# Supporting Information

# Nano-$K_2CO_3$: preparation, characterization and evaluation of reactive activities

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## Table of Contents

## 1. General experimental

All commercially reagents were used without further purification. $^{1}H$ NMR and $^{13}C$ NMR spectra were recorded with a Bruker Advance II 500 instrument at 500 and 126 MHz, respectively. Chemical shifts were given as $\delta$ values (ppm), with tetramethylsilane as internal standard. Coupling constants ($J$) were given in Hertz (Hz). The particle size of nano-$K_2CO_3$ was measured using a laser particle size analyzer (Zetasizer Nano S90, Malvern Instruments Ltd.). $CO_2$-TPD was measured with Chemisorb 2720 automatic chemical adsorption apparatus (Micromeritics Instrument Corp). Nano-$K_2CO_3$ was prepared using GZM-5 High Frequency Resonant Grinding Machine (Beijing More Open Source Technology Development Ltd., Beijing, China) (47.8 Hz) and was observed by scanning electronic microscopy (SEM) performed on a LEO 1530VP instrument.

## 2. Preparation of nano-$K_2CO_3$

Anhydrous $K_2CO_3$ (150 g), absolute ethanol (63 mL), and lauric acid (0.435 g) were poured into a resonance mill. The mixture was milled at room temperature for 8 h and then directly used for the next reaction.

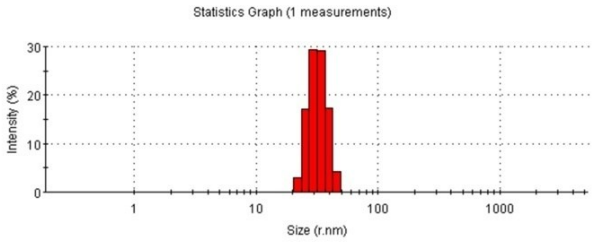
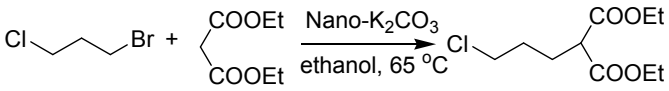



**Fig. 1** Particle size distribution of nano-$K_2CO_3$

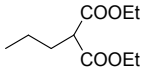
## 3. Typical procedure for the alkylation of active methylene compounds




**Diethyl 2-(3-chloropropyl)malonate** Nano-$K_2CO_3$ (1.3 mol, 179.7 g) and a solution of diethyl malonate (1.0 mol, 160.2 g) and 1-bromo-3-chloropropane (1.1 mol, 173.2 g) in absolute ethanol(500 mL) was added to a round-bottomed flask provided with a water-cooled reflux condenser and a thermometer. The mixture was heated to 65 $^{o}$C on oil bath and stirred for 8 h. The reaction was monitored by GC. The mixture was filtered and distilled to collect the product 205.9 g, yield 87.5%, bp 156-158 $^{o}$C/16 mmHg; $^{1}H$ NMR($CD_3OD$, 500MHz): δ 1.29 (t, $J$ = 7.0 Hz, 6H), 1.81 ~ 1.87 (m, 2H), 2.01~ 2.06(m, 2H), 3.48 (t, $J$ = 7.5 Hz, 1H), 3.63(t, $J$ = 6.5 Hz, 2H), 4.19 ~ 4.26(m, 4H); $^{13}C$ NMR($CD_3OD$, 125 MHz) : δ 14.5, 27.2, 31.3, 45.2, 49.3, 62.5, 170.7.

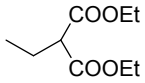
The other monoalkylated products were prepared similarly according to the procedure used for Diethyl 2-(3-chloropropyl)malonate.

**Diethyl 2-propylmalonate** bp 109-113 $^{o}$C/16 mmHg; $^{1}H$ NMR($CD_3Cl_3$, 500 MHz): δ 0.94 (t, $J$ = 7.0 Hz, 3H), 1.27 (t, $J$ = 7.0 Hz, 6H), 1.37 ~ 1.38 (m, 2H), 1.85 ~ 1.90 (m, 2H), 3.33 (t, $J$ = 7.5 Hz, 1H), 4.17 ~ 4.21 (q, $J$ = 7.0 Hz, 4H); $^{13}C$ NMR($CDCl_3$, 125 MHz) : δ 13.6, 14.0, 20.5, 30.7, 51.8, 61.2, 169.6.

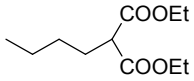



**Diethyl 2-ethylmalonate** bp 101-103 $^{o}$C/16 mmHg; $^{1}H$ NMR (500 MHz, $CDCl_3$): δ 0.97 (t, $J$ = 7.5 Hz, 3H),

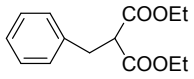
1.27 (t, *J* = 7.0 Hz, 3H), 1.90 ~ 1.96(m, 2H), 3.25(t, *J* = 7.0 Hz, 1H) 4.17 ~ 4.22(m, 4H). $^{13}$C NMR (125 MHz, $CDCl_3$): δ 14.0, 30.5, 61.9, 126.7, 128.5, 129.2 ,135.7, 150.7,162.2.



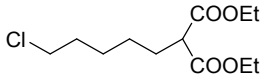

**Diethyl 2-butylmalonate** bp 121-124 °C/16 mmHg; $^1$H NMR($CD_3Cl_3$, 500MHz): δ 0.90 (t, *J* = 7.5 Hz, 3H), 1.27(t, *J* = 7.0 Hz, 6H), 1.29 ~ 1.35 (m, 4H), 1.87 ~ 1.92(m, 2H), 3.31(t, *J* = 7.5 Hz, 1H), 4.16 ~ 4.23(m, 4H); $^{13}$C NMR($CDCl_3$, 125 MHz): δ 13.8, 14.1, 22.3, 28.4, 29.4, 52.1, 61.2, 169.2.



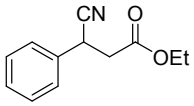

**Diethyl 2-benzylmalonate** bp 170-172 °C/16 mmHg; $^1$H NMR($CDCl_3$, 500MHz): δ 1.19 (t, *J* = 7.5 Hz, 6H), 3.21 (d, *J* = 4.0Hz, 2H), 3.64 (t, *J* = 8.0 Hz, 1H), 4.12 ~ 4.20 (m, 4H), 7.18 ~ 7.21(m, 3H), 7.25 ~ 7.28(m, 2H); $^{13}$C NMR($CDCl_3$, 125 MHz): δ 13.9, 34.7, 53.8, 61.4, 126.7, 128.5, 128.8, 137.9, 168.8.



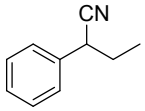

**Diethyl 2-(5-chloropentyl)malonate** bp 160-162 °C/16 mmHg; $^1$H NMR($CDCl_3$, 500MHz): δ 1.26 (t, *J* = 7.0 Hz, 6H), 1.33 ~ 1.37 (m, 2H), 1.45 ~ 1.50 (m, 2H), 1.75 ~ 1.81(m, 2H), 1.88 ~ 1.93(m, 2H), 3.32 (t, *J* = 7.5 Hz, 1H), 3.53(t, *J* = 7.0 Hz, 2H), 4.18 ~ 4.22(m, 4H); $^{13}$C NMR($CDCl_3$, 125 MHz): δ 14.5, 26.4, 26.5, 28.4, 32.2, 44.8, 51.9, 61.3, 169.4.



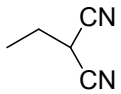

**Ethyl 3-cyano-3-phenylpropanoate** bp 178-181°C/16 mmHg; $^1$H NMR ($CDCl_3$, 500 MHz): δ 1.19(t, *J* = 7.5 Hz, 3H), 2.35 ~ 2.65 (m, 4H), 3.37 ~ 3.64 (m, 1H), 4.12 ~ 4.19 (d, *J* = 7.5 Hz, 2H), 7.06 ~ 7.269(m, 5H); $^{13}$C NMR($CD_3OD$, 125MHz): δ 13.9, 36.9, 40.1, 61.715, 117.6, 126.7, 128.4, 128.8, 137.9, 169.5.



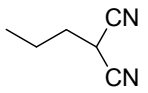

**2-Phenylbutanenitrile** bp 124-126 °C /16 mmHg; $^1$H NMR ($CDCl_3$, 500 MHz): δ 1.08 (t, *J* = 7.5 Hz, 3H), 1.90 ~ 2.02 (m, 2H), 3.74 (t, *J* = 7.0 Hz, 1H), 7.42 ~ 7..29 (m, 5H); $^{13}$C NMR (CDCl3, 125 MHz): δ 11.5, 29.0, 38.9, 120.7, 127.3, 127.9, 135.0.



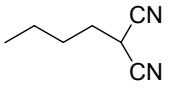

**2-Ethylmalononitrile** bp 109-112 °C/16 mmHg; $^1$H NMR($CD_3OD$, 500MHz): δ 0.98 (t, *J* = 7.0 Hz, 3H), 1.98 ~ 2.06 (m, 2H), 3.35(t, *J* = 8.0 Hz, 1H); $^{13}$C NMR($CD_3OD$, 125 MHz): δ 13.3, 18.2, 28.9, 117.6.



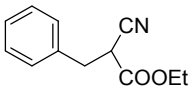

**2-Propylmalononitrile** bp 123-125℃/16 mmHg; $^1$H NMR($CD_3OD$, 500MHz): δ 0.99 (t, *J* = 7.5Hz, 3H), 1.35(m, 2H), 1.93 ~ 2.01(m, 2H), 3.39(t, *J* = 8.0Hz, 1H); $^{13}$C NMR($CD_3OD$, 125MHz): δ 13.4, 14.3, 18.3, 28.9, 117.7.



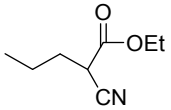
**2-Butylmalononitrile** bp 129-132 °C/16 mmHg; $^1$H NMR($CD_3OD$, 500MHz): δ 0.98 (t, *J* = 7.5Hz, 3H), 1.29 ~ 1.35 (m, 4H), 1.93 ~ 2.01(m, 2H), 3.31(t, *J* = 7.5Hz, 1H); $^{13}$C NMR($CD_3OD$, 125MHz): δ 13.5, 14.0, 18.5, 25.9, 27.1, 117.7.



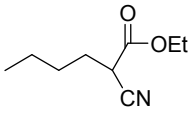

**Ethyl 2-cyano-3-phenylpropanoate** bp 165-173 °C/16 mmHg; $^1$H NMR($CDCl_3$, 500MHz): δ 1.26 (t, *J* = 7.5Hz, 3H), 3.19(dd, *J* = 11.0, 5.0 Hz, 1H), 3.29(dd, *J* = 11.0, 5.0Hz, 1H), 3.71(m, 1H), 4.23(q, *J* = 7.5Hz, 3H); $^{13}$C NMR($CDCl_3$, 125MHz): δ 14.1, 31.2, 36.9, 61.3, 117.6, 126.7, 128.3, 128.8, 136.9, 169.7.



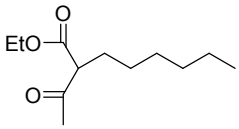

**Ethyl 2-cyanopentanoate** bp 111-113°C/16 mmHg; $^1$H NMR($CD_3OD$, 500MHz): δ 0.92 (t, *J* = 7.0Hz, 3H), 1.31 ~ 1.37(m, 5H), 1.85 ~ 1.90 (m, 2H), 3.33 (t, *J* = 7.5 Hz, 1H), 4.17 ~ 4.22(m, 2H); $^{13}$C NMR($CD_3OD$, 125 MHz): δ 13.6, 14.0, 20.5, 32.7, 34.6, 61.2, 117.7, 169.6.



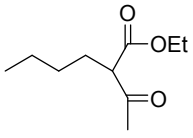

**Ethyl 2-cyanohexanoate** bp 133-135°C/16 mmHg; $^1$H NMR($CD_3OD$, 500MHz): δ 0.91 (t, *J* = 7.5 Hz, 3H), 1.25 ~ 1.69 (m, 7H), 1.89 ~ 2.01 (m, 2H), 3.31 (t, *J* = 7.5 Hz, 1H), 4.16 ~ 4.22 (m, 2H); $^{13}$C NMR($CD_3OD$, 125 MHz): δ 13.7, 14.1, 20.5, 23.9, 28.6, 30.9, 33.5, 61.2, 117.7, 169.6.



