Supporting Information

Solvent-free Horner-Wadsworth-Emmons Reaction Catalyzed by DBU

Kaori Ando, Kyohei Yamada

Department of Chemistry, Fuculty of Engineering, Gifu Univeresity, Yanagido 1-1, Gifu 501-1193, Japan

ando@gifu-u.ac.jp

Contents

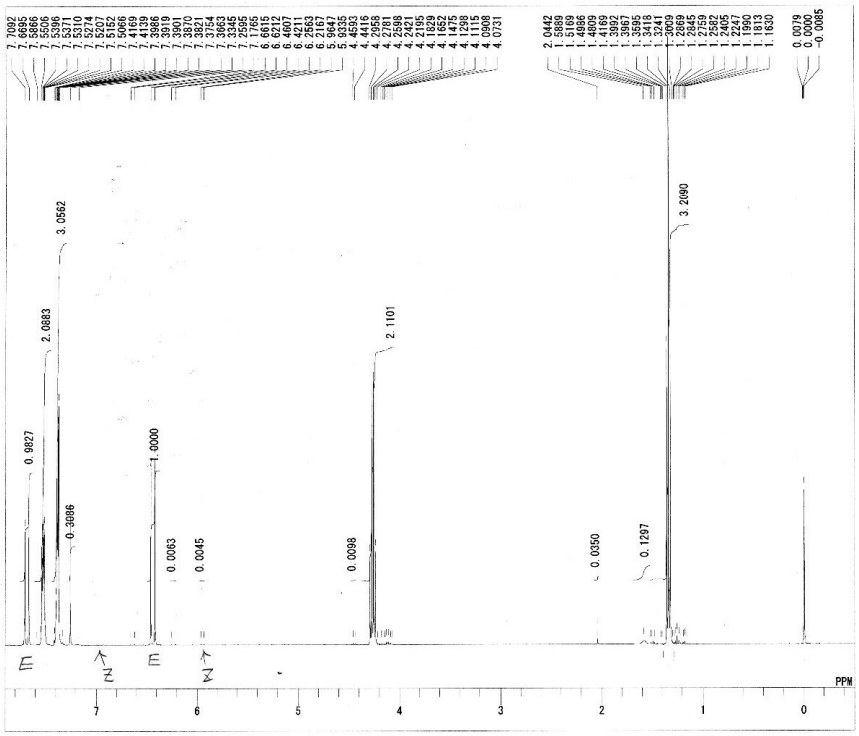
1) Instrumentation and Chemicals: The ^{1}H NMR spectra were recorded on a JEOL α-400 (400 MHz) in $CDCl_3$ using tetramethylsilane as an internal standard (δ = 0 ppm). The column chromatograhphy was carried out using silica gel 60N of Kanto Chemical Co., Ltd. Commercially available reagents were used without purification. K_2CO_3, NaOH, and KOH were ground in a mortar with a pestle.

A typical experimental procedure of the solvent-free HWE reaction of 1 with aldehydes (entry 11 in Table 3): To a mixture of **1** (0.224 g,1.0 mmol), DBU (0.004 mL, 0.03 mmol), and K_2CO_3 (0.278 g, 2.0 mmol) was added *n*-octanal (0.171 mL, 1.1 mmol) and the resulting mixture was stirred for 2 h at room temperature under Ar atmosphere. The reaction was quenched with water (3 mL) and extracted with AcOEt (5 mL). The extract was washed with brine, dried ($MgSO_4$), and concentrated to give **4g** in >90% purity containing 5% *n*-octanal (*E*:*Z* = 99:1). The product was isolated by flash chromatography (hexane:AcOEt = 30:1) as coloreless oil (0.190 g, 96% yield).

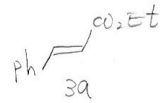
A tipical experimetal procedure of the solvent-free HWE reaction of 1 with ketones (entry 10 in Table 4): To a mixture of **1** (0.224 g,1.0 mmol), DBU (0.031 mL, 0.2 mmol), and Cs_2CO_3 (0.399 g, 1.2 mmol) was added 4'-nitoroacetophenone (0.169 g, 1.0 mmol) and the resulting mixture was stirred for 3 d at room temperature under Ar atmosphere. The reaction was quenched with water (3 mL) and extracted with AcOEt (5 mL). The extract was washed with brine, dried ($MgSO_4$), and concentrated to give **5b** (*E*:*Z* = 99:1). The product was isolated by flash chromatography (hexane:AcOEt = 15:1) as coloreless oil (0.185 g, 78% yield).

2) NMR charts of the HWE products.

kyo-332-3

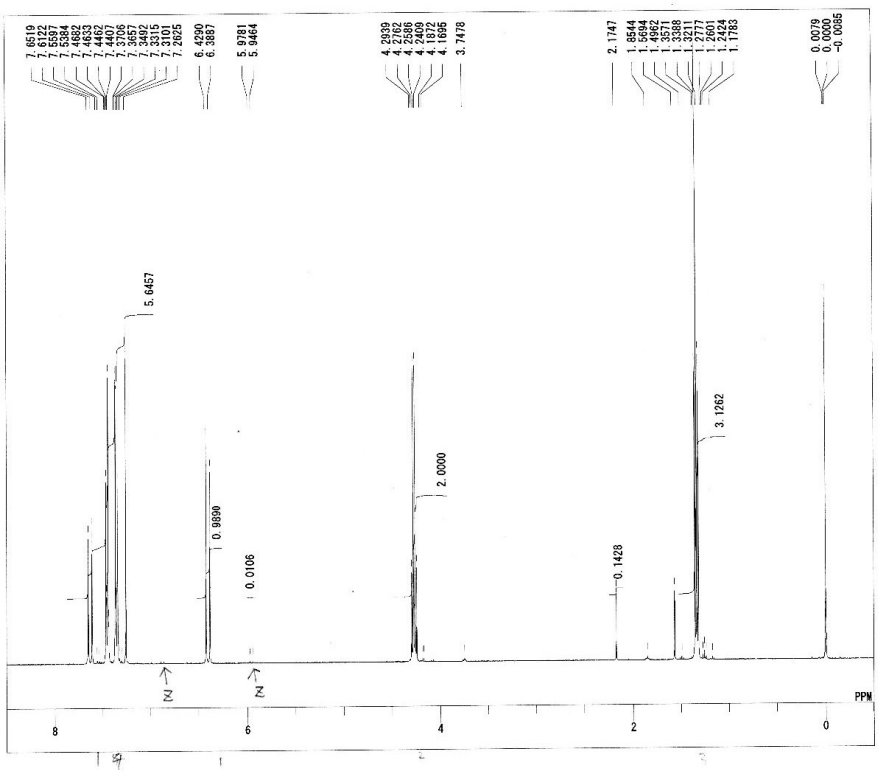


DATIM Thu Oct 14 11:39:37 2010
EXMOD SINGL
IRMOD NON
OBNUC 1H
OBFRQ 399.65 MHz
OBSET 0.00 KHz
PW1 6.50 usec
POINT 32768
SAMPO 32768
TIMES 8
SCANS 8
DUMMY 4
FREQU 7993.60 Hz
FILTR 4000 Hz
ACQTM 4.0993 sec
DEADT 71.75 usec
DELAY 50.00 usec
PD 1.9040 sec
ADBIT 16
RGAIN 17
IRNUC 1H
IRFRQ 399.65 MHz
IRSET 0.00 KHz
IRPPW 50 usec
IRATN 511
IRRNS 0
IRBP1 30
IRBP2 6
BF 0.10 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
XE 3358.40 Hz
XS 513.26 Hz
REFVL
DFILE kyo-332-3.als
MENUF A1H
SHMFL TH5AT
LKSET 0.00 KHz
LKLEV 200
LGAIN 22
LKPHS 20
LKSIG 681
CTEMP 20.7 c
CSPED 22 Hz
FIELD 4515

