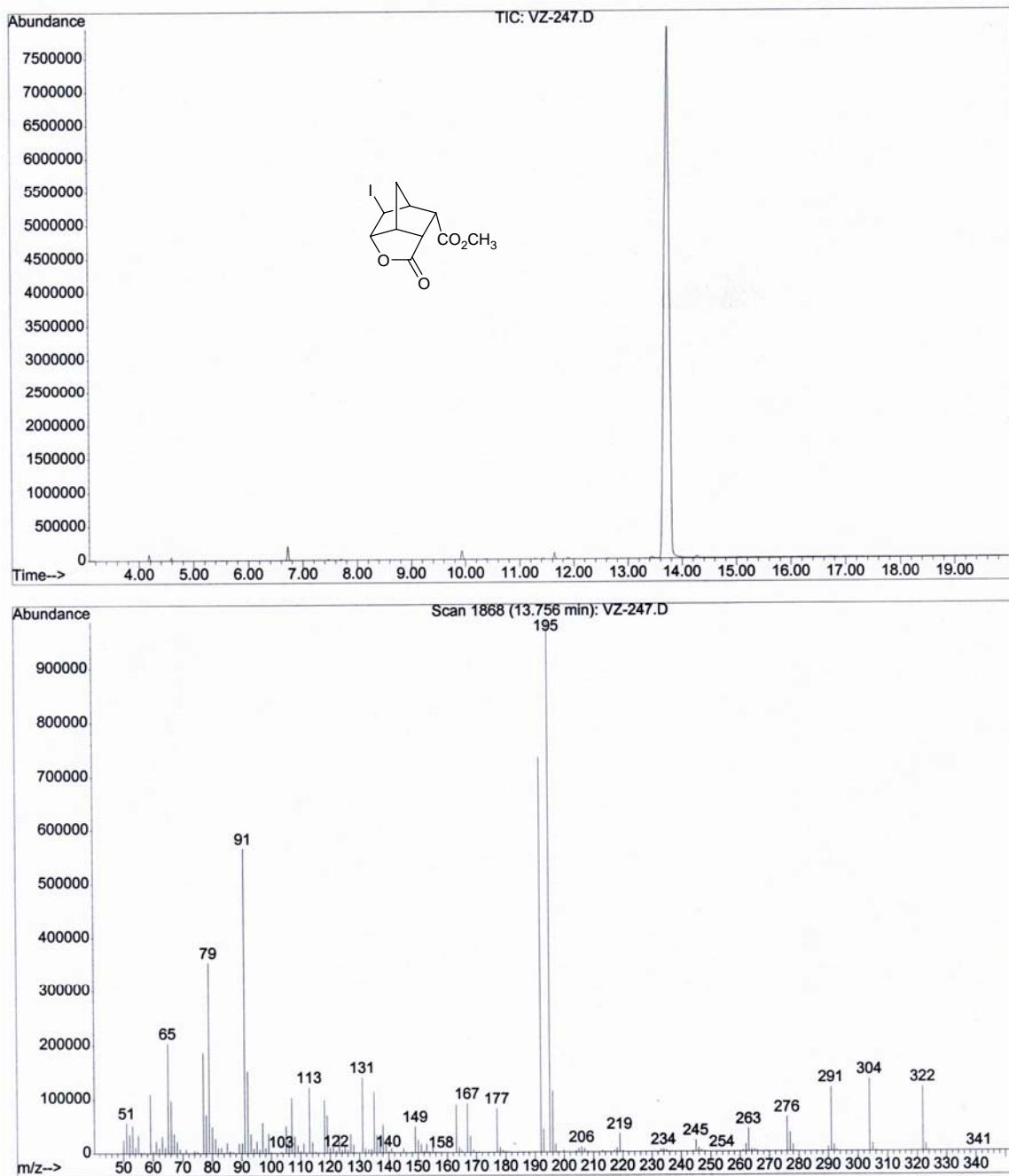
STANDARD 1H OBSERVE

Archive directory: /export/home/vnmr1/vnmrsys/data
 Sample directory: trans-4-L-0_16Jan2004

Pulse Sequence: s2pu1
 Solvent: CDCl₃
 Temperature: 313.1 K
 File: 02-21-08_D
 Mercury-400BB "Indian"
 Relax. delay 2.000 sec
 Pulse 51.8 degrees
 Acq. time 0.02 sec
 Width 6410.3 Hz
 2 repetitions
 OBSERVE 1H, 400.6260347 MHz
 DATA PROCESSING
 FT size 32768
 Total time 0 min, 21 sec



¹H NMR of bromolactone ester 18.