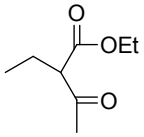

**Ethyl 2-acetyloctanoate** bp 159-162℃/16 mmHg; $^1$H NMR($CD_3OD$, 500MHz): δ 0.91(t, *J* = 7.5 Hz, 3H), 1.25 ~ 1.69 (m, 11H), 1.89 ~ 2.09 (m, 5H), 3.31 (t, *J* = 7.5 Hz, 1H), 4.16 ~ 4.22 (m, 2H); $^{13}$C NMR($CD_3OD$, 125 MHz): δ 12.1, 14.6, 18.2, 20.1, 21.1, 28.7, 29.8, 34.6, 59.7, 61.2, 169.5, 201.3.



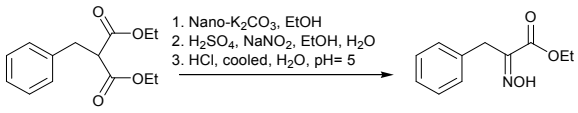

**Ethyl 2-acetylhexanoate** bp 133-135℃/16 mmHg; $^1$H NMR($CD_3OD$, 500MHz): δ 0.91(t, *J* = 7.5 Hz, 3H), 1.25 ~ 1.69 (m, 7H), 1.90 ~ 2.01 (m, 2H), 3.31 (t, *J* = 7.5 Hz, 1H), 4.16 ~ 4.22 (m, 2H); $^{13}$C NMR($CD_3OD$, 125 MHz): δ13.7, 14.1, 20.5, 21.9, 24.5, 30.1, 59.7, 61.2, 169.5, 201.3.




**Ethyl 2-ethyl-3-oxobutanoate** bp 93-96℃/16 mmHg; $^1$H NMR($CD_3OD$, 500MHz): δ 0.93 (t, *J* = 7.5 Hz, 3H), 1.26 (t, *J* = 7.5 Hz, 3H), 1.89 ~ 2.01 (m, 2H), 3.31 (t, *J* = 7.5 Hz, 1H), 4.16 ~ 4.22 (m, 2H); $^{13}$C NMR($CD_3OD$, 125 MHz): δ13.7, 14.0, 19.0, 24.5, 59.1, 61.2, 169.5, 201.3.



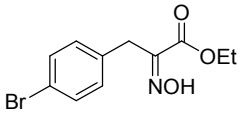
## 4. Typical procedure for the oximation of β-dicarbonyl compounds (Table 4, entry10)



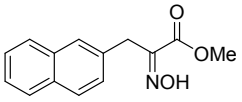

**Ethyl 2-(hydroxyimino)-3-phenylpropanoate** Nano-$K_2CO_3$ (0.5 mol, 69.1 g) was added to a solution of diethyl 2-benzylmalonate (0.2 mmol, 50.1 g) in ethanol (200 mL). Then, the mixture was cooled to 10 °C. Then, a solution of sodium nitrite (0.3 mmol, 20.7 g) in water (100 mL) or ethanol (14 mL) was placed in a 500 mL one-port flask. A solution of sulfuric acid (0.15 mol, 15 g) in water (200 mL) and ethanol (10 mL) was slowly added dropwise to generate ethyl nitrite. Ethyl nitrite was introduced into the reactor through a drying tube. Stirring was maintained for 5 h at a low temperature after adding the sulfuric acid solution. The reaction mixture was filtered and then concentrated to remove ethanol. Cold water (30 mL) was added to the residue, and solution pH was adjusted to 5 with cold hydrochloric acid (0.5 M). The solution was extracted with ethyl acetate (3 × 50 mL), and the organic phase was dried with anhydrous $MgSO_4$. Ethyl acetate was removed under reduced pressure to yield crude prduct. Pure product was obtained by recrystallization using ethyl acetate and hexane. Yield: 39.2 g, yellow solid (94.5%). mp 56 °C; $^1$H NMR (500 MHz, $CDCl_3$): δ 1.34 (t, *J* = 7.0Hz, 3H), 4.00 (s, 2H), 4.30 (q, *J* = 7.0 Hz, 2H), 7.22~7.35 (m, 5H), 9.66 (br, 1H); $^{13}$C NMR (125 MHz, $CDCl_3$): δ 14.0, 30.5, 61.9, 126.7, 128.5, 129.2 , 135.7, 150.7, 162.2.

The other oximes were prepared similarly according to the procedure used for Ethyl 2-(hydroxyimino)-3-phenylpropanoate

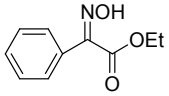
**Ethyl 3-(4-bromophenyl)-2-(hydroxyimino)propanoate** mp 96-98 °C; $^1$H NMR (500 MHz, $CDCl_3$): δ 1.31 (t, *J* = 7.0 Hz, 3H) , 3.92 (s,2H), 4.28 (q, *J* = 7.0 Hz, 2H),7.21 (d, *J* = 8.0 Hz, 2H),7.38 (d, *J* = 8.0 Hz, 2H), 10.30 (br, 1H); $^{13}$CNMR (125 MHz,$CDCl_3$): δ 14.0, 29.9, 62.1, 120.6, 130.9, 131.6, 134.7,150.3, 163.1.



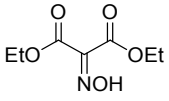

**Methyl 2-(hydroxyimino)-3-(naphthalen-2-yl)propanoate** $^1$H NMR (500 MHz, $CDCl_3$): δ 3.69 (s, 3H),4.04 (s, 2H),7.29～7.34 (m, 3H), 7.62～7.66 (m, 4H),10.36 (br, 1H); $^{13}$C NMR (125 MHz, $CDCl_3$): δ 30.8, 52.9, 125.7, 126.1, 127.5, 127.6, 127.7, 128.3, 132.4, 133.1, 133.6, 150.8, 163.8.




**Ethyl 2-(hydroxyimino)-2-phenylacetate** mp 102-104 °C; $^1$H NMR (500 MHz, MeOD): δ 1.30 (t, *J* = 7.0 Hz, 3H),4.29 (q, *J* = 7.0 Hz, 2H),7.37～7.43 (m, 5H); $^{13}$C NMR (125 MHz, MeOD): δ 12.9, 61.4, 127.4, 128.8, 128.9, 129.8, 149.2, 164.4.

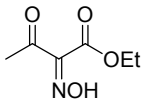



**Diethyl 2-(hydroxyimino)malonate** $^1$H NMR (500 MHz, MeOD): δ 1.37 (q, 6H),4.31 (m, 4H); $^{13}$C NMR (125 MHz, $CDCl_3$): δ 12.9, 13.0, 61.5, 61.7, 143.3, 159.0, 159.6; HRMS (ESI) calculated for $C_7H_{11}NO_5$ $[M+H]^+$ 190.0637, found: 190.0731.

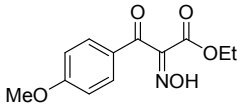



**Ethyl 2-(hydroxyimino)-3-oxobutanoate** $^1$H NMR (500 MHz, MeOD): δ 1.30 (t, *J* = 7.0 Hz, 3H) ,2.35 (s, 3H),

4.30 (q, *J* = 7.0 Hz, 2H); $^{13}$C NMR (125 MHz, MeOD): δ 13.1, 23.8, 61.5, 150.9, 162.3, 194.2 ; HRMS (ESI) calculated for $C_6H_9NO_4$ $[M+H]^+$ 160.0536, found: 160.0609.




**Ethyl 2-(Hydroxyimino)-3- (4-methoxy phenyl)-3-oxopropanoate** mp 112-114 °C; $^1$H NMR (500 MHz, $CDCl_3$): δ 1.26 (t, *J* = 7.0 Hz, 3H), 3.89 (s, 3H), 4.31 (q, *J* = 7.0 Hz, 2H), 6.98 (d, *J* = 8.5 Hz, 2H), 7.85 (d, *J* = 8.5 Hz, 2H), 9.52 (br, 1H); $^{13}$C NMR (125 MHz, $CDCl_3$): δ 14.0, 55.8, 62.7, 114.5, 127.5, 131.9, 149.8, 161.0, 165.1, 188.4.

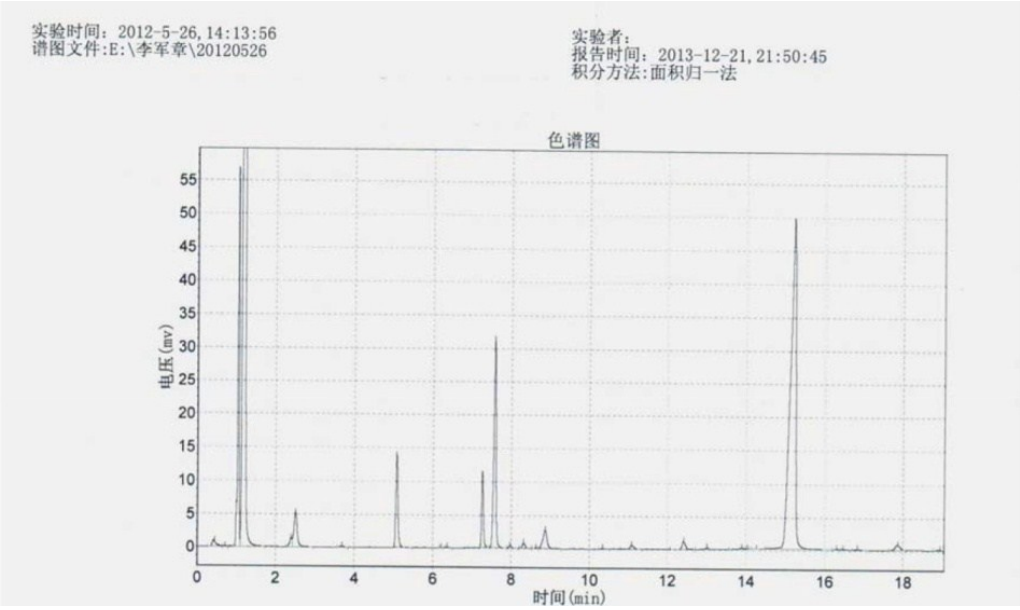



## 5. Regeneration of nano-$K_2CO_3$

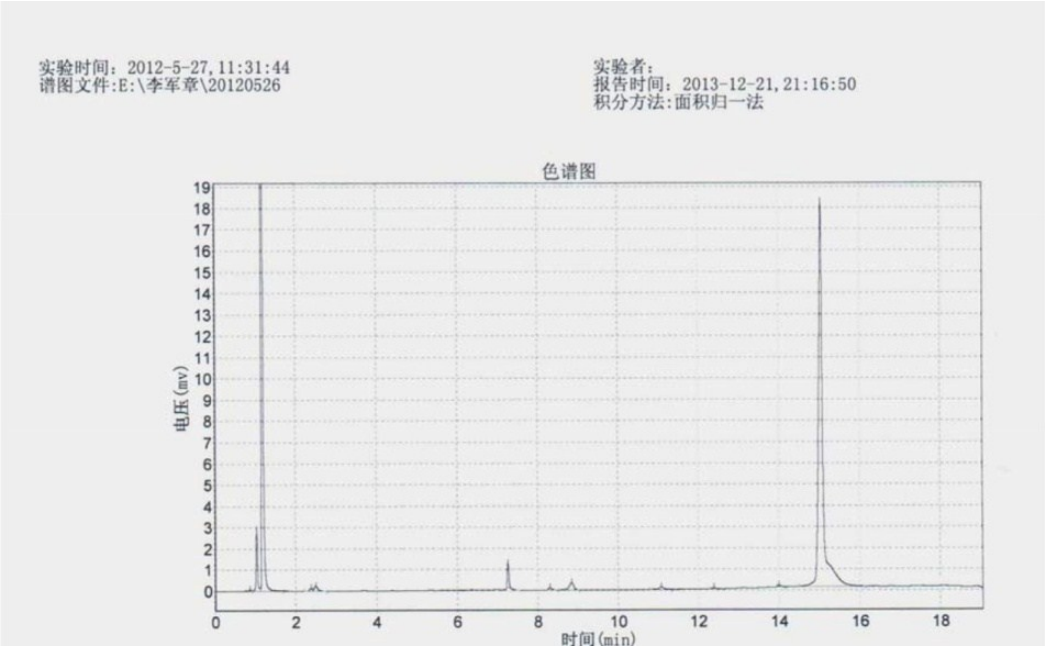
After oximation reaction completion, the mixture were filtered and washed with ethanol(3 × 30 mL). The filter was calcined in muffle at 250℃ for 4 hours to generate normal $K_2CO_3$ with ≥95% yield. The normal $K_2CO_3$ was milled as the procedure of preparation of nano-$K_2CO_3$ to generate product with average particle size of 64 nm.