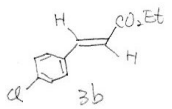


E:Z = 99.6:0.4

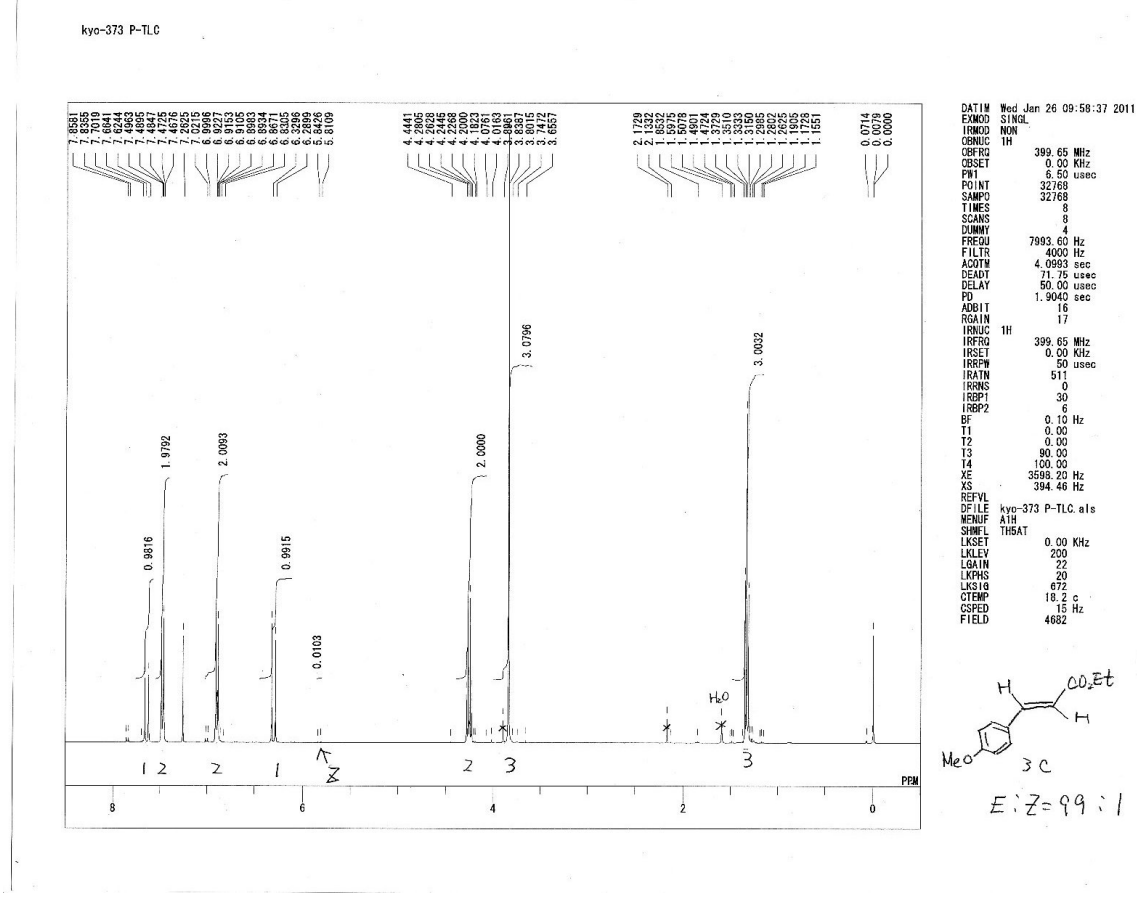
kyo-316-2 re



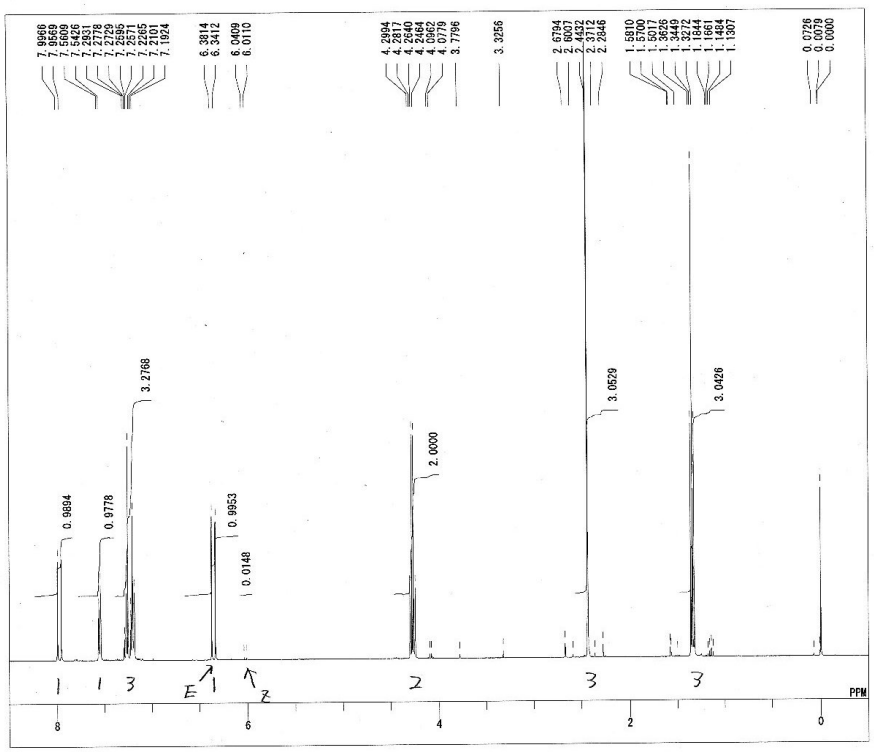
DATIM Sun Jan 23 09:03:28 2011
EXMOD SINGL
IRMOD NON
OBNUC 1H
OBFRQ 399.65 MHz
OBSET 0.00 KHz
PW1 6.50 usec
POINT 32768
SAMPO 32768
TIMES 8
SCANS 8
DUMMY 4
FREQU 7993.60 Hz
FILTR 4000 Hz
ACQTM 4.0993 sec
DEADT 71.75 usec
DELAY 50.00 usec
PD 1.9040 sec
ADBIT 16
RGAIN 19
IRNUC 1H
IRFRQ 399.65 MHz
IRSET 0.00 KHz
IRPPW 50 usec
IRATN 511
IRRNS 0
IRBP1 30
IRBP2 6
BF 0.10 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
XE 3598.20 Hz
XS 394.70 Hz
REFVL
DFILE _DEFAULT.ALS
MENUF A1H
SHMFL TH5AT
LKSET 0.00 KHz
LKLEV 200
LGAIN 22
LKPHS 20
LKSIG 722
CTEMP 18.0 c
CSPED 14 Hz
FIELD 4678



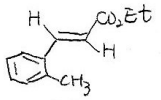
E:Z = 99:1


kyo-373 P-TLC
0.9816
1.9792
2.0093
0.9915
0.0103
2.0000
3.0796
3.0032
H2O
PPM
DATIM Wed Jan 26 09:58:37 2011
EXMOD SINGL
IRMOD NON
OBNUC 1H
OBFRQ 399.65 MHz
OBSET 0.00 KHz
PW1 6.50 usec
POINT 32768
SAMPD 32768
TIMES 8
SCANS 8
DUMMY 4
FREQU 7993.60 Hz
FILTR 4000 Hz
ACQTM 4.0993 sec
DEADT 71.75 usec
DELAY 50.00 usec
PD 1.9040 sec
ADBIT 16
RGAIN 17
IRNUC 1H
IRFRQ 399.65 MHz
IRSET 0.00 KHz
IRRPW 50 usec
IRATN 511
IRRNS 0
IRBP1 30
IRBP2 6
BF 0.10 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
XE 3598.20 Hz
XS 394.46 Hz
REFVL
DFILE kyo-373 P-TLC.als
MENUF A1H
SHMFL TH5AT
LKSET 0.00 KHz
LKLEV 200
LGAIN 22
LKPHS 20
LKSIG 672
CTEMP 18.2 c
CSPED 15 Hz
FIELD 4682
H
CO2Et
H
MeO
3c
E:Z=99:1

kyo-372-2



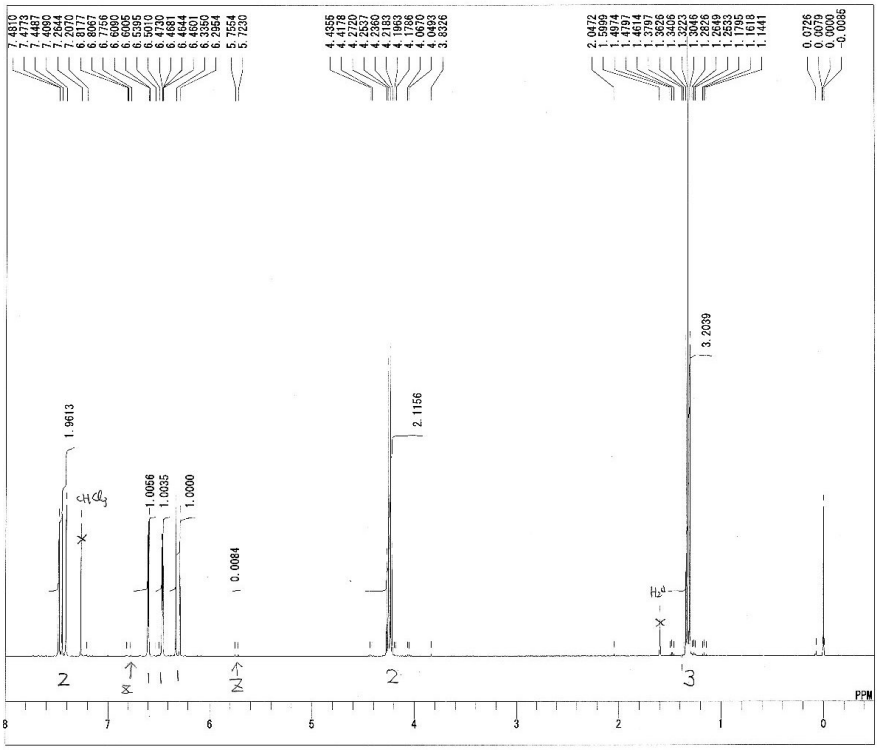
```
DATIM  Fri Jan 28 11:35:22 2011
EXMOD  SINGL
IRMOD  NON
OBNUC  1H
OBFRQ        399.65 MHz
OBSET          0.00 KHz
PW1            6.50 usec
POINT         32768
SAMPO         32768
TIMES             8
SCANS             8
DUMMY             4
FREQU       7993.60 Hz
FILTR          4000 Hz
ACQTM       4.0993 sec
DEADT         71.75 usec
DELAY         50.00 usec
PD          1.9040 sec
ADBIT            16
RGAIN            18
IRNUC  1H
IRFRQ        399.65 MHz
IRSET          0.00 KHz
IRRPW            50 usec
IRATN           511
IRRNS             0
IRBP1            30
IRBP2             6
BF             0.10 Hz
T1             0.00
T2             0.00
T3            90.00
T4           100.00
XE         3598.20 Hz
XS          393.24 Hz
REFVL
DFILE  kyo-372-2.als
MENUF  A1H
SHMFL  TH5AT
LKSET          0.00 KHz
LKLEV             200
LGAIN              22
LKPHS              20
LKSIG             764
CTEMP        18.2 c
CSPED          15 Hz
FIELD            4695
```