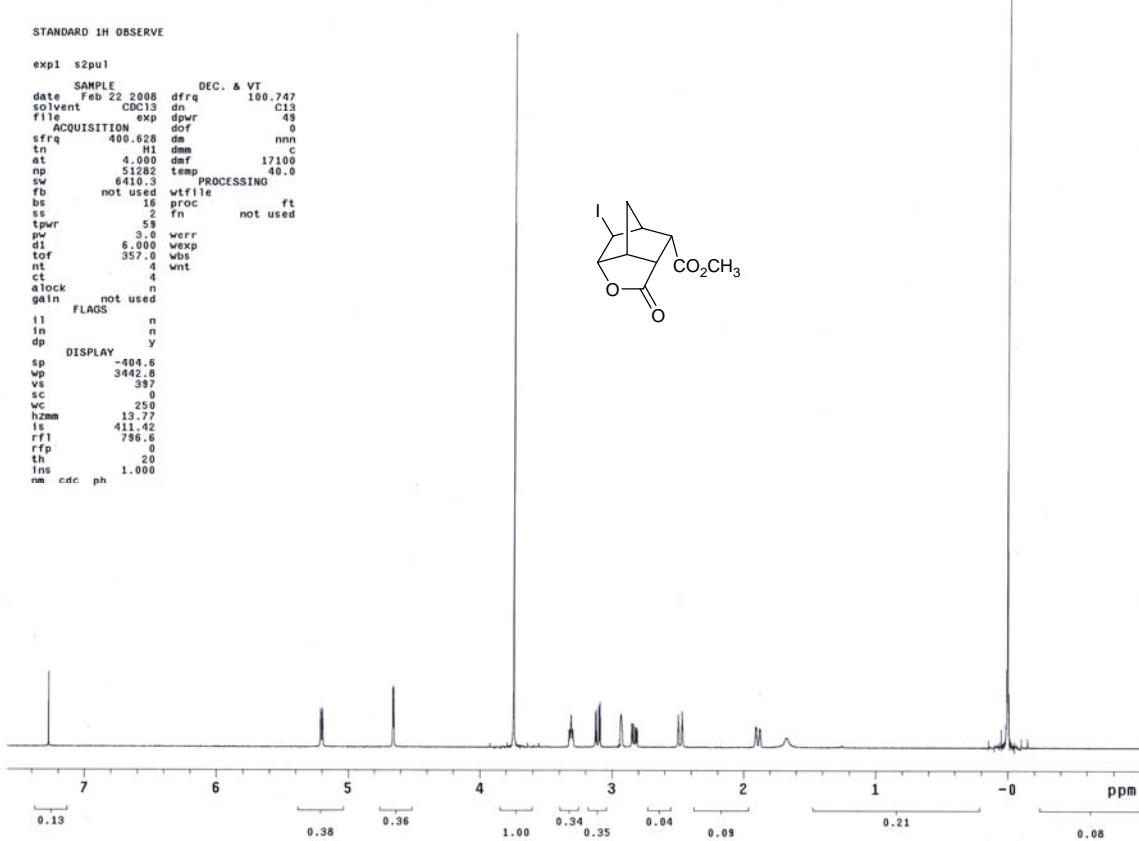
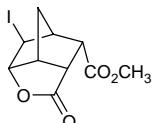
GC-MS of iodolactone ester **19**.