## 6. Copies of GC spectra, $^{1}H$ and $^{13}C$ NMR spectra of typical compounds

**GC spectra of the reaction mixture of 1-bromo-3-chloropropane with diethyl malonate**



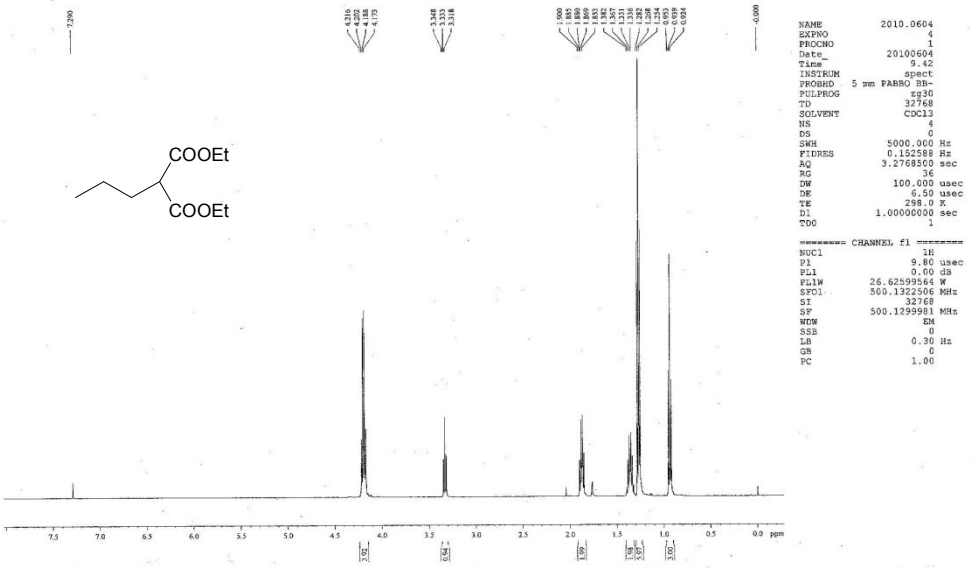

GC spectrum of the mixture after reacting for 4 h




GC spectrum of the mixture after reacting for 8 h

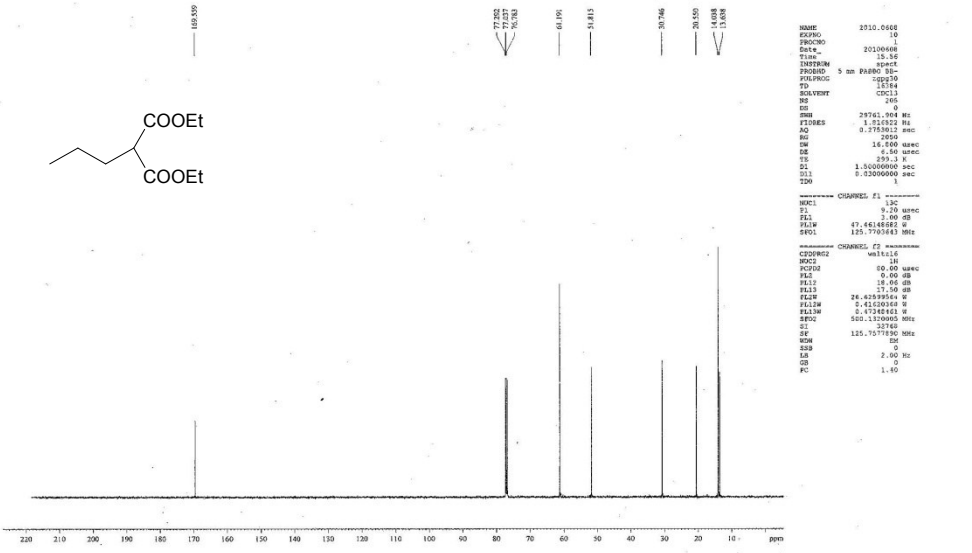
**$^{1}H$ and $^{13}C$ spectra of monoalkylated products**

liushouxin-LCH



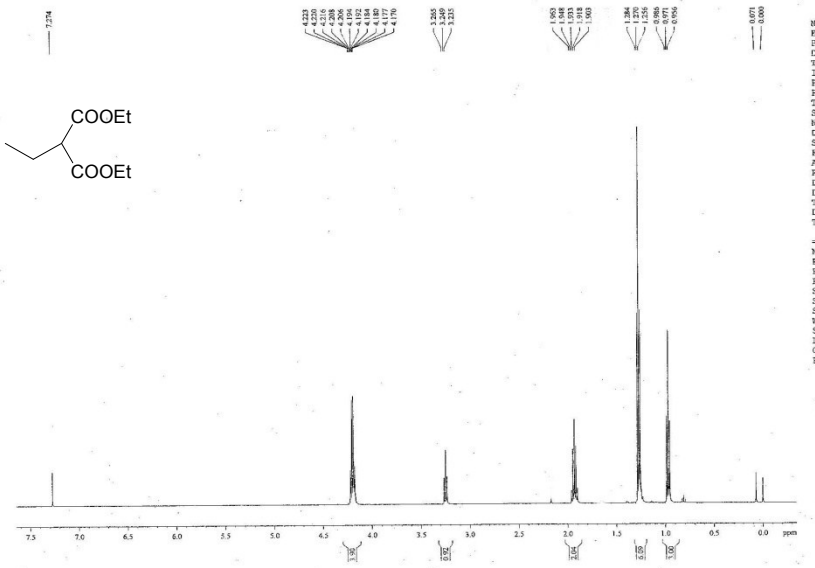

```
NAME                 2010.0604
EXPNO                       4
PROCNO                      1
Date_                20100604
Time                     9.42
INSTRUM                 spect
PROBHD  5 mm PABBO BB-
PULPROG                 zg30
TD                      32768
SOLVENT                 CDCl3
NS                          4
DS                          0
SWH                5000.000 Hz
FIDRES                0.152588 Hz
AQ                 3.27680500 sec
RG                         36
DW                   100.000 usec
DE                     6.50 usec
TE                    298.0 K
D1                 1.00000000 sec
TD0                         1

======== CHANNEL f1 ========
NUC1                       1H
P1                       9.80 usec
PL1                      0.00 dB
PL1W               26.62599564 W
SFO1              500.1322506 MHz
SI                      32768
SF                500.1299981 MHz
WDW                        EM
SSB                         0
LB                       0.30 Hz
GB                          0
PC                       1.00
```

liushouxin-lch



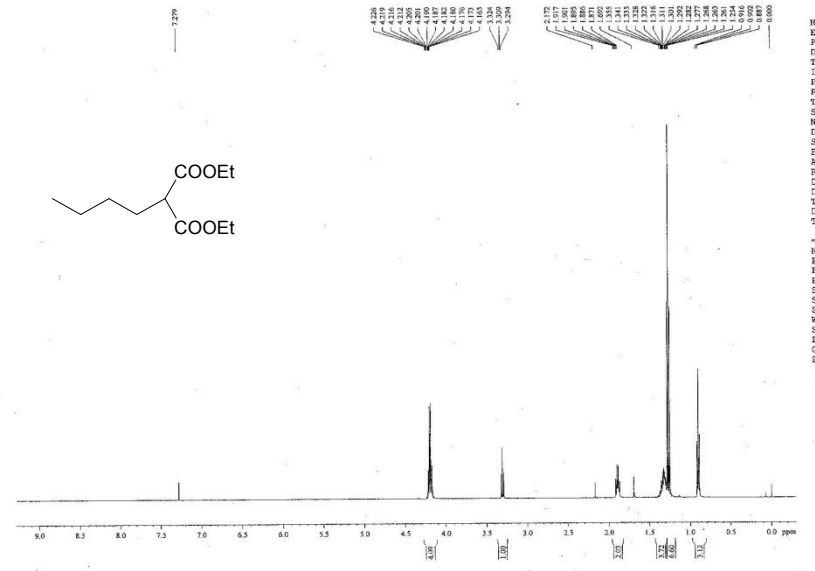

```
NAME                 2010.0608
EXPNO                      10
PROCNO                      1
Date_                20100608
Time                    15.56
INSTRUM                 spect
PROBHD  5 mm PABBO BB-
PULPROG              zgpg30
TD                      65536
SOLVENT                 CDCl3
NS                        205
DS                          0
SWH                29761.904 Hz
FIDRES                1.816822 Hz
AQ                 0.77539012 sec
RG                       2050
DW                     16.800 usec
DE                      6.50 usec
TE                    299.3 K
D1                 1.50000000 sec
D11                0.03000000 sec
TD0                         1

======== CHANNEL f1 ========
NUC1                       13C
P1                       9.20 usec
PL1                      3.00 dB
PL1W               47.66148682 W
SFO1              125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                   80.00 usec
PL2                      0.00 dB
PL12                    18.06 dB
PL13                    17.50 dB
PL2W               26.62599564 W
PL12W              0.41620168 W
PL13W              0.47348161 W
SFO2              500.1320005 MHz
SI                      32768
SF                125.7577890 MHz
WDW                        EM
SSB                         0
LB                       2.00 Hz
GB                          0
PC                       1.40
```

liushouxin-lch



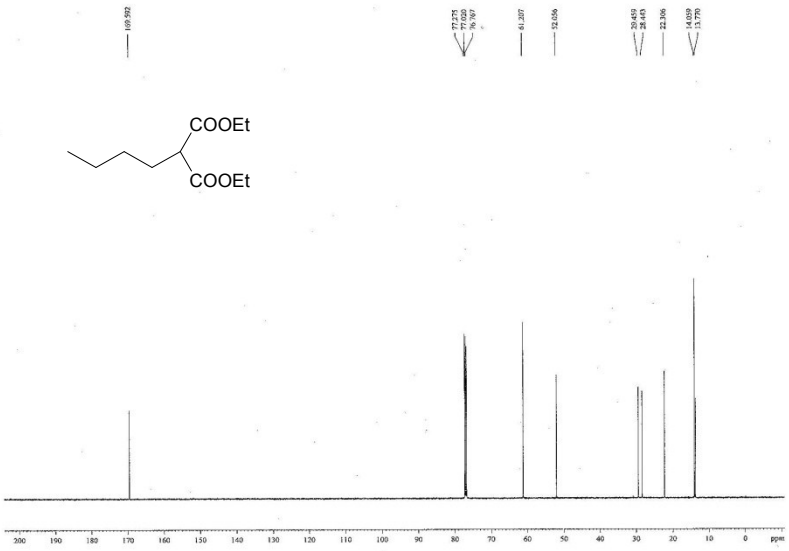

```
NAME                 2010.0608
EXPNO                        4
PROCNO                       1
Date_                 20100608
Time                     14.39
INSTRUM                  spect
PROBHD     5 mm PABBO BB-
PULPROG                  zg30
TD                      32768
SOLVENT                 CDCl3
NS                           4
DS                           0
SWH                 7500.000 Hz
FIDRES              0.228882 Hz
AQ                  2.18458834 sec
RG                         181
DW                    66.667 usec
DE                     6.50 usec
TE                    298.0 K
D1                 1.00000000 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                        1H
P1                        9.80 usec
PL1                       0.00 dB
PL1W               26.62599564 W
SFO1               500.1322506 MHz
SI                       65536
SF                 500.1300063 MHz
WDW                         EM
SSB                          0
LB                        0.30 Hz
GB                           0
PC                        1.00
```

liushouxin-licuihong



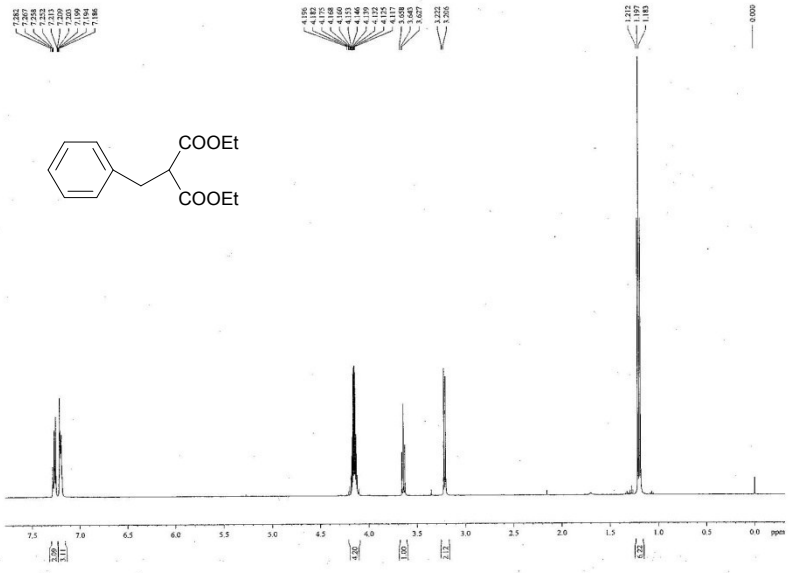

```
NAME                 2010.0601
EXPNO                        9
PROCNO                       1
Date_                 20100601
Time                     14.55
INSTRUM                  spect
PROBHD     5 mm PABBO BB-
PULPROG                  zg30
TD                      32768
SOLVENT                 CDCl3
NS                           1
DS                           0
SWH                 5000.000 Hz
FIDRES              0.152588 Hz
AQ                  3.2768500 sec
RG                        90.5
DW                   100.000 usec
DE                     6.50 usec
TE                    298.1 K
D1                 1.00000000 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                        1H
P1                        9.80 usec
PL1                       0.00 dB
PL1W               26.62599564 W
SFO1               500.1322506 MHz
SI                       32768
SF                 500.1300039 MHz
WDW                         EM
SSB                          0
LB                        0.30 Hz
GB                           0
PC                        1.00
```