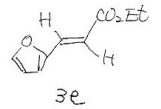
3d

E:Z=99:1

kyo-241-2



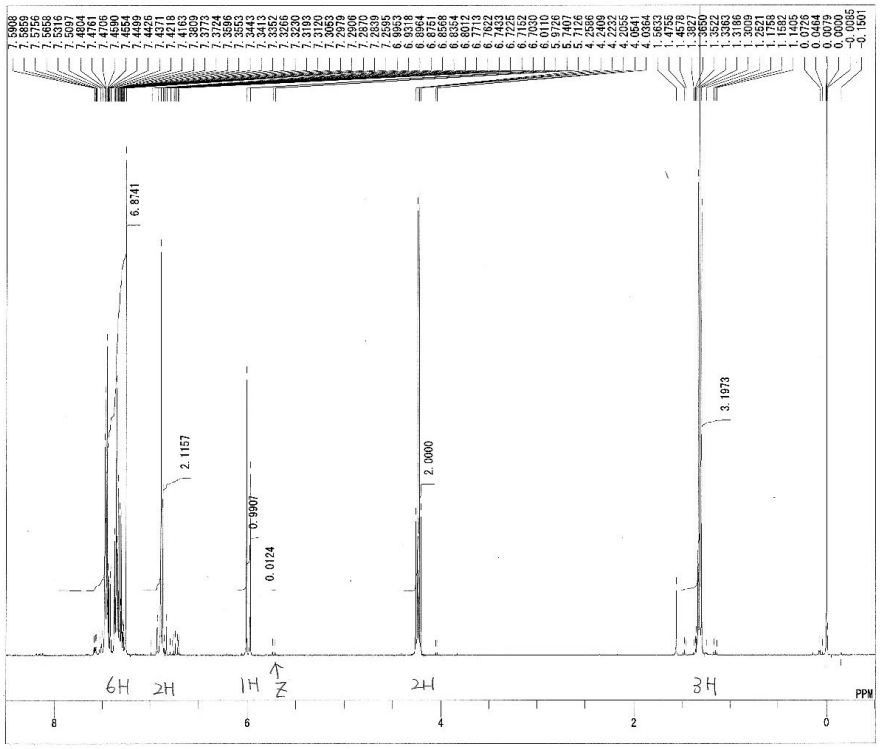
```
DATIM  Thu Jan 28 16:57:38 2010
EXMOD  SINGL
IRMOD  NON
OBNUC  1H
OBFRQ        399.65 MHz
OBSET          0.00 KHz
PW1            6.50 usec
POINT         32768
SAMPO         32768
TIMES             8
SCANS             8
DUMMY             4
FREQU       7993.60 Hz
FILTR          4000 Hz
ACQTM       4.0993 sec
DEADT         71.75 usec
DELAY         50.00 usec
PD          1.9040 sec
ADBIT            16
RGAIN            18
IRNUC  1H
IRFRQ        399.65 MHz
IRSET          0.00 KHz
IRRPW            50 usec
IRATN           511
IRRNS             0
IRBP1            30
IRBP2             6
BF             0.10 Hz
T1             0.00
T2             0.00
T3            90.00
T4           100.00
XE         3398.40 Hz
XS          494.72 Hz
REFVL
DFILE  kyo-241-2.als
MENUF  A1H
SHMFL  TH5AT
LKSET          0.00 KHz
LKLEV             200
LGAIN              22
LKPHS              41
LKSIG             757
CTEMP        18.7 c
CSPED          23 Hz
FIELD            4192
```



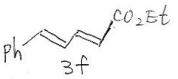
3e

E:Z=99:1

kyo-239-2




```
DATIM   Fri Jan 22 10:02:50 2010
EXMOD   SINGL
IRMOD   NON
OBNUC   1H
OBFRQ        399.65 MHz
OBSET          0.00 KHz
PW1            6.50 usec
POINT         32768
SAMPO         32768
TIMES             8
SCANS             8
DUMMY             4
FREQU       7993.60 Hz
FILTR       4000 Hz
ACQTM          4.0993 sec
DEADT         71.75 usec
DELAY         50.00 usec
PD             1.9040 sec
ADBIT            16
RGAIN            20
IRNUC   1H
IRFRQ        399.65 MHz
IRSET          0.00 KHz
IRRPW            50 usec
IRATN           511
IRRNS             0
IRBP1            30
IRBP2             6
BF             0.10 Hz
T1             0.00
T2             0.00
T3            90.00
T4           100.00
XE          3598.20 Hz
XS           393.00 Hz
REFVL
DFILE   kyo-239-2.als
MENUF   A1H
SHMFL   TH5AT
LKSET          0.00 KHz
LKLEV           200
LGAIN            22
LKPHS            41
LKSIG           746
CTEMP          19.3 c
CSPED            19 Hz
FIELD          4176
```