STANDARD 1H OBSERVE

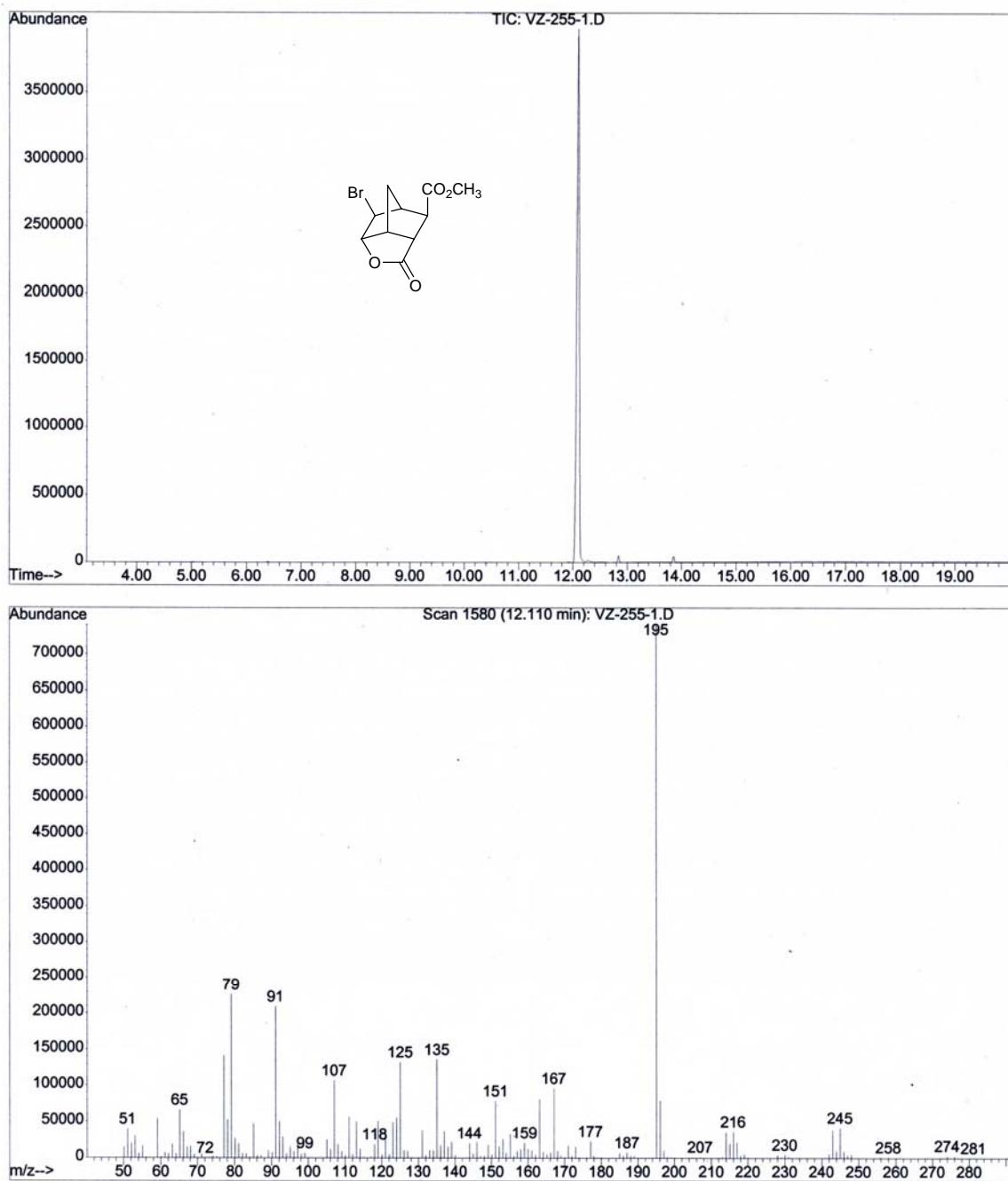
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solvent  CDCl3 dn       C13
file    exp dpwr   48
      ACQUISITION off    0
sfrq   400.628 dm      nnn
tn      H1 dmm      c
at     4.000 dmf     17100
sp      519.0 temp   40.0
sw     6410.3 PROCESSING
fb      not used wfile
bs      16 proc      ft
ss      51 fn       not used
tpwr   58
pw      3.0 werr
di     6.000 wexp
of     357.1 wbs
nt      4 wnt
ct      4
alock   n
gain    not used
FLAGS
il      n
in      n
dp      y
      DISPLAY
sp     -404.6
wp     3442.8
vs     397
vc     0
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rf1    79.6
rfp    0
th     20
ins   1.000
nm cdc ph