liushouxin-licuihong-C



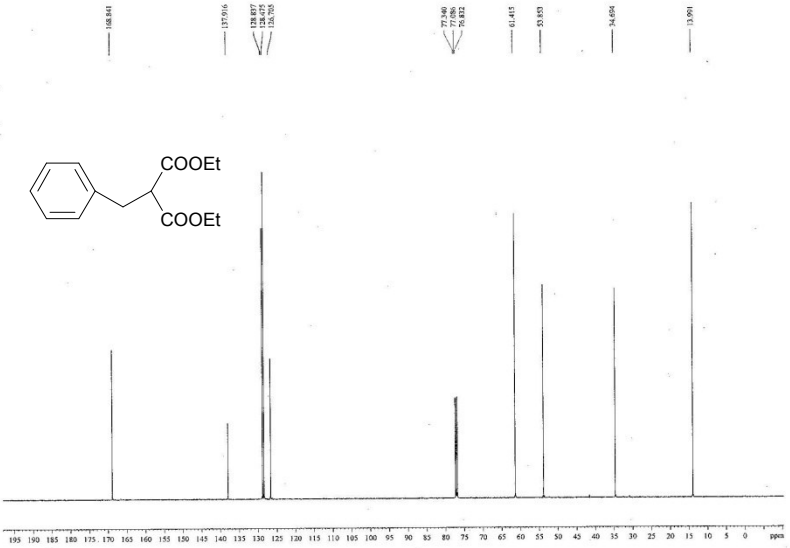

```
NAME                 2010.0604
EXPNO                        13
PROCNO                        1
Date_                20100604
Time_                   20.59
INSTRUM                  spect
PROBHD     5 mm PABBO BB-
PULPROG                 zgpg30
TD                       65536
SOLVENT                  CDCl3
NS                         506
DS                           4
SWH                  32894.738 Hz
FIDRES               0.501934 Hz
AQ                   0.9961972 sec
RG                        2050
DW                     15.200 usec
DE                      6.50 usec
TE                     300.0 K
D1                   2.00000000 sec
D11                  0.03000000 sec
TD0                           1

======== CHANNEL f1 ========
NUC1                       13C
P1                        9.20 usec
PL1                       3.00 dB
PL1W                47.46146682 W
SFO1               125.7728785 MHz

======== CHANNEL f2 ========
CPDPRG2                 waltz16
NUC2                        1H
PCPD2                    80.00 usec
PL2                       0.00 dB
PL12                     18.06 dB
PL13                     17.50 dB
PL1W                26.62599564 W
PL12W                0.41620368 W
PL13W                0.47248461 W
SFO2               500.1320005 MHz
SI                         32768
SF                 125.7577890 MHz
WDW                           EM
SSB                            0
LB                          1.00 Hz
GB                             0
PC                          1.40
```

liushouxin-Da



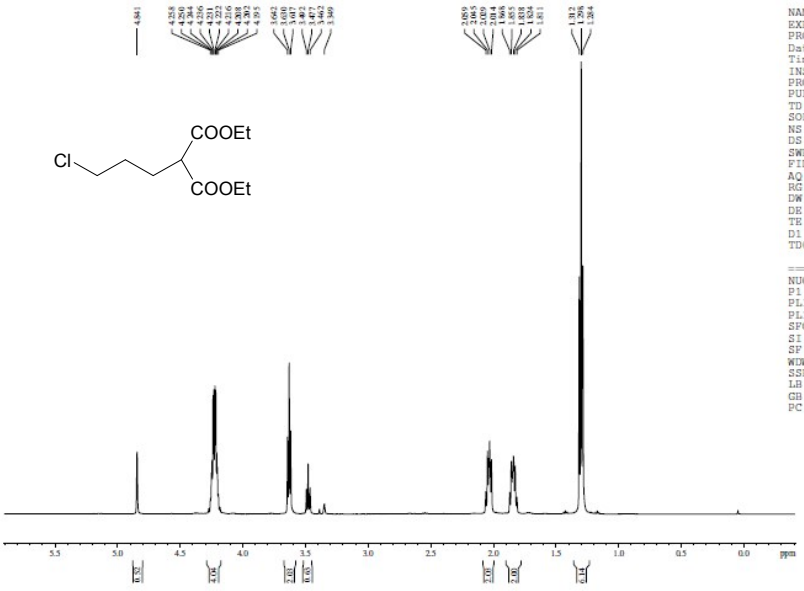

```
NAME                 2010.0524
EXPNO                        5
PROCNO                       1
Date_                20100524
Time                    17.12
INSTRUM                  spect
PROBHD     5 mm PABBO BB-
PULPROG                  zg30
TD                       32768
SOLVENT                  CDCl3
NS                           8
DS                           0
SWH                  5000.000 Hz
FIDRES               0.152588 Hz
AQ                   3.2768500 sec
RG                        40.3
DW                     100.000 usec
DE                      6.50 usec
TE                     298.6 K
D1                   1.00000000 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                        1H
P1                        9.80 usec
PL1                       0.00 dB
PL1W                26.62599564 W
SFO1               500.1322506 MHz
SI                         65536
SF                 500.1300142 MHz
WDW                           EM
SSB                            0
LB                          0.30 Hz
GB                             0
PC                          1.00
```

liushouxin-Da



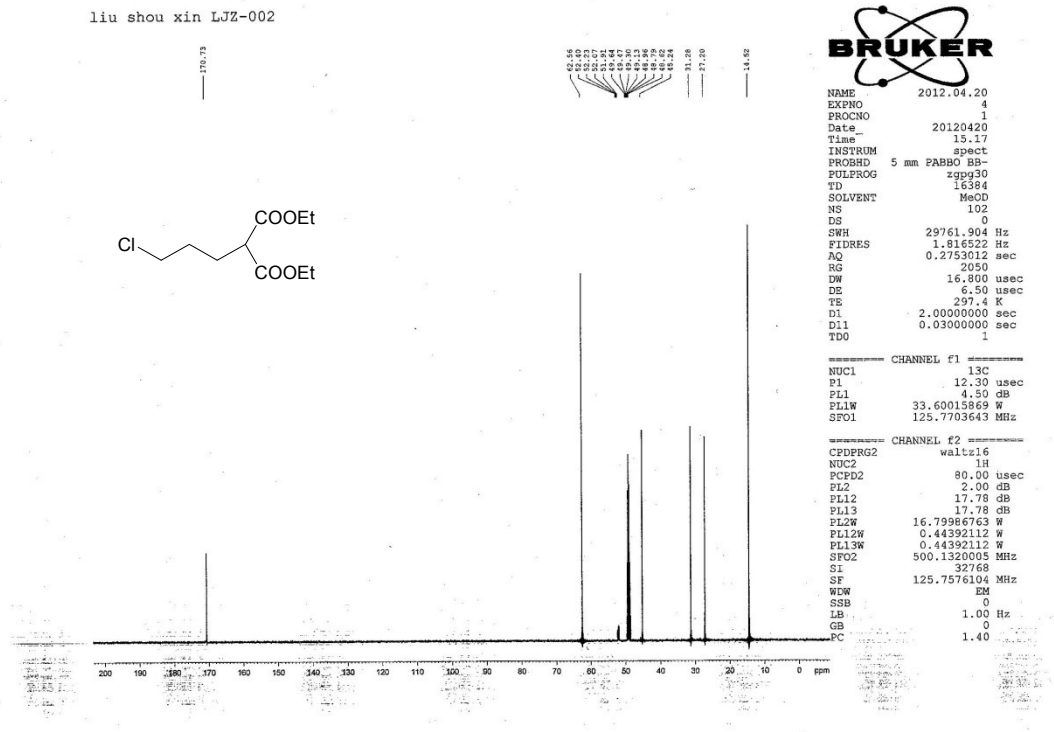

```
NAME                 2010.0526
EXPNO                        4
PROCNO                       1
Date_                 20100526
Time                     12.02
INSTRUM                  spect
PROBHD     5 mm PABBO BB-
PULPROG                 zgpg30
TD                       65536
SOLVENT                  CDCl3
NS                         811
DS                           4
SWH                  32894.733 Hz
FIDRES               0.501834 Hz
AQ                   0.9961972 sec
RG                        2050
DW                     15.200 usec
DE                      6.50 usec
TE                     300.1 K
D1                   2.00000000 sec
D11                  0.03000000 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                       13C
P1                        9.20 usec
PL1                       3.00 dB
PL1W                47.46168682 W
SFO1               125.7728799 MHz

======== CHANNEL f2 ========
CPDPRG2                 waltz16
NUC2                        1H
PCPD2                     80.00 usec
PL2                        0.00 dB
PL12                      18.06 dB
PL13                      17.50 dB
PL2W                26.62595564 W
PL12W                0.41620369 W
PL13W                0.47348461 W
SFO2               500.1330885 MHz
SI                        32768
SF                 125.7577890 MHz
WDW                          EM
SSB                           0
LB                         1.00 Hz
GB                            0
PC                         1.40
```

liu shou xin LJZ-002



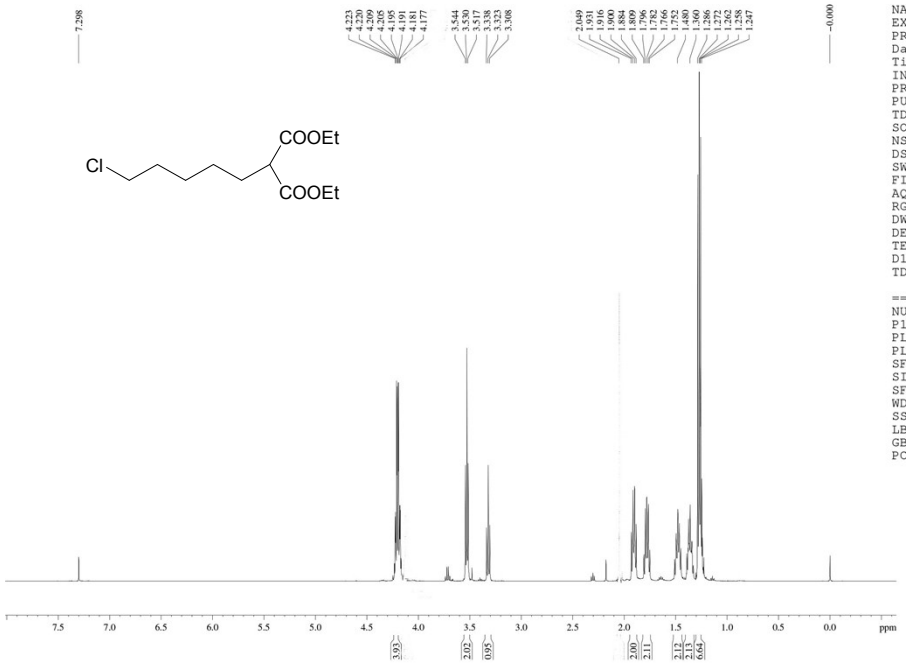

```
NAME                 2012.04.06
EXPNO                        5
PROCNO                       1
Date_                 20120406
Time                     8.53
INSTRUM                  spect
PROBHD     5 mm PABBO BB-
PULPROG                 zg30
TD                       65536
SOLVENT                  MeOD
NS                           1
DS                           2
SWH                  10000.000 Hz
FIDRES               0.152588 Hz
AQ                   3.2768500 sec
RG                        45.2
DW                     50.000 usec
DE                      6.50 usec
TE                     293.9 K
D1                   1.00000000 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                        1H
P1                        13.00 usec
PL1                        2.00 dB
PL1W                16.79986763 W
SFO1               500.1340010 MHz
SI                        32768
SF                 500.1299914 MHz
WDW                          EM
SSB                           0
LB                         0.30 Hz
GB                            0
PC                         1.00
```