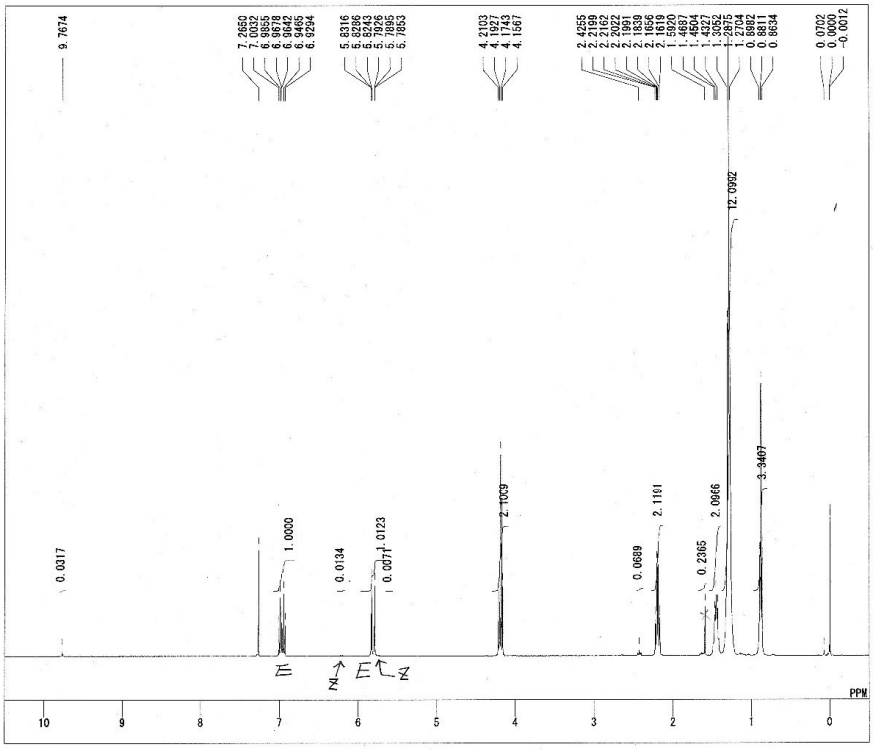


3f

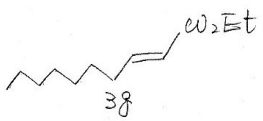
EE : EZ

99 : 1

kyo-233-2

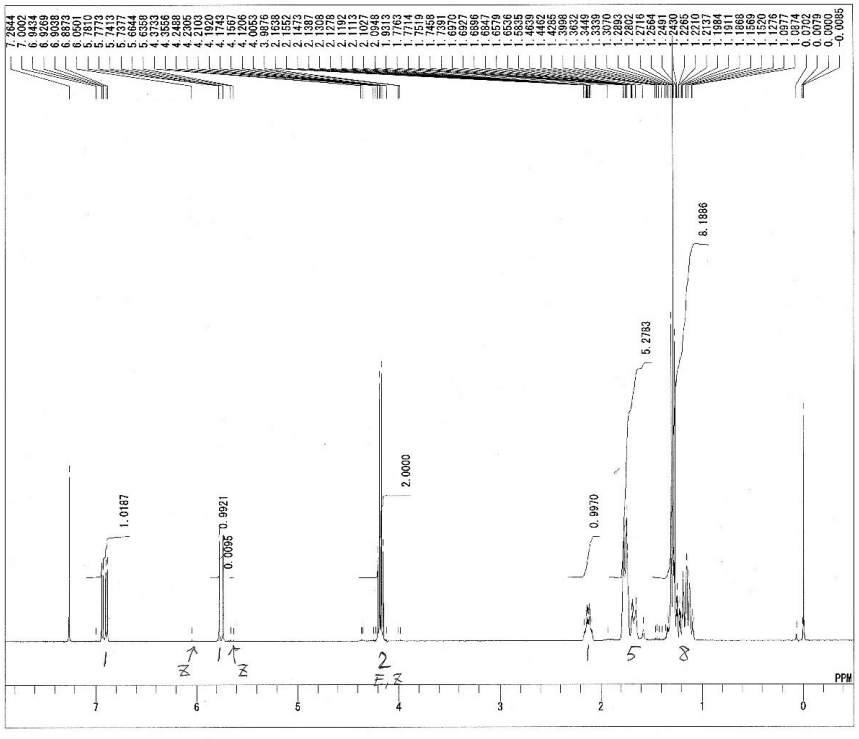


DATIM Thu Dec 24 10:09:04 2009
EXMOD SINGL
IRMOD NON
OBNUC 1H
OBFRQ 399.65 MHz
OBSET 0.00 KHz
PW1 6.50 usec
POINT 32768
SAMPO 32768
TIMES 8
SCANS 8
DUMMY 4
FREQU 7993.60 Hz
FILTR 4000 Hz
ACQTM 4.0993 sec
DEADT 71.75 usec
DELAY 50.00 usec
PD 1.9040 sec
ADBIT 16
RGAIN 14
IRNUC 1H
IRFRQ 399.65 MHz
IRSET 0.00 KHz
IRPPW 50 usec
IRATN 511
IRRNS 0
IRBP1 30
IRBP2 6
BF 0.10 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
XE 4397.85 Hz
XS -4.88 Hz
REFVL
DFILE kyo-233-2.als
MENUF A1H
SHMFL TH5AT
LKSET 0.00 KHz
LKLEV 200
LGAIN 22
LKPHS 41
LKSTG 724
CTEMP 17.8 c
CSPED 17 Hz
FIELD 4119

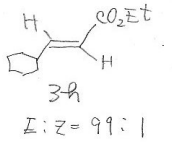


E:Z = 99:1

kyo-236-2

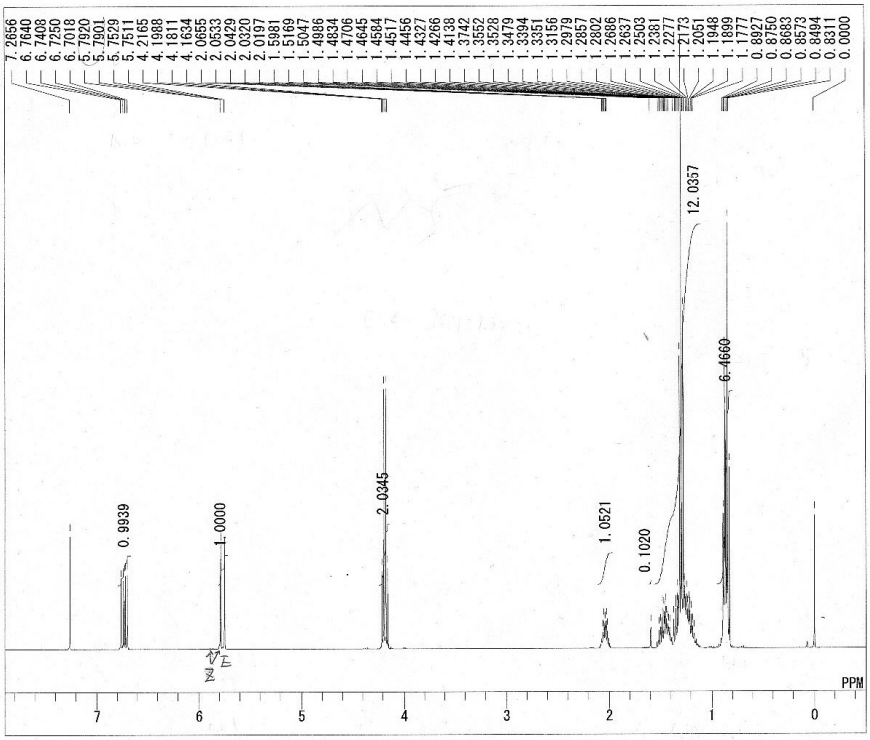


DATIM Tue Jan 12 10:11:36 2010
EXMOD SINGL
IRMOD NON
OBNUC 1H
OBFRQ 399.65 MHz
OBSET 0.00 KHz
PW1 6.50 usec
POINT 32768
SAMPO 32768
TIMES 8
SCANS 8
DUMMY 4
FREQU 7993.60 Hz
FILTR 4000 Hz
ACQTM 4.0993 sec
DEADT 71.75 usec
DELAY 50.00 usec
PD 1.9040 sec
ADBIT 16
RGAIN 15
IRNUC 1H
IRFRQ 399.65 MHz
IRSET 0.00 KHz
IRPPW 50 usec
IRATN 511
IRRNS 0
IRBP1 30
IRBP2 6
BF 0.10 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
XE 3358.40 Hz
XS 514.72 Hz
REFVL
DFILE kyo-236-2.als
MENUF A1H
SHMFL TH5AT
LKSET 0.00 KHz
LKLEV 200
LGAIN 22
LKPHS 41
LKSTG 767
CTEMP 18.4 c
CSPED 22 Hz
FIELD 4158

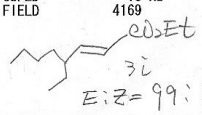


E:Z = 99:1

kyo-238-2

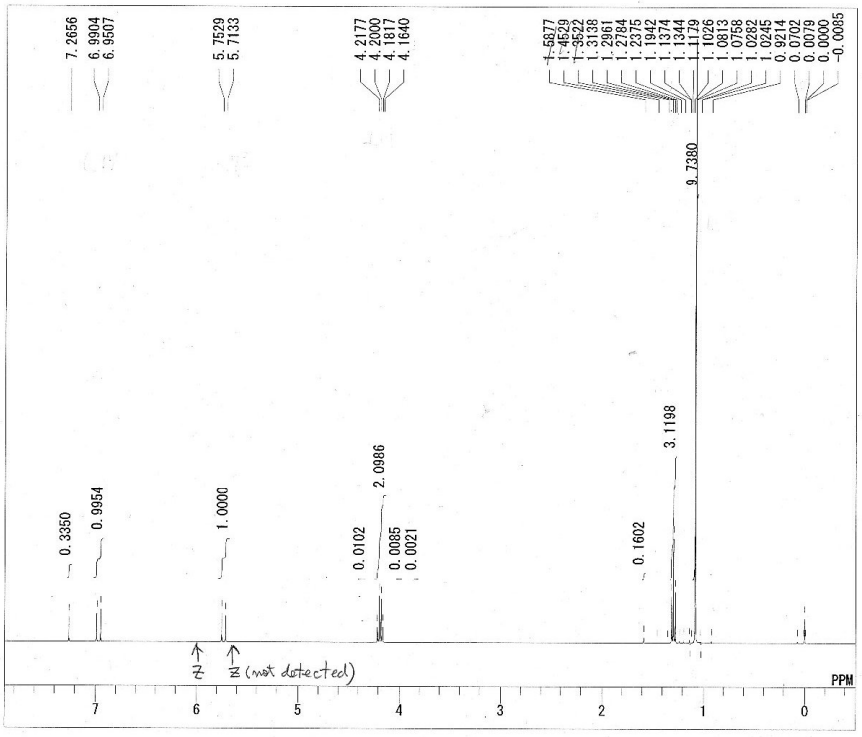


DATIM Tue Jan 19 14:46:13 2010
EXMOD SINGL
IRMOD NON
OBNUC 1H
OBFRQ 399.65 MHz
OBSET 0.00 KHz
PW1 6.50 usec
POINT 32768
SAMPO 32768
TIMES 8
SCANS 8
DUMMY 4
FREQU 7993.60 Hz
FILTR 4000 Hz
ACQTM 4.0993 sec
DEADT 71.75 usec
DELAY 50.00 usec
PD 1.9040 sec
ADBIT 16
RGAIN 13
IRNUC 1H
IRFRQ 399.65 MHz
IRSET 0.00 KHz
IRRPW 50 usec
IRATN 511
IRRNS 0
IRBP1 30
IRBP2 6
BF 0.10 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
XE 3358.40 Hz
XS 515.46 Hz
REFVL
DFILE kyo-238-2.als
MENUF A1H
SHMFL TH5AT
LKSET 0.00 KHz
LKLEV 200
LGAIN 22
LKPHS 41
LKSIG 734
CTEMP 19.8 c
CSPED 18 Hz
FIELD 4169

