Anthrone-Derived NHPI Analogues as Catalysts in Reactions Using Oxygen as an Oxidant

Juan Shen and Choon-Hong Tan*

Department of Chemistry, 3 Science Drive 3, National University of Singapore, Singapore 117543

Email: chmtanch@nus.edu.sg

Supplementary data

1. General methods
2. Experimental procedures
3. Characterization of the oxidation products
4. Copies of NMR spectrum

1. General methods

^{1}H and ^{13}C NMR spectra were recorded at room temperature in $CDCl_3$ with a Bruker ACF300 (300MHz) or AMX500 (500MHz) spectrometer. Chemical shifts (*d*) are reported in parts per million (ppm). Coupling constants are given as absolute values expressed in Hz. Low resolution mass spectra were obtained on a VG Micromass 7035 spectrometer in EI mode, a Finnigan/MAT LCQ spectrometer in ESI mode, and a Finnigan/MAT 95XL-T mass spectrometer in FAB mode. All high resolution mass spectra were obtained on a Finnigan/MAT 95XL-T spectrometer. Infrared spectra were recorded on a BIO-RAD FTS 165 FTIR spectrometer. Enantiomeric excesses were determined by chiral HPLC analysis on Jasco HPLC units, including a Jasco DG-980-50 Degasser, a LG-980-02 Ternary Gradient Unit, a PU-980 Intelligient HPLC Pump, UV-975 Intelligient UV/VIS Detectors, and an AS-950 Intelligient Sampler. Optical rotations were recorded on a Jasco DIP-1000 polarimeter. Melting points were determined on a BÜCHI B-540 melting point apparatus. Analytical thin layer chromatography (TLC) was performed with Merck pre-coated TLC plates, silica gel 60F-254, layer thickness 0.25 mm. Flash chromatography separations were performed on Merck 60 (0.040 - 0.063mm) mesh silica gel. All distilled solvents were stored under N_2. All other reagents and solvents are commercial grade and were used as supplied without further purification, unless otherwise stated.

2. Experimental procedures

General procedures for bicyclic guanidine catalyzed reactions between substituted anthrone and maleimides.

To a 5 ml RBF containing catalyst **1** (5.8 mg, 0.02 mmol, 10 mol %) and a stirring bar, anhydrous CH_2Cl_2 (1.0 ml), 1, 8-dichloro-9-anthrone **2a** (58 mg, 0.22mmol) and *N*-acetoxymaleimide **3a** (31.0 mg, 0.2mmol) were added in this sequence. After stirring at -20 ^oC for 8 hrs, the reaction mixture was loaded onto a short silica gel column, followed by flash chromatography (gradient elution with hexane/EA mixtures; 9/1 to 4/1). Product **4a** (72mg) was obtained as a white solid in 86% yield and 92% ee.

General procedures for the chiral anthrone-derived NHPI analogue mediated oxidations of benzylic compounds and diol.

A solution of substrate **6** (0.2 mmol), catalyst **5** (0.02 mmol), and $Co(OAc)_2$ (0.002 mmol) in anhydrous acetonitrile (1 ml) was placed in a round bottom flask. The flask was equipped with a balloon filled with O_2 (1atm). The mixture was vigorously stirred at 60 ^oC for 10h, evaporation of the solvent followed by flash chromatography on silica gel afforded product as a white solid.

General Procedures for the synthesis of β-hydroxy ketal 17.

A solution of substrate **15a** (1mmol), substrate **16a** (0.2 mmol), catalyst **14** (0.02 mmol), and $Co(OAc)_2$ (0.002 mmol) was placed in a round bottom flask. The flask was equipped with a balloon filled with O_2 (1atm). The mixture was vigorously stirred at rt for 5 h. The recovery of unreacted **15a** under a reduced pressure followed by flash chromatography on silica gel (*n*-hexane–AcOEt = 1:2) afforded product **17a** (60% yield) as a colorless liquid.

Procedures for the synthesis of α-hydroxy-γ-lactone 19.