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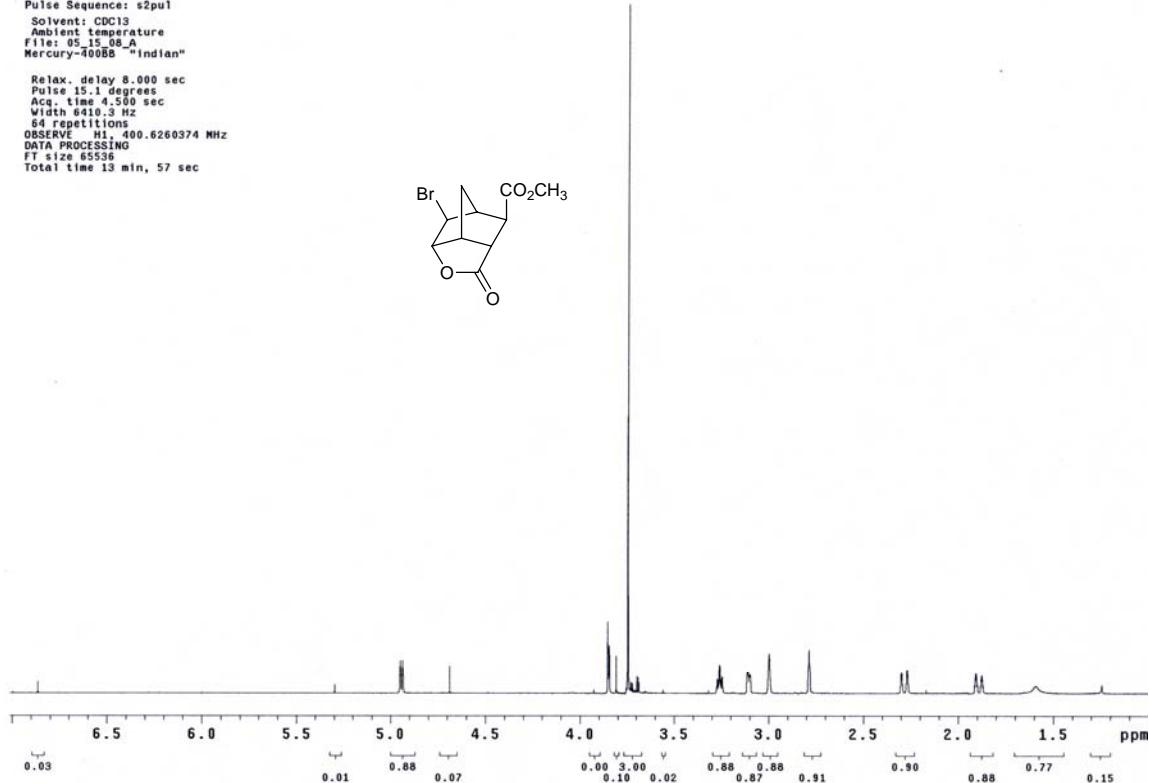
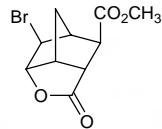
¹H NMR of iodolactone ester 19.