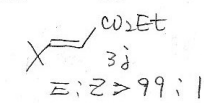


E:Z= 99:1

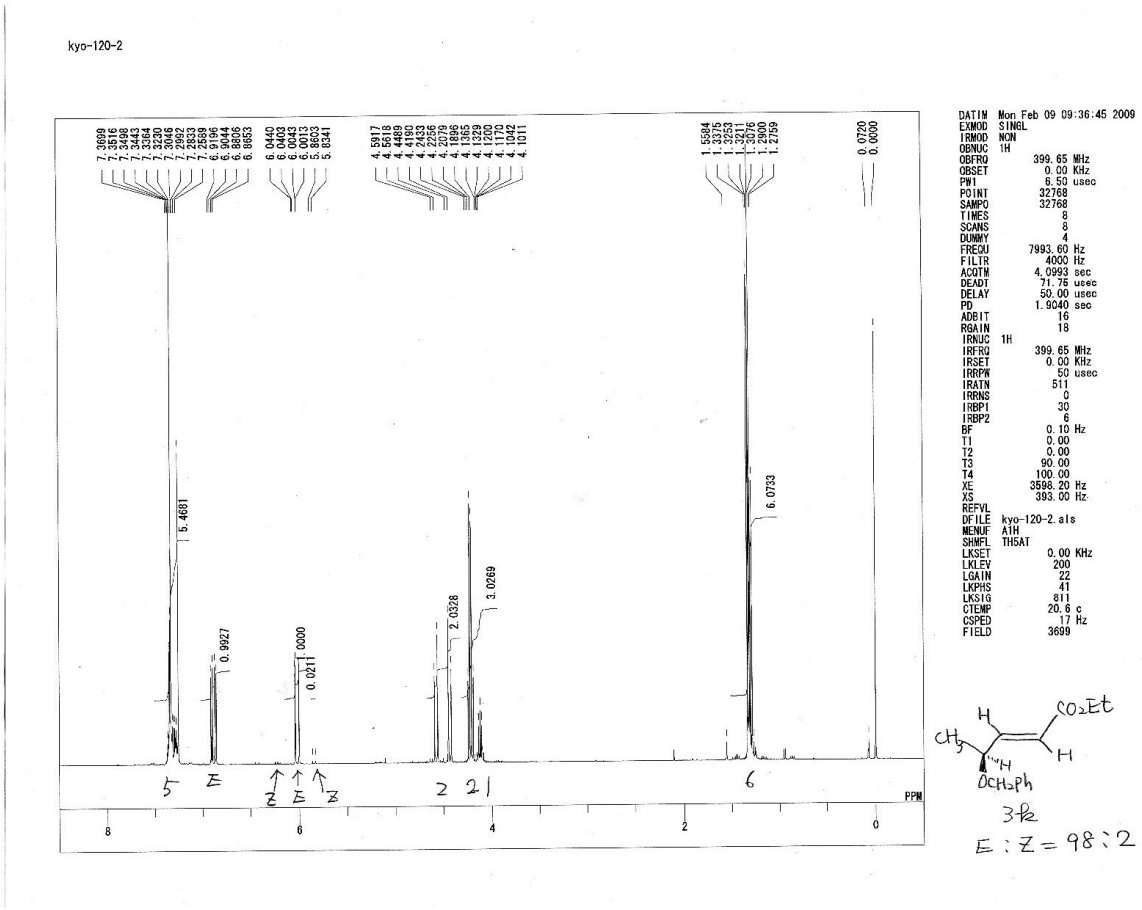
kyo-242-2



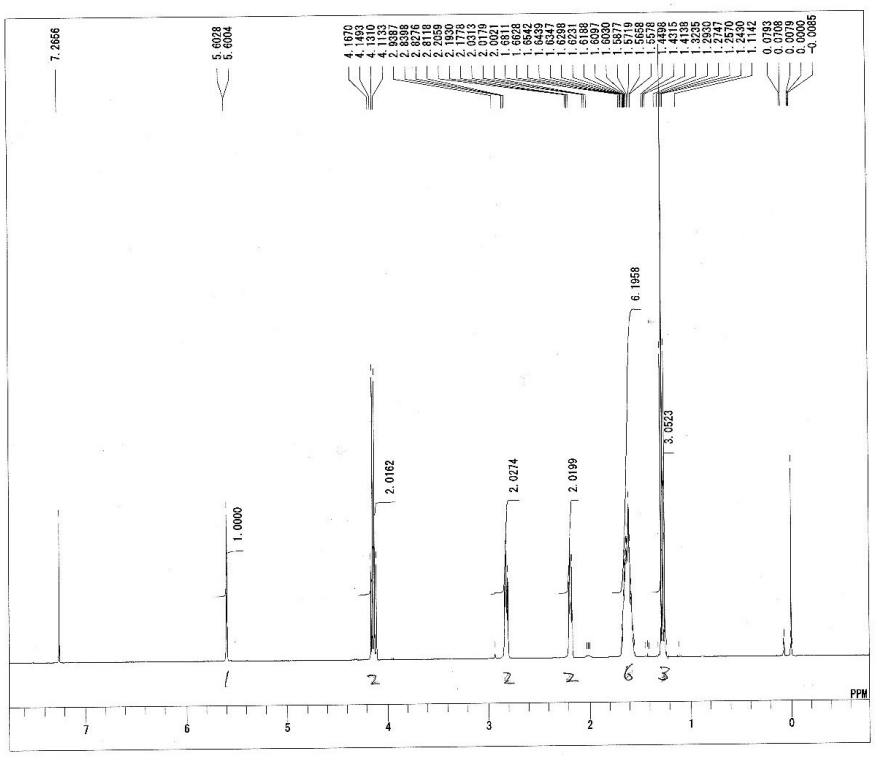
DATIM Thu Jan 28 17:09:38 2010
EXMOD SINGL
IRMOD NON
OBNUC 1H
OBFRQ 399.65 MHz
OBSET 0.00 KHz
PW1 6.50 usec
POINT 32768
SAMPO 32768
TIMES 8
SCANS 8
DUMMY 4
FREQU 7993.60 Hz
FILTR 4000 Hz
ACQTM 4.0993 sec
DEADT 71.75 usec
DELAY 50.00 usec
PD 1.9040 sec
ADBIT 16
RGAIN 16
IRNUC 1H
IRFRQ 399.65 MHz
IRSET 0.00 KHz
IRRPW 50 usec
IRATN 511
IRRNS 0
IRBP1 30
IRBP2 6
BF 0.10 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
XE 3358.40 Hz
XS 515.21 Hz
REFVL
DFILE kyo-242-2.als
MENUF A1H
SHMFL TH5AT
LKSET 0.00 KHz
LKLEV 200
LGAIN 22
LKPHS 41
LKSIG 764
CTEMP 18.9 c
CSPED 21 Hz
FIELD 4186



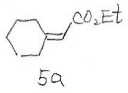
E:Z>99:1


kyo-120-2
7.3699
7.3516
7.3498
7.3443
7.3384
7.3330
7.3046
7.2992
7.2833
7.2658
6.9146
6.9044
6.8806
6.8653
6.0440
6.0403
6.0043
6.0013
5.8603
5.8341
4.5917
4.5618
4.4489
4.4160
4.2433
4.2256
4.2079
4.1895
4.1765
4.1329
4.1200
4.1170
4.1042
4.1011
1.5584
1.3375
1.3253
1.3211
1.3076
1.2900
1.2759
0.0720
0.0000
5.4681
0.9927
1.0000
0.0211
2.0328
3.0269
6.0733
5
E
Z
E
Z
2
2 1
6
PPM
8
6
4
2
0
DATIM Mon Feb 09 09:36:45 2009
EXMOD SINGL
IRMOD NON
OBNUC 1H
OBFRQ 399.65 MHz
OBSET 0.00 KHz
PW1 6.50 usec
POINT 32768
SAMPO 32768
TIMES 8
SCANS 8
DUMMY 4
FREQU 7993.60 Hz
FILTR 4000 Hz
ACQTM 4.0993 sec
DEADT 71.75 usec
DELAY 50.00 usec
PD 1.9040 sec
ADBIT 16
RGAIN 18
IRNUC 1H
IRFRQ 399.65 MHz
IRSET 0.00 KHz
IRPPW 50 usec
IRATN 511
IRRNS 0
IRBP1 30
IRBP2 6
BF 0.10 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
XE 3598.20 Hz
XS 393.00 Hz
REFVL
DFILE kyo-120-2.als
MENUF A1H
SHMFL TH5AT
LKSET 0.00 KHz
LKLEV 200
LGAIN 22
LKPHS 41
LKSIG 811
CTEMP 20.6 c
CSPED 17 Hz
FIELD 3699
H
CO2Et
CH3
H
OCH2Ph
3k
E : Z = 98 : 2