liu shou xin LJZ-002



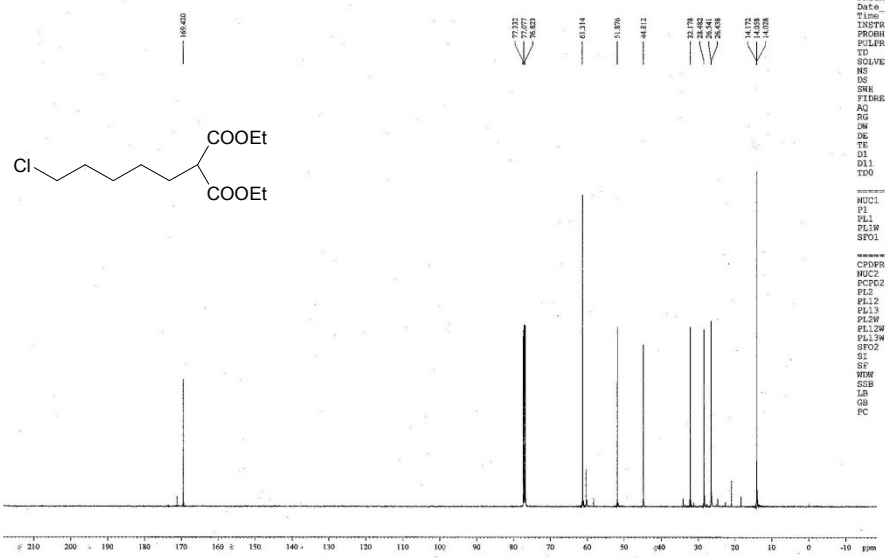

| | |
|---|---|
| NAME | 2012.04.20 |
| EXPNO | 4 |
| PROCNO | 1 |
| Date_ | 20120420 |
| Time | 15.17 |
| INSTRUM | spect |
| PROBHD | 5 mm PABBO BB- |
| PULPROG | zgpg30 |
| TD | 16384 |
| SOLVENT | MeOD |
| NS | 102 |
| DS | 0 |
| SWH | 29761.904 Hz |
| FIDRES | 1.816522 Hz |
| AQ | 0.2753012 sec |
| RG | 2050 |
| DW | 16.800 usec |
| DE | 6.50 usec |
| TE | 297.4 K |
| D1 | 2.00000000 sec |
| D11 | 0.03000000 sec |
| TD0 | 1 |
| ======== CHANNEL f1 ======== | |
| NUC1 | 13C |
| P1 | 12.30 usec |
| PL1 | 4.50 dB |
| PL1W | 33.60015869 W |
| SFO1 | 125.7703643 MHz |
| ======== CHANNEL f2 ======== | |
| CPDPRG2 | waltz16 |
| NUC2 | 1H |
| PCPD2 | 80.00 usec |
| PL2 | 2.00 dB |
| PL12 | 17.78 dB |
| PL13 | 17.78 dB |
| PL2W | 16.79986763 W |
| PL12W | 0.44392112 W |
| PL13W | 0.44392112 W |
| SFO2 | 500.1320005 MHz |
| SI | 32768 |
| SF | 125.7576104 MHz |
| WDW | EM |
| SSB | 0 |
| LB | 1.00 Hz |
| GB | 0 |
| PC | 1.40 |

liu shou xin 5-C-W



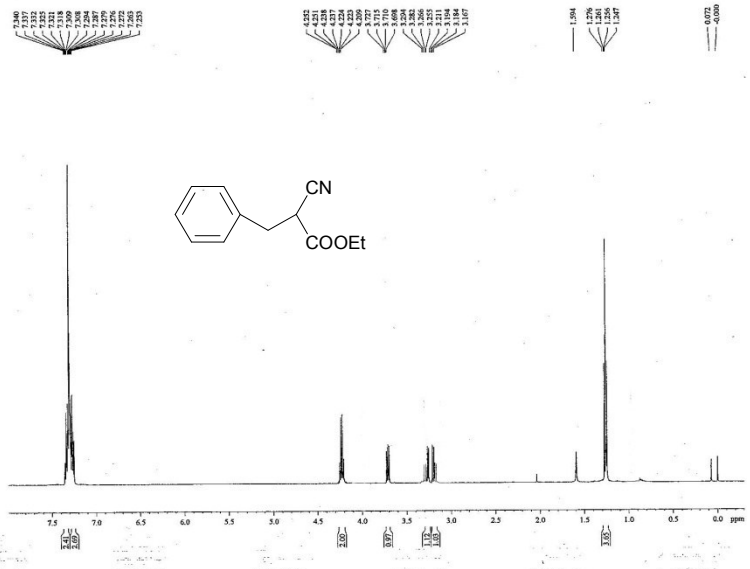

| | |
|---|---|
| NAME | 2012.03.20 |
| EXPNO | 8 |
| PROCNO | 1 |
| Date_ | 20120320 |
| Time | 13.57 |
| INSTRUM | spect |
| PROBHD | 5 mm PABBO BB- |
| PULPROG | zg30 |
| TD | 65536 |
| SOLVENT | CDCl3 |
| NS | 1 |
| DS | 2 |
| SWH | 10000.000 Hz |
| FIDRES | 0.152588 Hz |
| AQ | 3.2768500 sec |
| RG | 45.2 |
| DW | 50.000 usec |
| DE | 6.50 usec |
| TE | 291.0 K |
| D1 | 1.00000000 sec |
| TD0 | 1 |
| ======== CHANNEL f1 ======== | |
| NUC1 | 1H |
| P1 | 13.00 usec |
| PL1 | 2.00 dB |
| PL1W | 16.79986763 W |
| SFO1 | 500.1340010 MHz |
| SI | 32768 |
| SF | 500.1299938 MHz |
| WDW | EM |
| SSB | 0 |
| LB | 0.30 Hz |
| GB | 0 |
| PC | 1.00 |

liu shou xin5-C-W



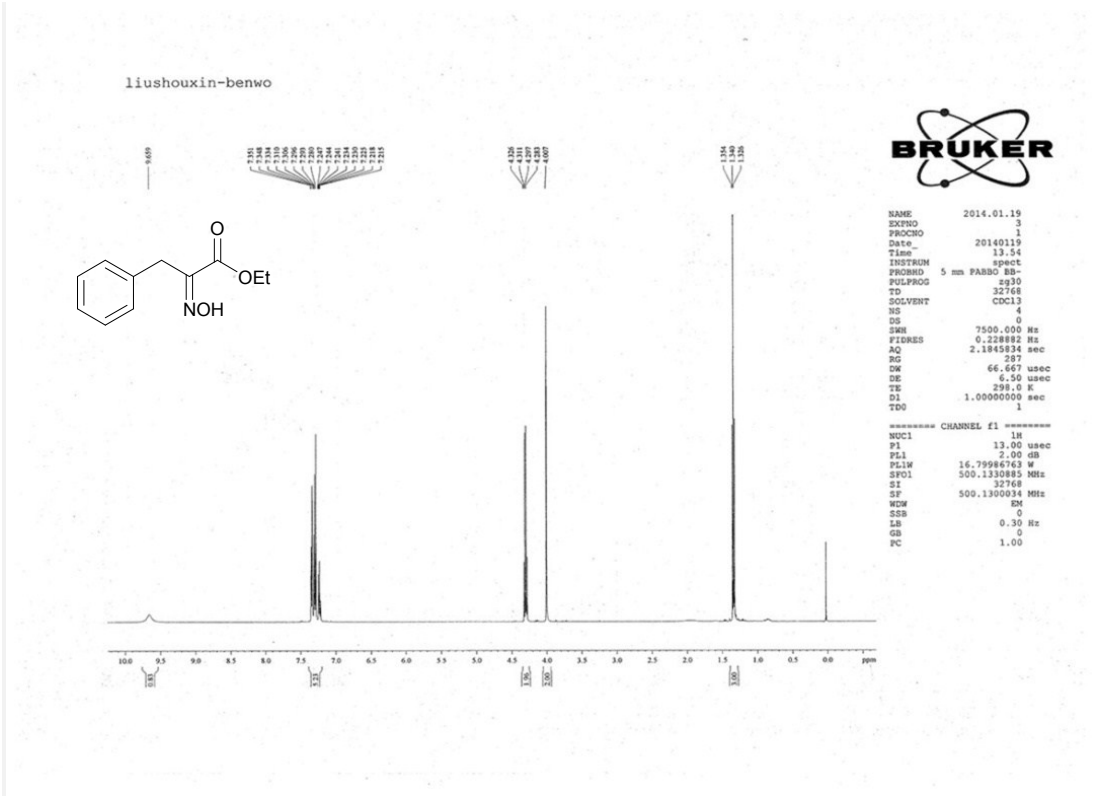

```
NAME                 2012.03.21
EXPNO                         6
PROCNO                        1
Date_                  20120321
Time                      17.00
INSTRUM                   spect
PROBHD   5 mm PABBO BB-
PULPROG                   zgpg30
TD                         16384
SOLVENT                   CDCl3
NS                         1116
DS                            0
SWH                   29761.904 Hz
FIDRES                1.816522 Hz
AQ                    0.2753012 sec
RG                         2050
DW                       16.800 usec
DE                        6.50 usec
TE                       292.2 K
D1                    2.00000000 sec
D11                   0.03000000 sec
TD0                           1

======== CHANNEL f1 ========
NUC1                        13C
P1                        12.30 usec
PL1                        4.50 dB
PL1W                33.60015869 W
SFO1               125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG2                 waltz16
NUC2                         1H
PCPD2                     80.00 usec
PL2                        2.00 dB
PL12                      17.78 dB
PL13                      17.78 dB
PL2W                16.79986763 W
PL12W               0.44392112 W
PL13W               0.44392112 W
SFO2               500.1320005 MHz
SI                         32768
SF                 125.7577890 MHz
WDW                           EM
SSB                           0
LB                         2.00 Hz
GB                            0
PC                         1.40
```