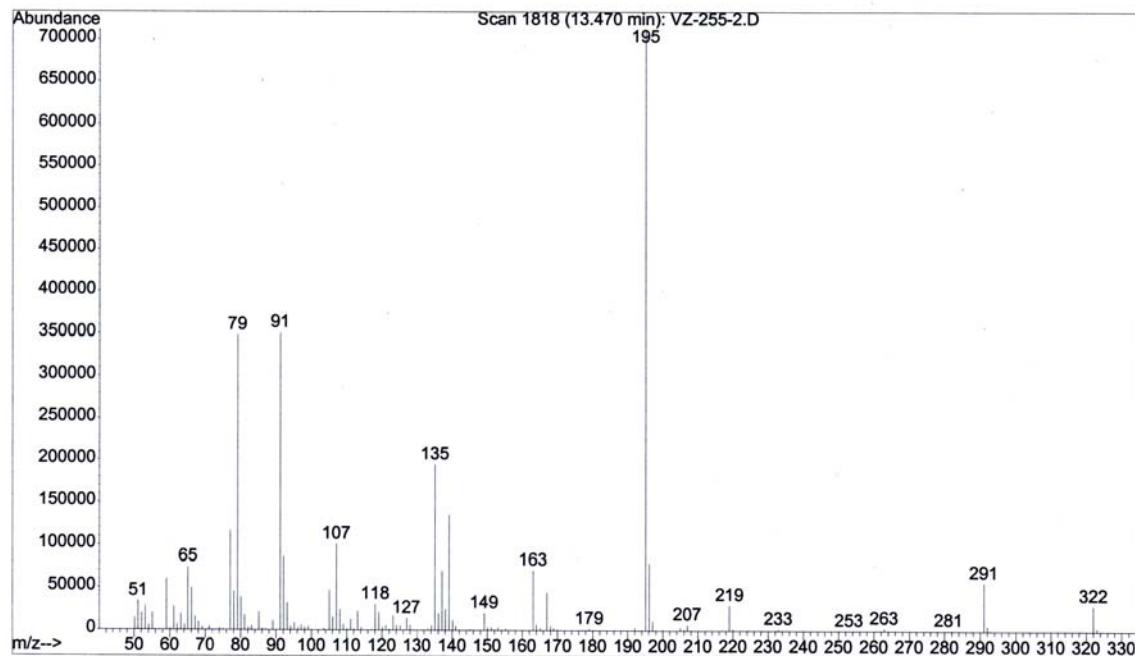
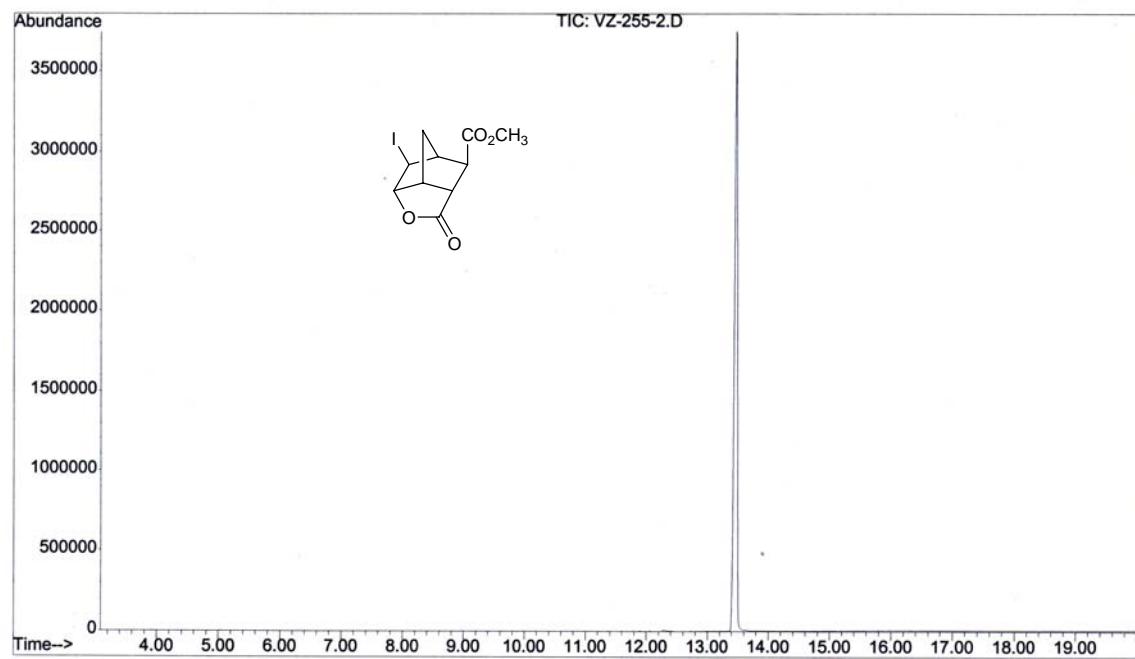
GC-MS of bromolactone ester 21.