kyo-237-2

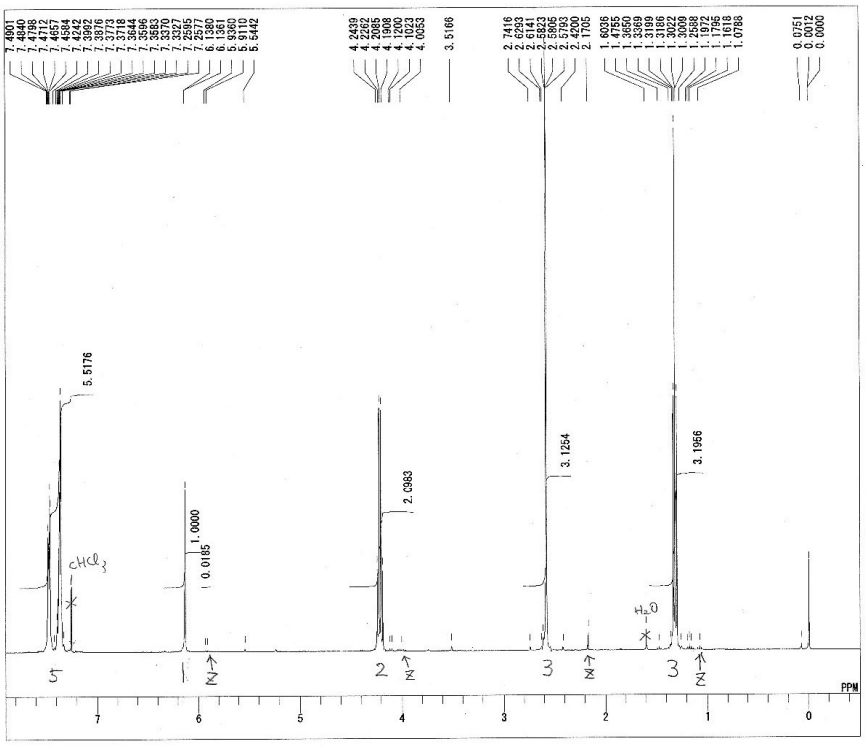



```
DATIM   Tue Jan 12 12:45:47 2010
EXMOD   SINGL
IRMOD   NON
OBNUC   1H
OBFRQ       399.65 MHz
OBSET         0.00 KHz
PW1           6.50 usec
POINT        32768
SAMPO        32768
TIMES            8
SCANS            8
DUMMY            4
FREQU      7993.60 Hz
FILTR      4000 Hz
ACQTM         4.0993 sec
DEADT        71.75 usec
DELAY        50.00 usec
PD            1.9040 sec
ADBIT           16
RGAIN           16
IRNUC   1H
IRFRQ       399.65 MHz
IRSET         0.00 KHz
IRRPW           50 usec
IRATN          511
IRRNS            0
IRBP1           30
IRBP2            6
BF            0.10 Hz
T1            0.00
T2            0.00
T3           90.00
T4          100.00
XE         3411.58 Hz
XS          597.18 Hz
REFVL
DFILE   kyo-237-2.als
MENUF   AIH
SHMFL   TH5AT
LKSET         0.00 KHz
LKLEV          200
LGAIN           22
LKPHS           41
LKSIG          751
CTEMP        18.0 c
CSPED          21 Hz
FIELD        4159
```

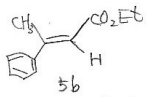


5a

acet



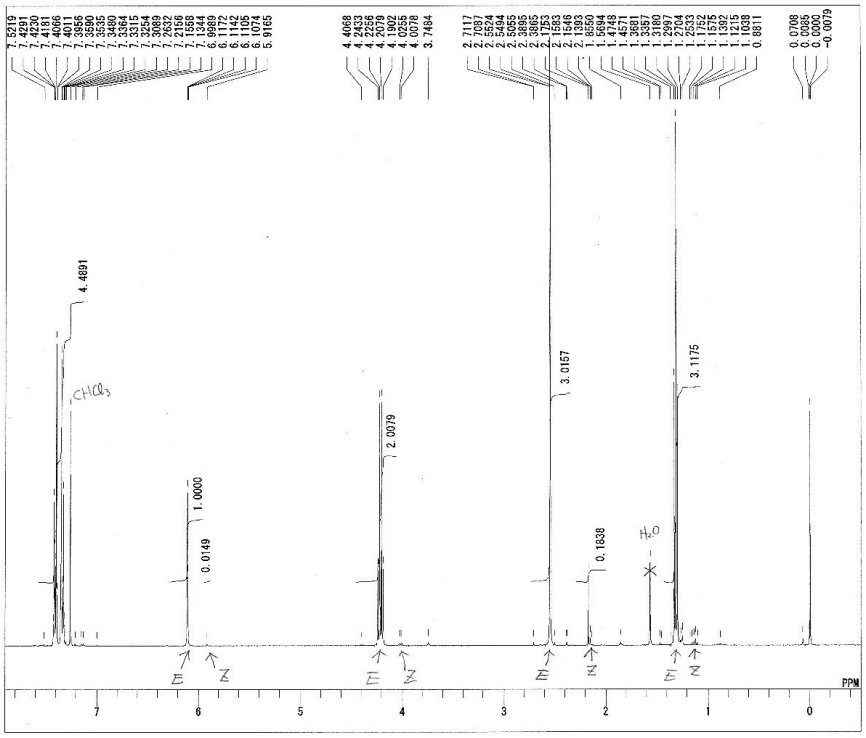

```
DATIM   Sun Jan 23 09:16:35 2011
EXMOD   SINGL
IRMOD   NON
OBNUC   1H
OBFRQ       399.65 MHz
OBSET         0.00 KHz
PW1           6.50 usec
POINT        32768
SAMPO        32768
TIMES            8
SCANS            8
DUMMY            4
FREQU      7993.60 Hz
FILTR      4000 Hz
ACQTM         4.0993 sec
DEADT        71.75 usec
DELAY        50.00 usec
PD            1.9040 sec
ADBIT           16
RGAIN           15
IRNUC   1H
IRFRQ       399.65 MHz
IRSET         0.00 KHz
IRRPW           50 usec
IRATN          511
IRRNS            0
IRBP1           30
IRBP2            6
BF            0.10 Hz
T1            0.00
T2            0.00
T3           90.00
T4          100.00
XE         3358.40 Hz
XS          513.02 Hz
REFVL
DFILE   _DEFAULT.ALS
MENUF   AIH
SHMFL   TH5AT
LKSET         0.00 KHz
LKLEV          200
LGAIN           22
LKPHS           20
LKSIG          639
CTEMP        17.8 c
CSPED          17 Hz
FIELD        4672
```



5b

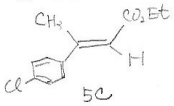
E:Z = 99:1

kyo-340 P-TLC




```
DATIM  Sat Jan 22 09:24:30 2011
EXMOD  SINGL
IRMOD  NON
OBNUC  1H
OBFRQ        399.65 MHz
OBSET          0.00 KHz
PW1            6.50 usec
POINT         32768
SAMPO         32768
TIMES             8
SCANS             8
DUMMY             4
FREQU       7993.60 Hz
FILTR       4000 Hz
ACQTM          4.0993 sec
DEADT         71.75 usec
DELAY         50.00 usec
PD             1.9040 sec
ADBIT            16
RGAIN            19
IRNUC  1H
IRFRQ        399.65 MHz
IRSET          0.00 KHz
IRRPW           50 usec
IRATN           511
IRRNS             0
IRBP1            30
IRBP2             6
BF             0.10 Hz
T1             0.00
T2             0.00
T3            90.00
T4           100.00
XE          3358.40 Hz
XS           514.48 Hz
REFVL
DFILE  _DEFAULT.ALS
MENUF  A1H
SHMFL  TH5AT
LKSET          0.00 KHz
LKLEV           200
LGAIN            22
LKPHS            20
LKSIG           742
CTEMP         18.3 c
CSPED           16 Hz
FIELD         4676
```

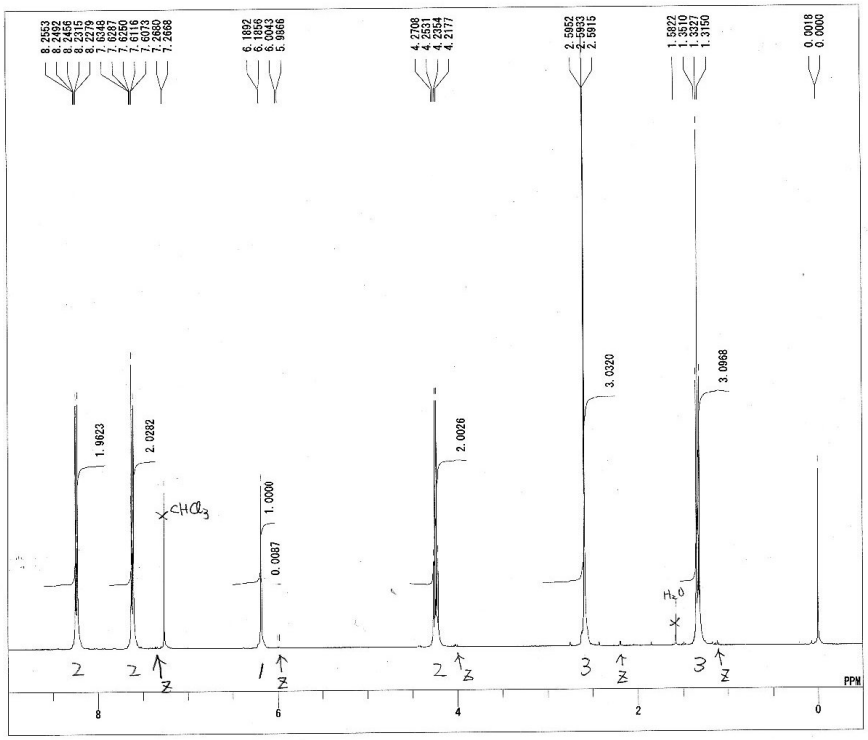
The mixture (E:Z=95:5) was purified by p-TLC to give a mixture (E:Z=99:1).