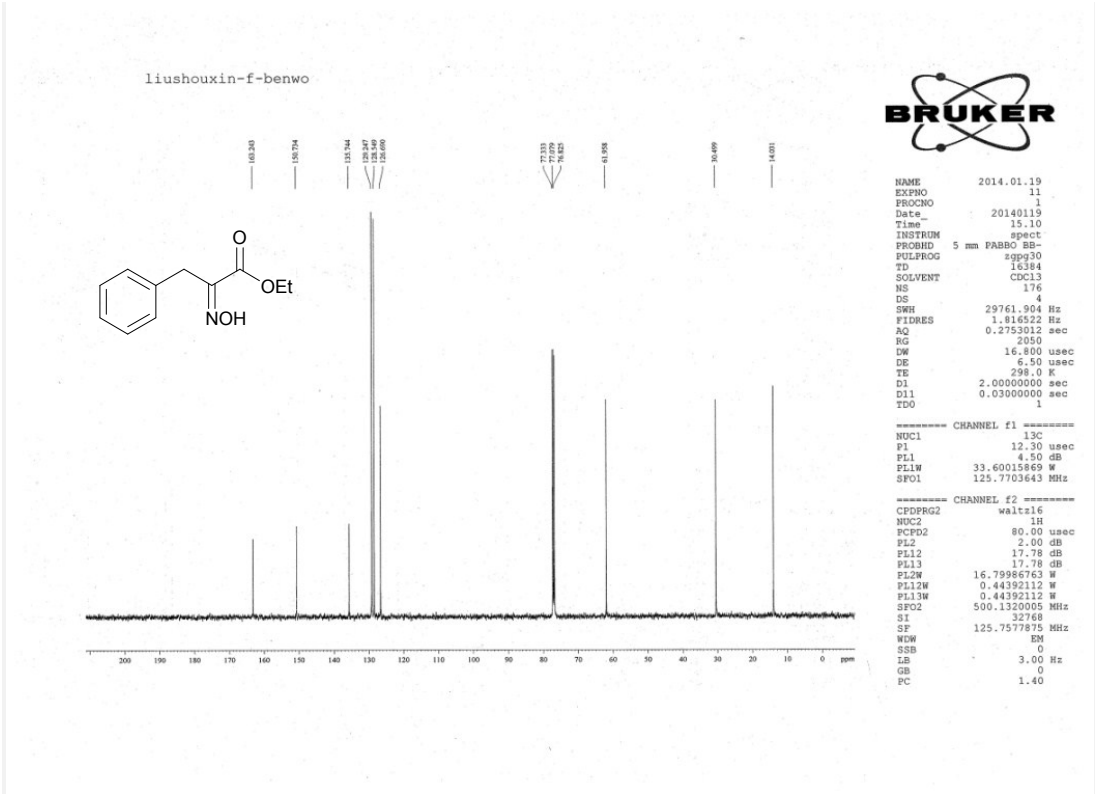
liushouxin-YQ

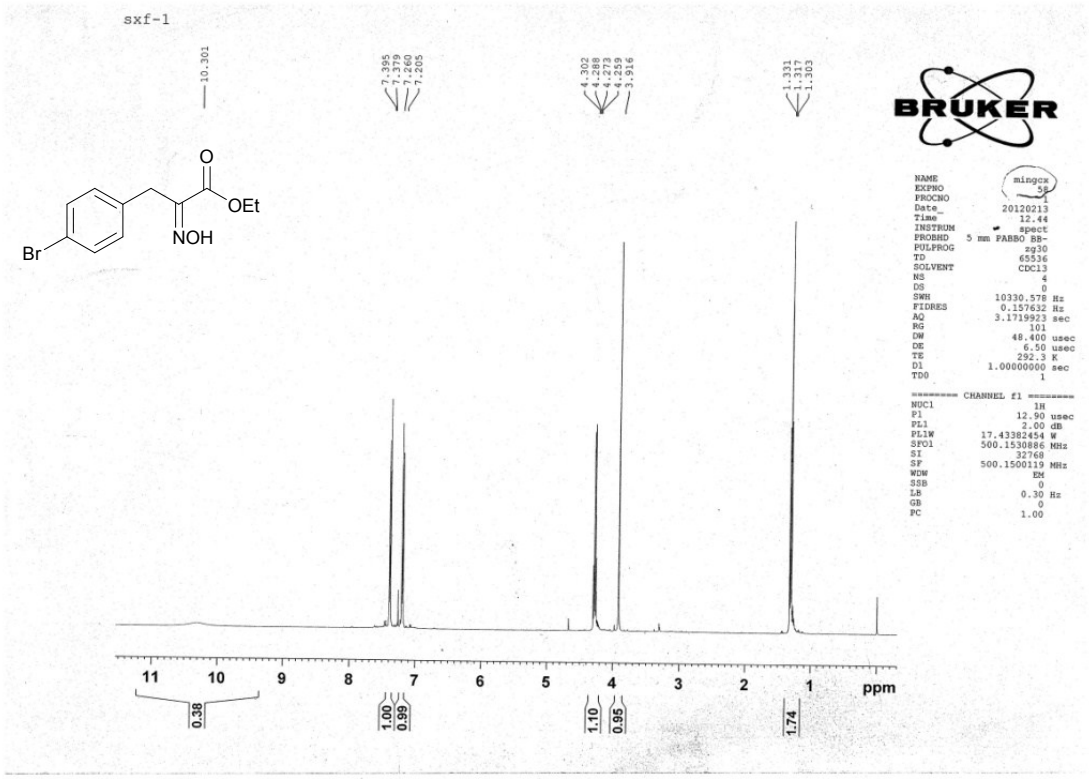


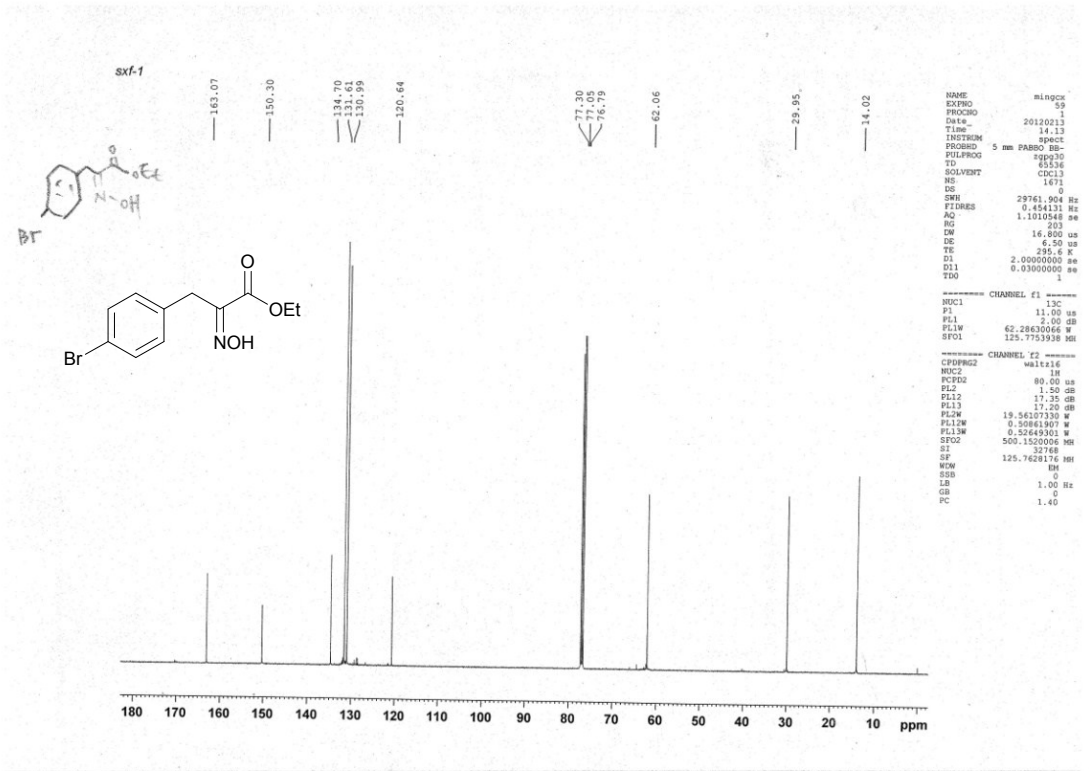

```
NAME                 2010.0604
EXPNO                         2
PROCNO                        1
Date_                  20100604
Time                       9.27
INSTRUM                   spect
PROBHD   5 mm PABBO BB-
PULPROG                    zg30
TD                         32768
SOLVENT                   CDCl3
NS                             8
DS                             0
SWH                 5000.000 Hz
FIDRES                0.152588 Hz
AQ                    3.2768500 sec
RG                          90.5
DW                      100.000 usec
DE                        6.50 usec
TE                       298.1 K
D1                    1.00000000 sec
TD0                           1

======== CHANNEL f1 ========
NUC1                         1H
P1                         9.80 usec
PL1                        0.00 dB
PL1W                26.62599564 W
SFO1               500.1322506 MHz
SI                         32768
SF                 500.1300172 MHz
WDW                           EM
SSB                           0
LB                         0.30 Hz
GB                            0
PC                         1.00
```

**$^{1}H$ and $^{13}C$ spectra of oximes**

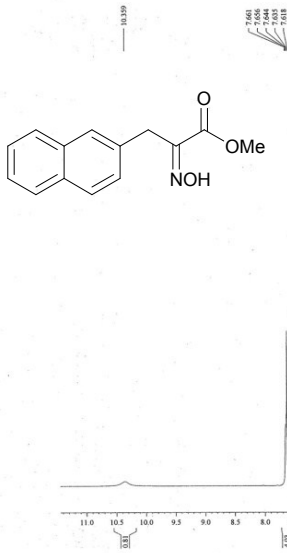



sxf-1
10.301
7.395
7.379
7.260
7.205
4.302
4.288
4.273
4.259
3.936
1.331
1.317
1.303
O
OEt
NOH
Br
11
10
9
8
7
6
5
4
3
2
1
ppm
0.38
1.00
0.99
1.10
0.95
1.74

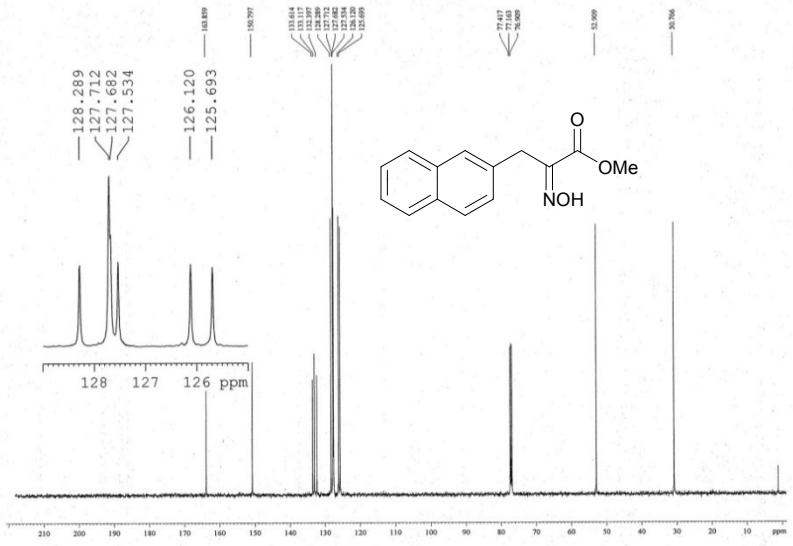

sxf-1
163.07
150.30
134.70
131.61
130.99
120.64
77.30
77.05
76.79
62.06
29.95
14.02
O
OEt
NOH
Br
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

liushouxin-f-naiwo-jiazhi



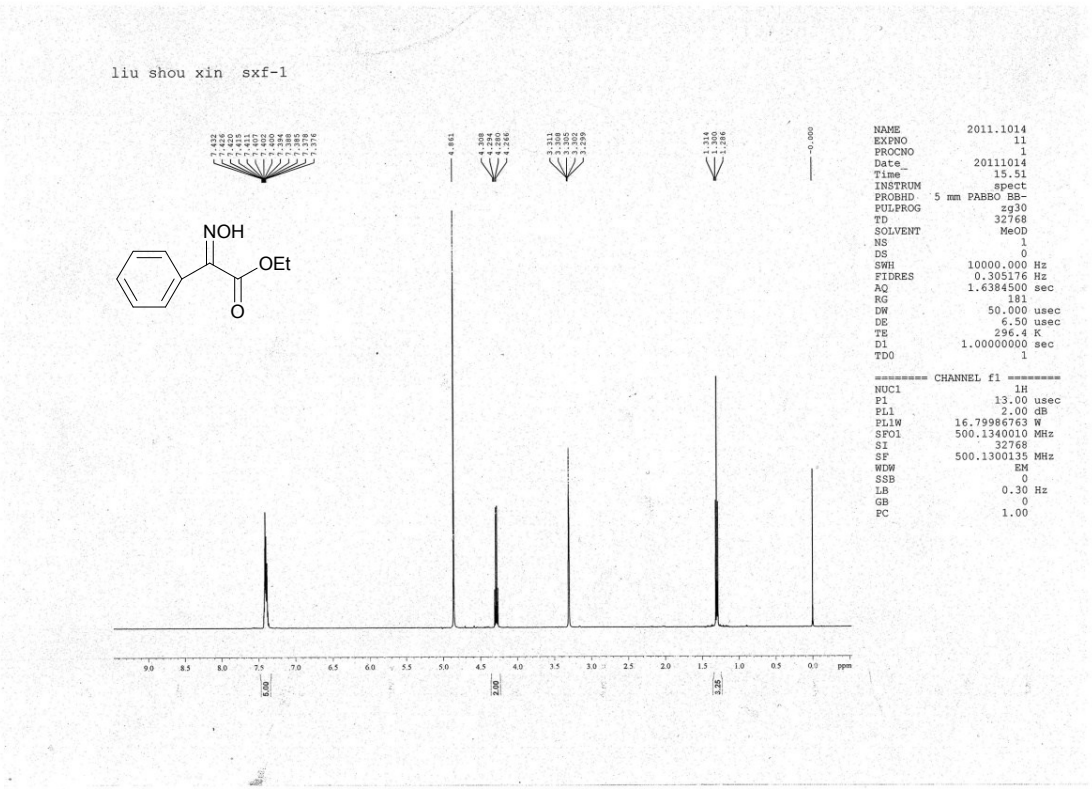

```
NAME                2014.01.19
EXPNO                        9
PROCNO                       1
Date_                 20140119
Time_                    14.30
INSTRUM                  spect
PROBHD     5 mm PABBO BB-
PULPROG                  zg30
TD                      32768
SOLVENT                 CDCl3
NS                           4
DS                           0
SWH                 7500.000 Hz
FIDRES              0.228882 Hz
AQ                  2.1845834 sec
RG                       50.8
DW                     66.667 usec
DE                      6.50 usec
TE                     298.0 K
D1                  1.00000000 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                       1H
P1                     13.00 usec
PL1                     2.00 dB
PL1W               16.79986763 W
SFO1               500.1330885 MHz
SI                       32768
SF                 500.1300979 MHz
WDW                         EM
SSB                          0
LB                      0.30 Hz
GB                           0
PC                        1.00
```