05_15_08_A
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Sample directory: trans-4-L-0_16Jan2004

Pulse Sequence: s2pul
Solvent: CDCl₃
Ambient temperature
File: 05_15_08_A
Mercury-400B6 "Indian"
Relax. delay 8.000 sec
Pulse 15.1 degrees
Acq. time 4.500 sec
Width 6410.3 Hz
64 repetitions
OBSERVE: H1 400.6260374 MHz
DATA PROCESSING
FT size 65536
Total time 13 min, 57 sec

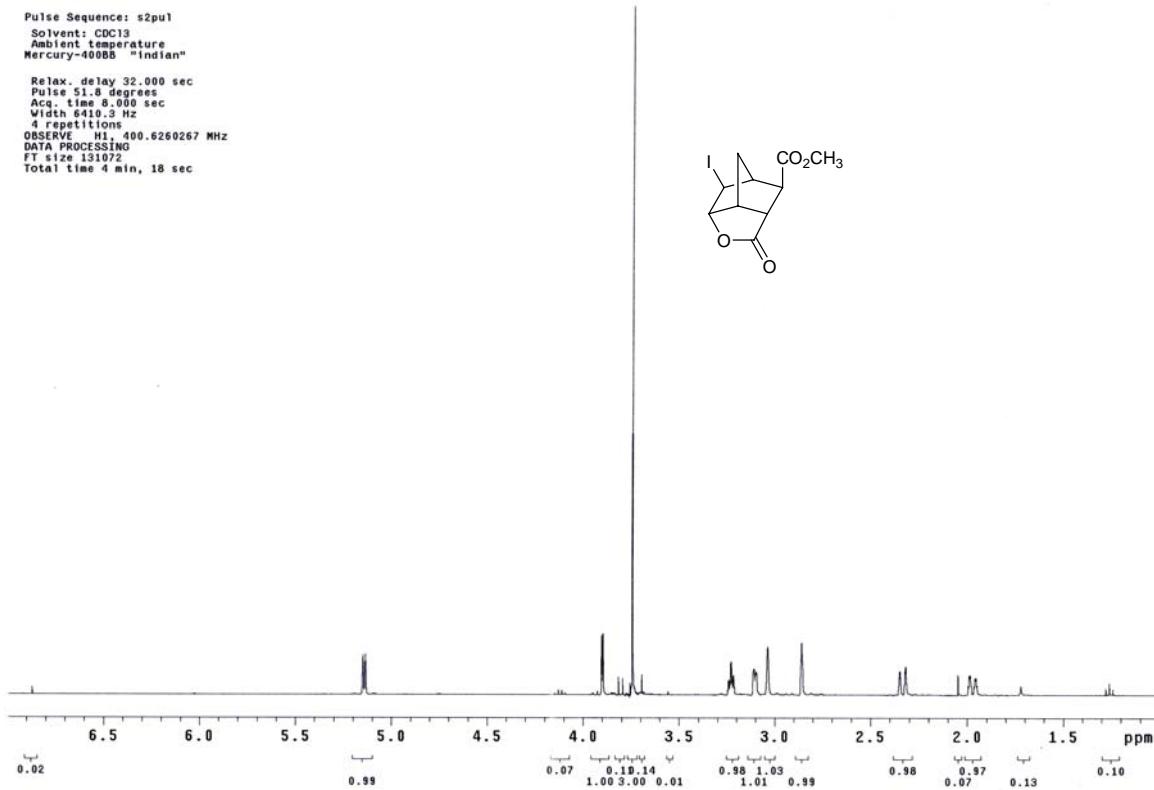


¹H NMR of bromolactone ester 21.



GC-MS of iodolactone ester **22**.

05_14_08_A
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Sample directory: trans-4-L-0_16Jan2004
File: PROTON
Pulse Sequence: s2pul
Solvent: CDCl₃
Ambient temperature
Mercury-400BPC "Indian"
Relax. delay 32.000 sec
Pulse 51.8 degrees
Acq. time 8.000 sec
Width 6410.3 Hz
4 Repetitions
OBSERVE FREQ 400.6260267 MHz
DATA PROCESSING
FT size 131072
Total time 4 min, 18 sec



^1H NMR of iodolactone ester **22**.