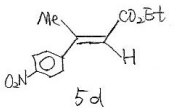
5c

E:Z=99:1

kyo-361-2



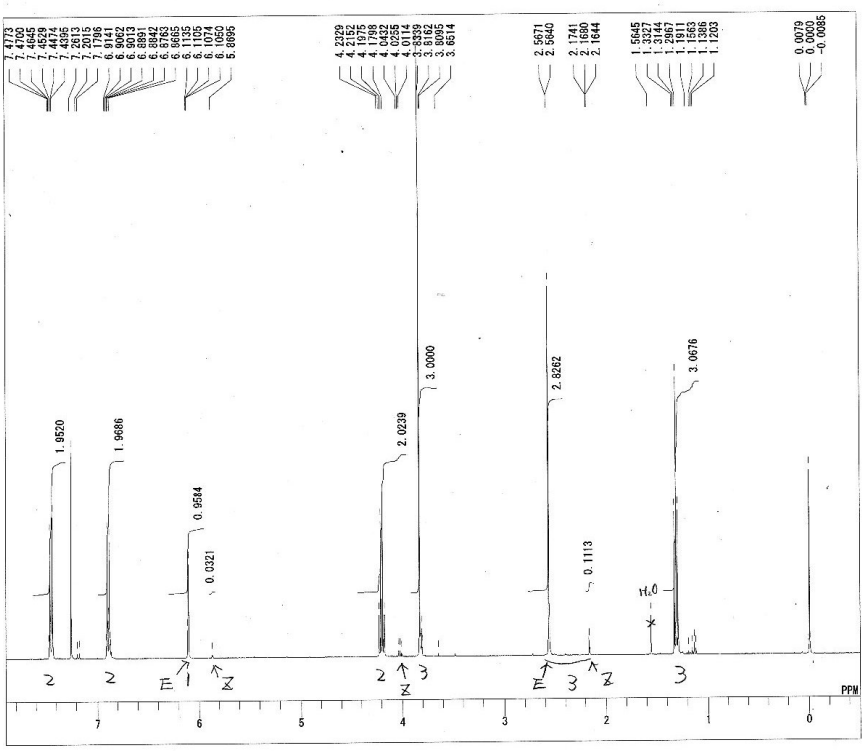

```
DATIM  Wed Dec 15 10:55:59 2010
EXMOD  SINGL
IRMOD  NON
OBNUC  1H
OBFRQ        399.65 MHz
OBSET          0.00 KHz
PW1            6.50 usec
POINT         32768
SAMPO         32768
TIMES             8
SCANS             8
DUMMY             4
FREQU       7993.60 Hz
FILTR       4000 Hz
ACQTM          4.0993 sec
DEADT         71.75 usec
DELAY         50.00 usec
PD             1.9040 sec
ADBIT            16
RGAIN            17
IRNUC  1H
IRFRQ        399.65 MHz
IRSET          0.00 KHz
IRRPW           50 usec
IRATN           511
IRRNS             0
IRBP1            30
IRBP2             6
BF             0.10 Hz
T1             0.00
T2             0.00
T3            90.00
T4           100.00
XE          3797.99 Hz
XS           296.64 Hz
REFVL
DFILE  kyo-361-2.als
MENUF  A1H
SHMFL  TH5AT
LKSET          0.00 KHz
LKLEV           200
LGAIN            22
LKPHS            20
LKSIG           522
CTEMP         18.2 c
CSPED           17 Hz
FIELD         4628
```



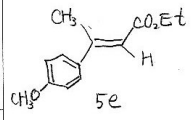
5d

E:Z=99:1

kyo-368-2



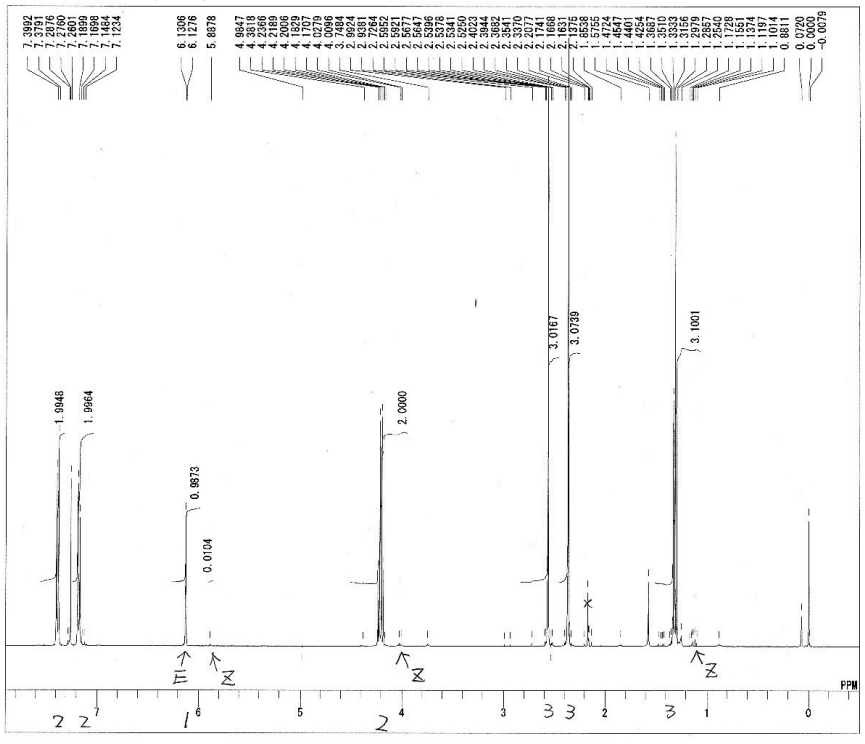
```
DATIM  Thu Jan 20 11:40:22 2011
EXMOD  SINGL
IRMOD  NON
OBNUC  1H
OBFRQ        399.65 MHz
OBSET          0.00 KHz
PW1            6.50 usec
POINT         32768
SAMPO         32768
TIMES             8
SCANS             8
DUMMY             4
FREQU       7993.60 Hz
FILTR       4000 Hz
ACQTM          4.0993 sec
DEADT         71.75 usec
DELAY         50.00 usec
PD             1.9040 sec
ADBIT            16
RGAIN            20
IRNUC  1H
IRFRQ        399.65 MHz
IRSET          0.00 KHz
IRRPW           50 usec
IRATN          511
IRRNS            0
IRBP1           30
IRBP2            6
BF             0.10 Hz
T1             0.00
T2             0.00
T3            90.00
T4           100.00
XE          3358.40 Hz
XS           513.75 Hz
REFVL
DFILE  kyo-368-2 re.als
MENUF  A1H
SHMFL  TH5AT
LKSET          0.00 KHz
LKLEV           200
LGAIN            22
LKPHS            20
LKSIG           719
CTEMP         18.2 c
CSPED           20 Hz
FIELD         4674
```



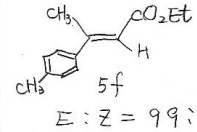
5e

E : Z = 97 : 3

369-2 P-TLC



```
DATIM  Tue Jan 25 15:00:22 2011
EXMOD  SINGL
IRMOD  NON
OBNUC  1H
OBFRQ        399.65 MHz
OBSET          0.00 KHz
PW1            6.50 usec
POINT         32768
SAMPO         32768
TIMES             8
SCANS             8
DUMMY             4
FREQU       7993.60 Hz
FILTR       4000 Hz
ACQTM          4.0993 sec
DEADT         71.75 usec
DELAY         50.00 usec
PD             1.9040 sec
ADBIT            16
RGAIN            17
IRNUC  1H
IRFRQ        399.65 MHz
IRSET          0.00 KHz
IRRPW           50 usec
IRATN          511
IRRNS            0
IRBP1           30
IRBP2            6
BF             0.10 Hz
T1             0.00
T2             0.00
T3            90.00
T4           100.00
XE          3358.40 Hz
XS           513.51 Hz
REFVL
DFILE  kyo-369-2 P-TLC.als
MENUF  A1H
SHMFL  TH5AT
LKSET          0.00 KHz
LKLEV           200
LGAIN            22
LKPHS            20
LKSIG           801
CTEMP         18.1 c
CSPED           12 Hz
FIELD         4680
```

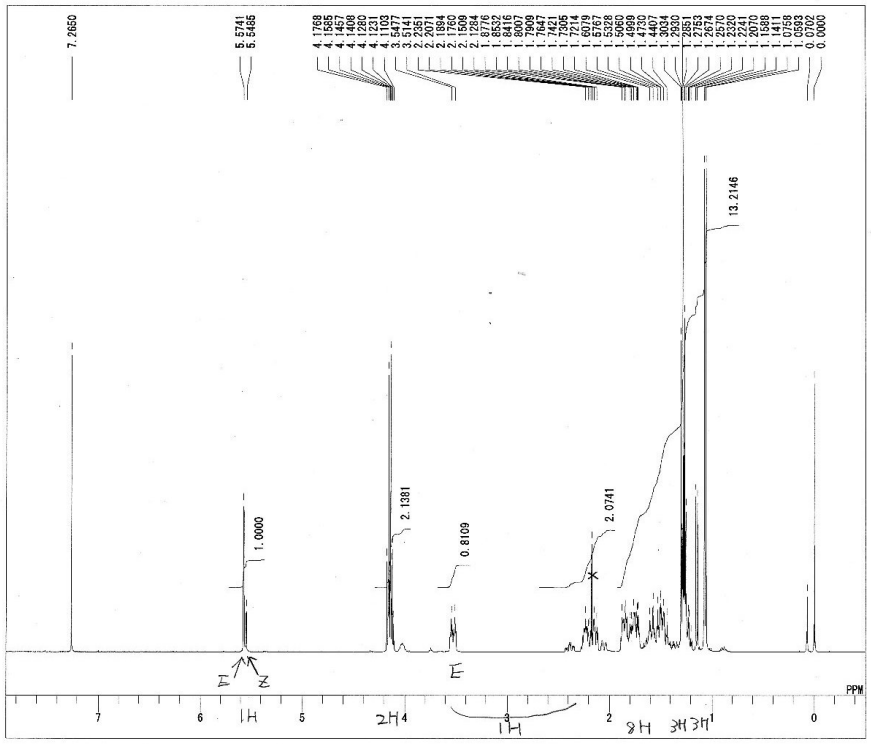


5f

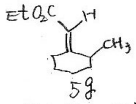
E : Z = 99 : 1

The mixture (E:Z=97:3) was purified by p-TLC to give a mixture (E:Z=99:1).