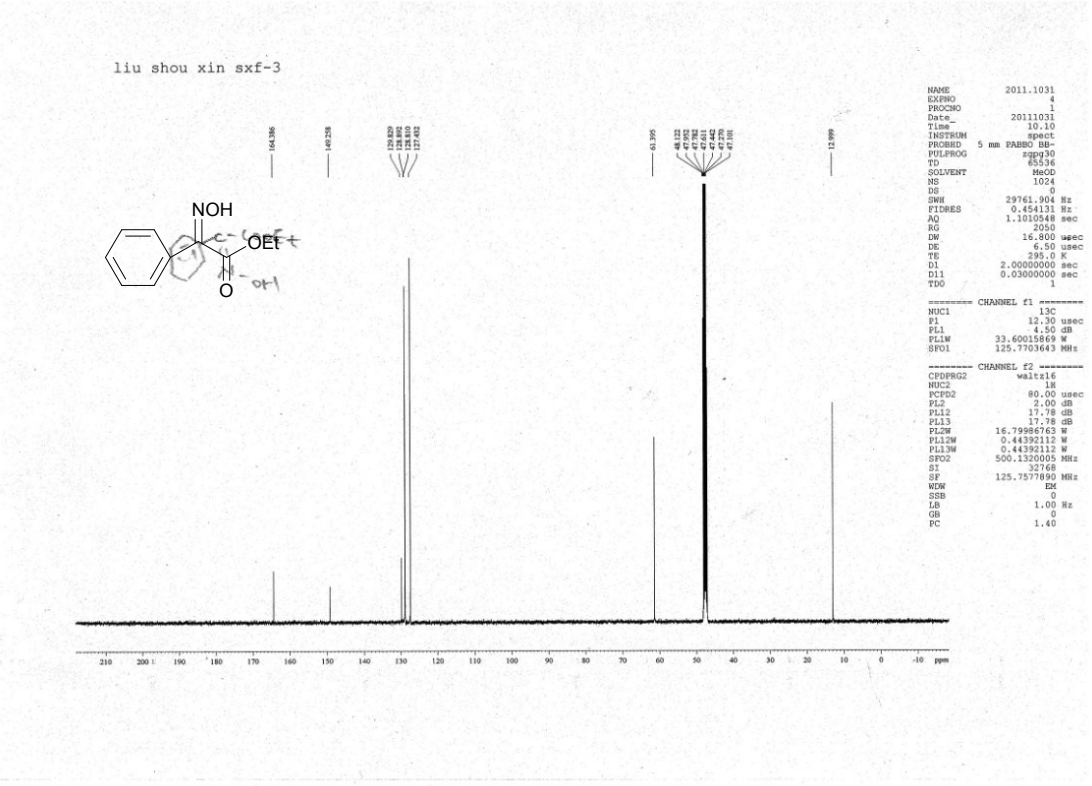
liushouxin-f-naiwo-jiazhi

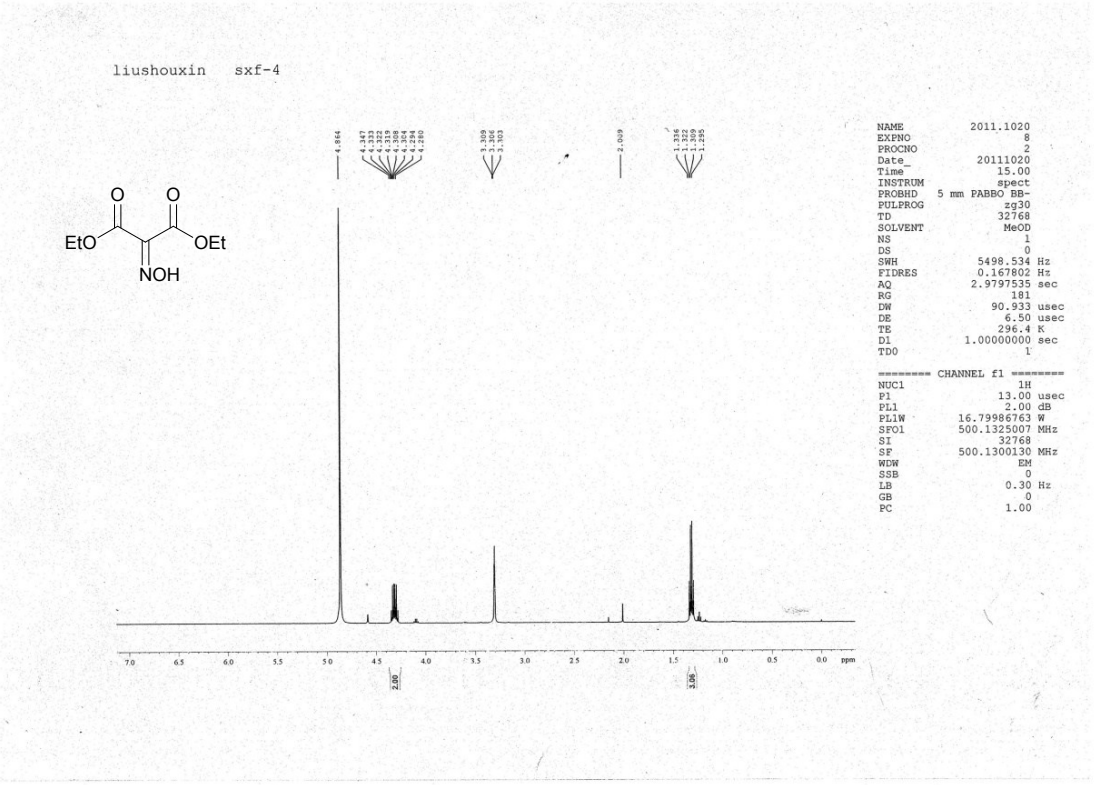


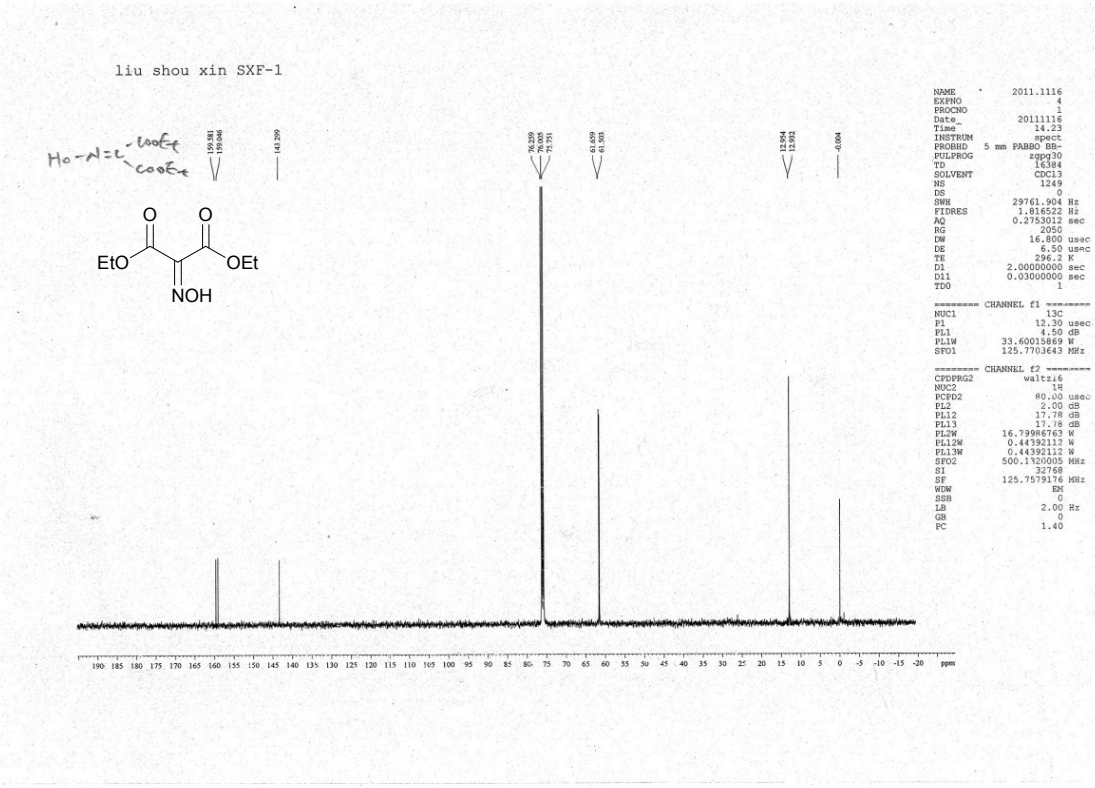

BRUKER

```
NAME                2014.01.19
EXPNO                       10
PROCNO                       1
Date_                 20140119
Time_                    14.42
INSTRUM                  spect
PROBHD     5 mm PABBO BB-
PULPROG                 zgpg30
TD                      16384
SOLVENT                 CDCl3
NS                         162
DS                           4
SWH                29761.904 Hz
FIDRES              1.816522 Hz
AQ                  0.2753012 sec
RG                        2050
DW                    16.800 usec
DE                      6.50 usec
TE                     298.0 K
D1                  2.00000000 sec
D11                 0.03000000 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                       13C
P1                     12.30 usec
PL1                     4.50 dB
PL1W               33.60015869 W
SFO1               125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                  80.00 usec
PL2                     2.00 dB
PL12                   17.78 dB
PL13                   17.78 dB
PL2W               16.79986763 W
PL12W               0.44392112 W
PL13W               0.44392112 W
SFO2               500.1320005 MHz
SI                       32768
SF                 125.7577875 MHz
WDW                         EM
SSB                          0
LB                      3.00 Hz
GB                           0
PC                        1.40
```

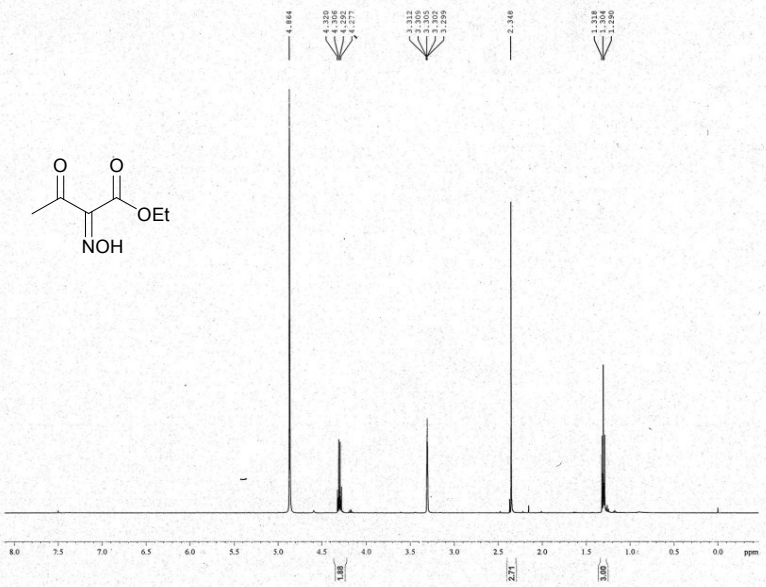

liu shou xin sxf-1
NOH
OEt
O
NAME 2011.1014
EXPNO 11
PROCNO 1
Date_ 20111014
Time 15.51
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 32768
SOLVENT MeOD
NS 1
DS 0
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 1.6384500 sec
RG 181
DW 50.000 usec
DE 6.50 usec
TE 296.4 K
D1 1.00000000 sec
TD0 1
CHANNEL f1
NUC1 1H
P1 13.00 usec
PL1 2.00 dB
PL1W 16.79986763 W
SFO1 500.1340010 MHz
SI 32768
SF 500.1300135 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00


liu shou xin sxf-3
NOH
OEt
O
NAME 2011.1031
EXPNO 4
PROCNO 1
Date_ 20111031
Time 10.10
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT MeOD
NS 1024
DS 0
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 2050
DW 16.800 usec
DE 6.50 usec
TE 295.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
CHANNEL f1
NUC1 13C
P1 12.30 usec
PL1 4.50 dB
PL1W 33.60015869 W
SFO1 125.7703643 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.00 dB
PL12 17.78 dB
PL13 17.78 dB
PL2W 16.79986763 W
PL12W 0.44392112 W
PL13W 0.44392112 W
SFO2 500.1320005 MHz
SI 32768
SF 125.7577890 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40


liushouxin sxf-4
EtO
OEt
NOH
4.864
4.347
4.332
4.318
4.308
4.294
4.280
3.309
3.304
3.303
2.009
1.336
1.322
1.308
1.295
2.00
3.06
ppm
NAME 2011.1020
EXPNO 8
PROCNO 2
Date_ 20111020
Time 15.00
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 32768
SOLVENT MeOD
NS 1
DS 0
SWH 5498.534 Hz
FIDRES 0.167802 Hz
AQ 2.9797535 sec
RG 181
DW 90.933 usec
DE 6.50 usec
TE 296.4 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.00 usec
PL1 2.00 dB
PL1W 16.79986763 W
SFO1 500.1325007 MHz
SI 32768
SF 500.1300130 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