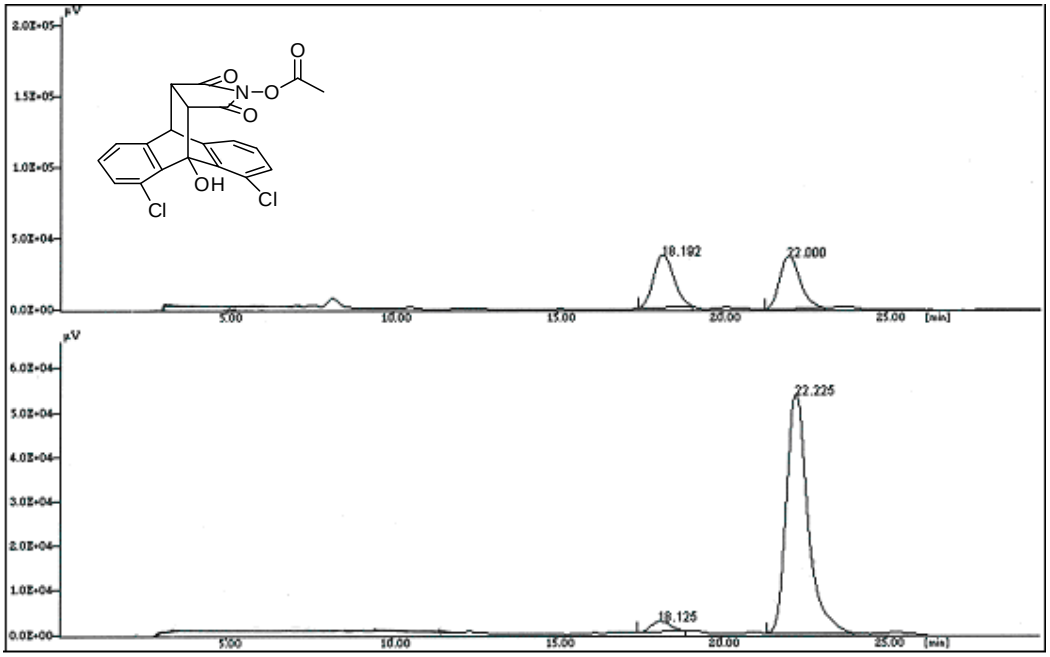
To a solution of alcohol **18** (2 mmol), catalyst **14** (0.02 mmol), $Co(OAc)_2$ (0.002 mmol) in acetonitrile (0.5 ml) placed in a round bottom flask, **16a** (0.2 mmol) was was added. The flask was equipped a ballon filled with O_2 (1atm). After the mixture was stirred at rt for 8 h, rotary evaporation of the solvent and unreacted alcohol followed by flash chromatography on silica gel afforded product **19** in 65 % yield.

3. Characterization of the oxidation products

(**4a**) 2-Acetoxy-5, 13-dichloro-4-hydroxy-3a, 4, 9, 9a-tetrahydro-4, 9-[1', 2'] benzeno -1*H*-benz[*f*]isoindole-1, 3(2*H*)-dione

White solid. mp 251.9-252.7 ^{o}C. 86% yield, 92% ee. $[\alpha]_D^{25}$ +58 (*c* 0.25, $CHCl_3$). ^{1}H NMR (500 MHz, $CDCl_3$, ppm)：δ 2.15 (s, 3H), 3.32 (dd, 1H, J = 3.1, 8.8Hz), 3.43 (d, 1H, J = 9.5Hz), 4.69 (d, 1H, J = 3.2Hz), 4.86 (s, 1H), 7.12-7.27 (m, 6H). ^{13}C NMR 125 MHz, $CDCl_3$, ppm): 17.3, 43.0, 44.9, 48.6, 81.3, 122.7, 124.2, 128.5, 129.1, 130.0, 130.1, 131.3, 134.8, 138.7, 141.5, 168.4, 168.6. IR (film): 1215.4, 1637.8, 3019.0 cm^{-1}. LRMS(FAB) m/z 417.8 (M^+), HRMS(FAB) m/z 418.0249 ($M+H^+$), calc. for $C_{20}H_{14}Cl_2NO_5$ 418.0249.