kyo-336-2 P-TLC



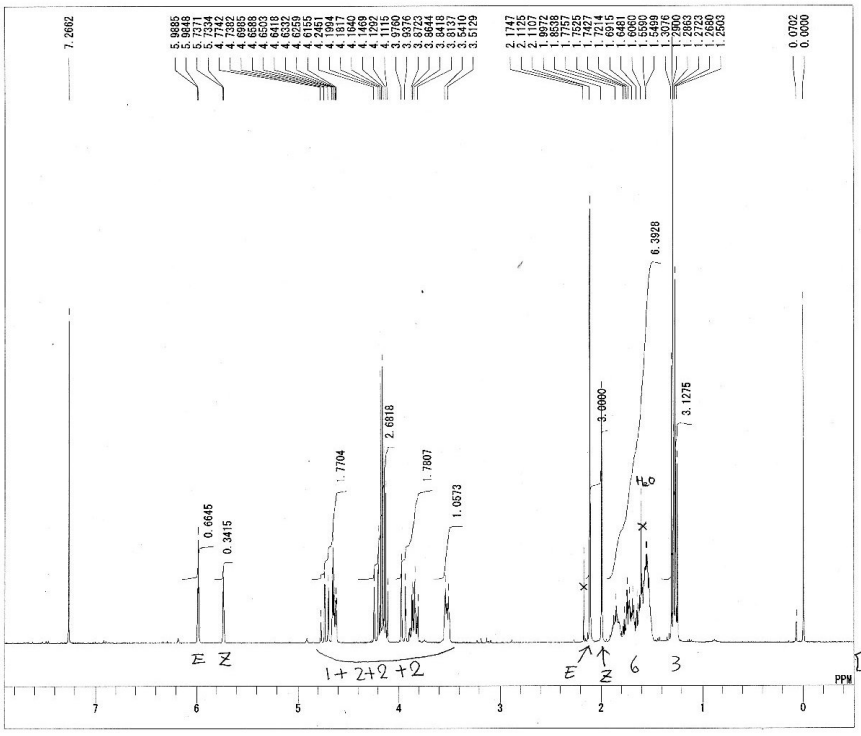

```
DATIM   Tue Jan 25 17:05:51 2011
EXMOD   SINGL
IRMOD   NON
OBNUC   1H
OBFRQ       399.65 MHz
OBSET         0.00 KHz
PW1           6.50 usec
POINT        32768
SAMPO        32768
TIMES            8
SCANS            8
DUMMY            4
FREQU       7993.60 Hz
FILTR       4000 Hz
ACQTM          4.0993 sec
DEADT         71.75 usec
DELAY         50.00 usec
PD             1.9040 sec
ADBIT           16
RGAIN           17
IRNUC   1H
IRFRQ       399.65 MHz
IRSET         0.00 KHz
IRRPW           50 usec
IRATN          511
IRRNS            0
IRBP1           30
IRBP2            6
BF            0.10 Hz
T1            0.00
T2            0.00
T3           90.00
T4          100.00
XE         3358.40 Hz
XS          515.21 Hz
REFVL
DFILE   kyo-336-2 P-TLC.als
MENUF   A1H
SHMFL   TH5AT
LKSET         0.00 KHz
LKLEV          200
LGAIN           22
LKPHS           20
LKSIG          759
CTEMP        18.2 c
CSPED          17 Hz
FIELD        4686
```



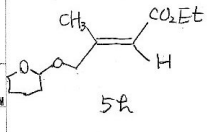
5g

E : Z = 81 : 19

kyo-298-2 P-TLC



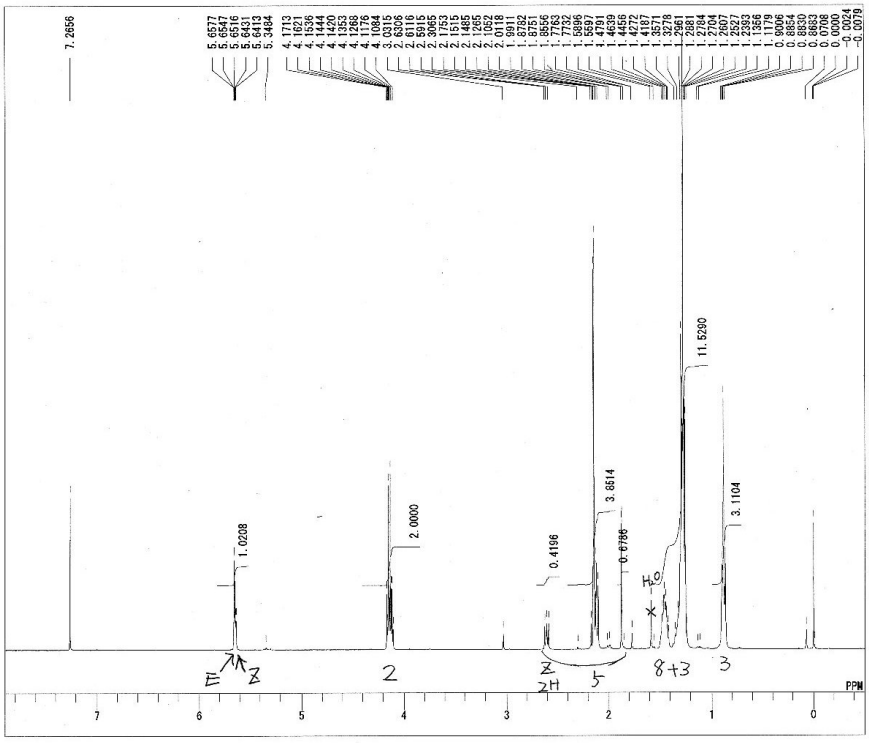

```
DATIM   Wed Jan 26 09:46:41 2011
EXMOD   SINGL
IRMOD   NON
OBNUC   1H
OBFRQ       399.65 MHz
OBSET         0.00 KHz
PW1           6.50 usec
POINT        32768
SAMPO        32768
TIMES            8
SCANS            8
DUMMY            4
FREQU       7993.60 Hz
FILTR       4000 Hz
ACQTM          4.0993 sec
DEADT         71.75 usec
DELAY         50.00 usec
PD             1.9040 sec
ADBIT           16
RGAIN           15
IRNUC   1H
IRFRQ       399.65 MHz
IRSET         0.00 KHz
IRRPW           50 usec
IRATN          511
IRRNS            0
IRBP1           30
IRBP2            6
BF            0.10 Hz
T1            0.00
T2            0.00
T3           90.00
T4          100.00
XE         3358.40 Hz
XS          515.94 Hz
REFVL
DFILE   kyo-298-2 P-TLC.als
MENUF   A1H
SHMFL   TH5AT
LKSET         0.00 KHz
LKLEV          200
LGAIN           22
LKPHS           20
LKSIG          775
CTEMP        18.8 c
CSPED          16 Hz
FIELD        4678
```



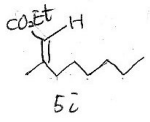
5h

E : Z = 66 : 34

kyo-371-2 P-TLC




```
DATIM  Thu Jan 27 10:37:07 2011
EXMOD  SINGL
IRMOD  NON
OBNUC  1H
OBFRQ        399.65 MHz
OBSET          0.00 KHz
PW1            6.50 usec
POINT         32768
SAMPO         32768
TIMES             8
SCANS             8
DUMMY             4
FREQU       7993.60 Hz
FILTR       4000 Hz
ACQTM          4.0993 sec
DEADT         71.75 usec
DELAY         50.00 usec
PD             1.9040 sec
ADBIT            16
RGAIN            14
IRNUC  1H
IRFRQ        399.65 MHz
IRSET          0.00 KHz
IRRPW           50 usec
IRATN          511
IRRNS            0
IRBP1           30
IRBP2            6
BF             0.10 Hz
T1             0.00
T2             0.00
T3            90.00
T4           100.00
XE          3358.40 Hz
XS           515.70 Hz
REFVL
DFILE  371-2 P-TLC.als
MENUF  A1H
SHMFL  TH5AT
LKSET          0.00 KHz
LKLEV           200
LGAIN            22
LKPHS            20
LKSIG           715
GTEMP         18.2 c
GSPED           12 Hz
FIELD         4684
```



5i

E:Z=79:21