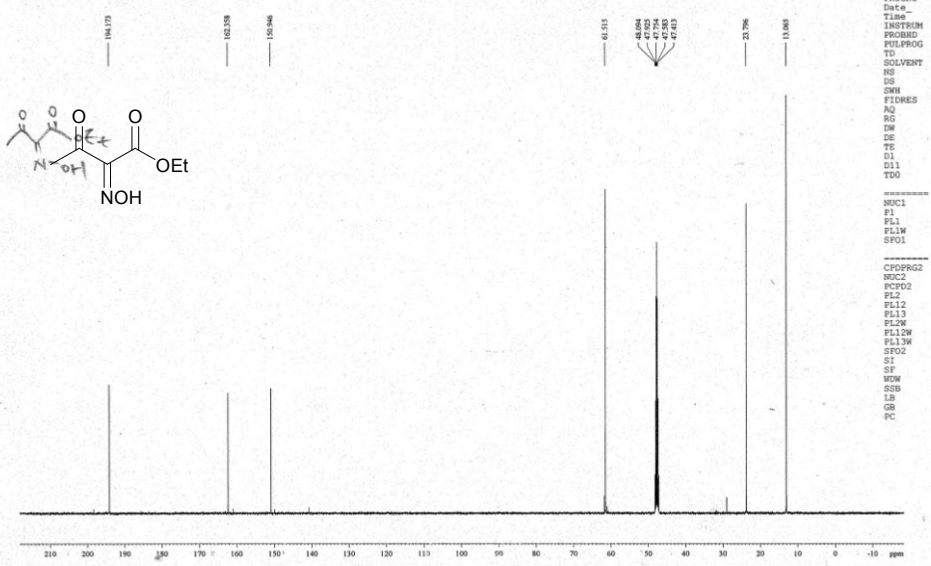

liu shou xin SXF-1
EtO
OEt
NOH
160.581
159.866
141.299
76.329
76.005
75.791
61.659
61.503
15.904
15.852
-0.004
ppm
NAME 2011.1116
EXPNO 4
PROCNO 1
Date_ 20111116
Time 14.23
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 16384
SOLVENT CDCl3
NS 1249
DS 0
SWH 29761.904 Hz
FIDRES 1.816522 Hz
AQ 0.2753012 sec
RG 2050
DW 16.800 usec
DE 6.50 usec
TE 296.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 12.30 usec
PL1 4.50 dB
PL1W 33.60015869 W
SFO1 125.7703643 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.00 dB
PL12 17.78 dB
PL13 17.78 dB
PL2W 16.79986763 W
PL12W 0.44392112 W
PL13W 0.44392112 W
SFO2 500.1320005 MHz
SI 32768
SF 125.7579176 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.40

liushouxin SXF-2

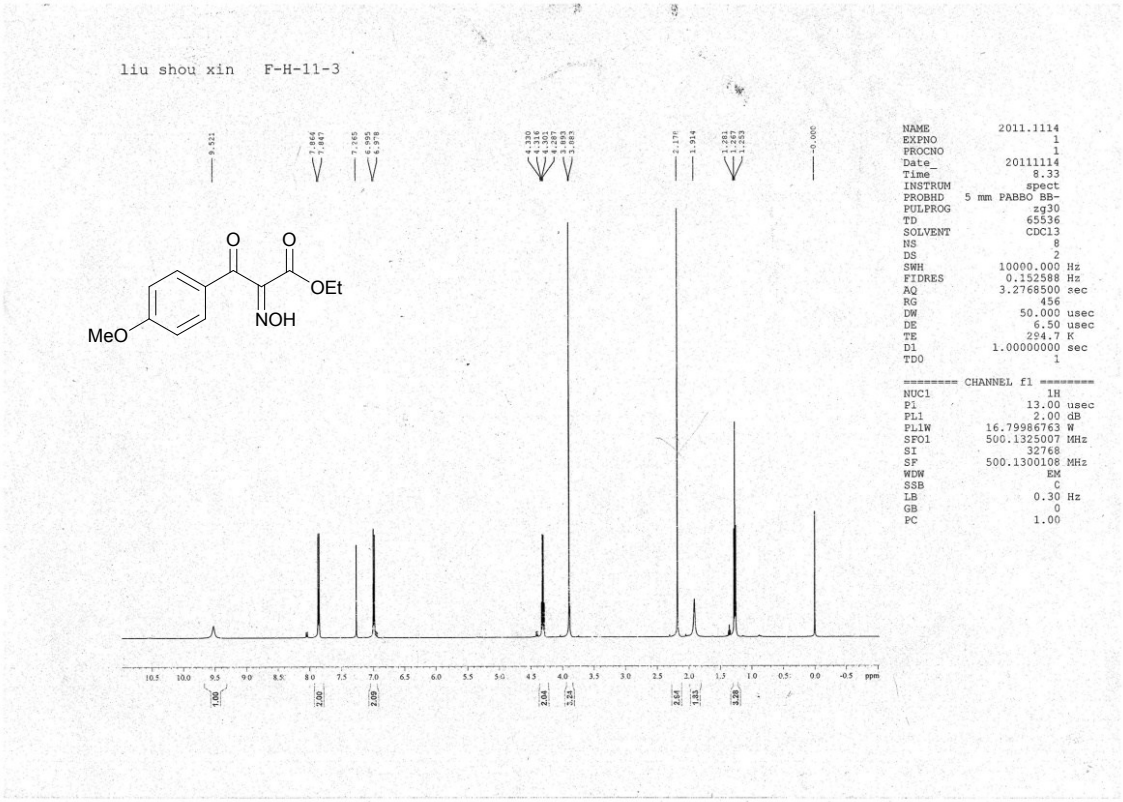



```
NAME                 2011.1018
EXPNO                       18
PROCNO                       1
Date_                 20111018
Time                     15.17
INSTRUM                  spect
PROBHD   5 mm PABBO BB-
PULPROG                   zg30
TD                       32768
SOLVENT                   MeOD
NS                           1
DS                           0
SWH                10000.000 Hz
FIDRES                 0.305176 Hz
AQ                    1.6384500 sec
RG                         362
DW                      50.000 usec
DE                       6.50 usec
TE                      296.2 K
D1                 1.00000000 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                       1H
P1                      13.00 usec
PL1                      2.00 dB
PL1W               16.79986763 W
SFO1              500.1322506 MHz
SI                       32768
SF                500.1300131 MHz
WDW                         EM
SSB                          0
LB                      0.30 Hz
GB                           0
PC                        1.00
```

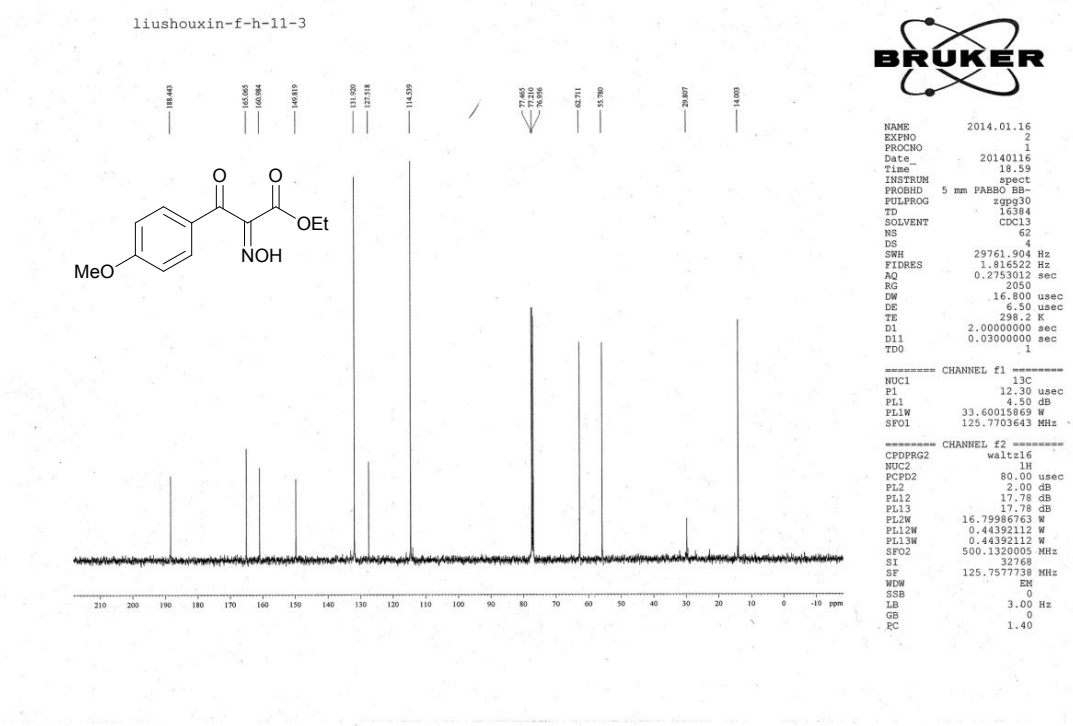
liu shou xin sxf-1




```
NAME                 2011.1031
EXPNO                        2
PROCNO                       1
Date_                 20111031
Time                     9.49
INSTRUM                  spect
PROBHD   5 mm PABBO BB-
PULPROG                zgpg30
TD                       65536
SOLVENT                   MeOD
NS                          78
DS                           0
SWH                29761.904 Hz
FIDRES                 0.454131 Hz
AQ                    1.1010548 sec
RG                        2050
DW                      16.800 usec
DE                       6.50 usec
TE                      294.9 K
D1                 2.00000000 sec
D11                0.03000000 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                       13C
P1                      12.30 usec
PL1                      4.50 dB
PL1W               33.60015869 W
SFO1              125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                   80.00 usec
PL2                      2.00 dB
PL12                    17.78 dB
PL13                    17.78 dB
PL2W               16.79986763 W
PL12W               0.44392112 W
PL13W               0.44392112 W
SFO2              500.1320005 MHz
SI                       32768
SF                125.7577890 MHz
WDW                         EM
SSB                          0
LB                      1.00 Hz
GB                           0
PC                        1.40
```




```
NAME                2011.1114
EXPNO                       1
PROCNO                      1
Date_               20111114
Time                    8.33
INSTRUM                spect
PROBHD    5 mm PABBO BB-
PULPROG                 zg30
TD                     65536
SOLVENT                CDCl3
NS                          8
DS                          2
SWH                10000.000 Hz
FIDRES             0.152588 Hz
AQ                 3.2768500 sec
RG                        456
DW                   50.000 usec
DE                    6.50 usec
TE                    294.7 K
D1                 1.00000000 sec
TD0                         1

======== CHANNEL f1 ========
NUC1                       1H
P1                    13.00 usec
PL1                    2.00 dB
PL1W              16.79986763 W
SFO1              500.1325007 MHz
SI                       32768
SF                500.1300108 MHz
WDW                        EM
SSB                         0
LB                     0.30 Hz
GB                          0
PC                     1.00
```




```
NAME                2014.01.16
EXPNO                       2
PROCNO                      1
Date_               20140116
Time                   18.59
INSTRUM                spect
PROBHD    5 mm PABBO BB-
PULPROG                zgpg30
TD                     16384
SOLVENT                CDCl3
NS                         62
DS                          4
SWH                29761.904 Hz
FIDRES             1.816522 Hz
AQ                 0.2753012 sec
RG                       2050
DW                   16.800 usec
DE                    6.50 usec
TE                    298.2 K
D1                 2.00000000 sec
D11                0.03000000 sec
TD0                         1

======== CHANNEL f1 ========
NUC1                      13C
P1                    12.30 usec
PL1                    4.50 dB
PL1W              33.60015869 W
SFO1              125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                 80.00 usec
PL2                    2.00 dB
PL12                  17.78 dB
PL13                  17.78 dB
PL2W              16.79986763 W
PL12W             0.44392112 W
PL13W             0.44392112 W
SFO2              500.1320005 MHz
SI                       32768
SF                125.7577738 MHz
WDW                        EM
SSB                         0
LB                     3.00 Hz
GB                          0
PC                     1.40
```