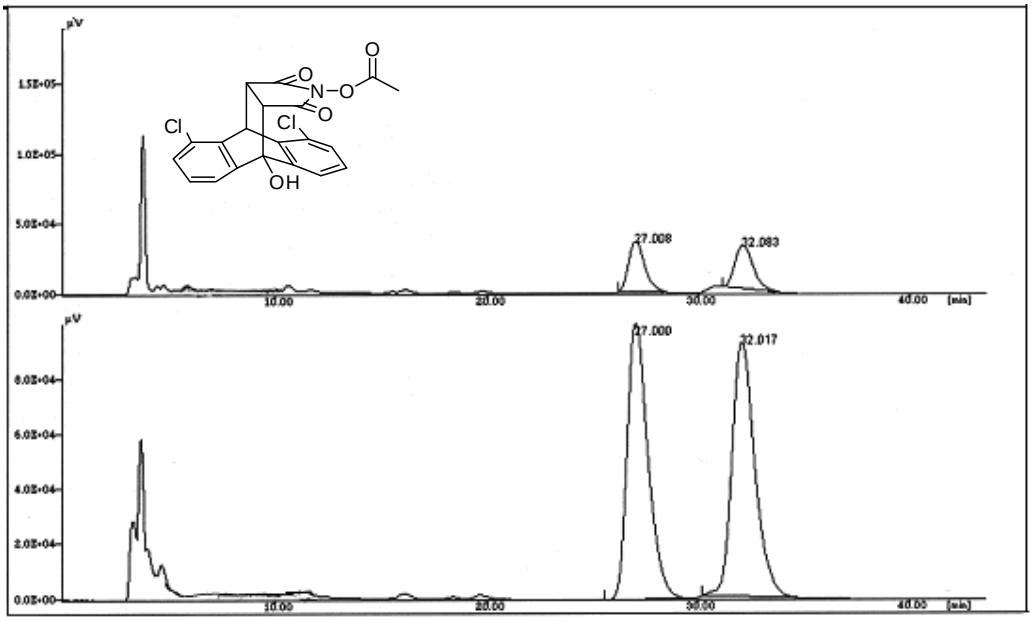
The ee determined by chiral HPLC; CHIRALCEL AD-H (4.6 mm i.d. x 250 mm); hexane/2-propanol 80/20; flow rate 1.0 ml/min; temp 25 °C; detection UV 230 nm; retention time: 18.1 min and 22.2 min.



(**4b**) 8, 10-Dichloro-2-acetoxy-4-hydroxy-3a, 4, 9, 9a-tetrahydro-4, 9-[1', 2'] benzeno-1*H*-benz[*f*]isoindole-1, 3(2*H*)-dione

White solid. mp 244.1-245.0 ^{o}C. 85% yield, 12% ee. ^{1}H NMR (300 MHz, $CDCl_3$, ppm) ： δ 2.16 (s, 3H), 3.20 (d, 1H, J = 8.7Hz), 3.42 (dd, 1H, J = 3.1, 8.4Hz), 4.33 (s, 1H), 5.87 (d, 1H, J = 3.1Hz), 7.20-7.60 (m, 6H). ^{13}C NMR (75 MHz, $CDCl_3$, ppm): δ 14.1, 17.3, 36.9, 43.4, 47.1, 119.5, 119.9, 127.7, 128.2, 128.4, 128.6, 130.0, 131.1, 133.3, 142.4, 144.3, 167.7, 169.9. IR (film): 1216.0, 1643.3, 3019.5 cm^{-1}. LRMS(FAB) m/z 418.0 (M^+), HRMS(FAB) m/z 418.0248 ($M+H^+$), calc. for $C_{20}H_{14}Cl_2NO_5$ 418.0249.

The ee determined by chiral HPLC; CHIRALCEL AD-H (4.6 mm i.d. x 250 mm); hexane/2-propanol 80/20; flow rate 1.0 ml/min; temp 25 °C; detection UV 230 nm; retention time: 27.0 min and 32.1 min.

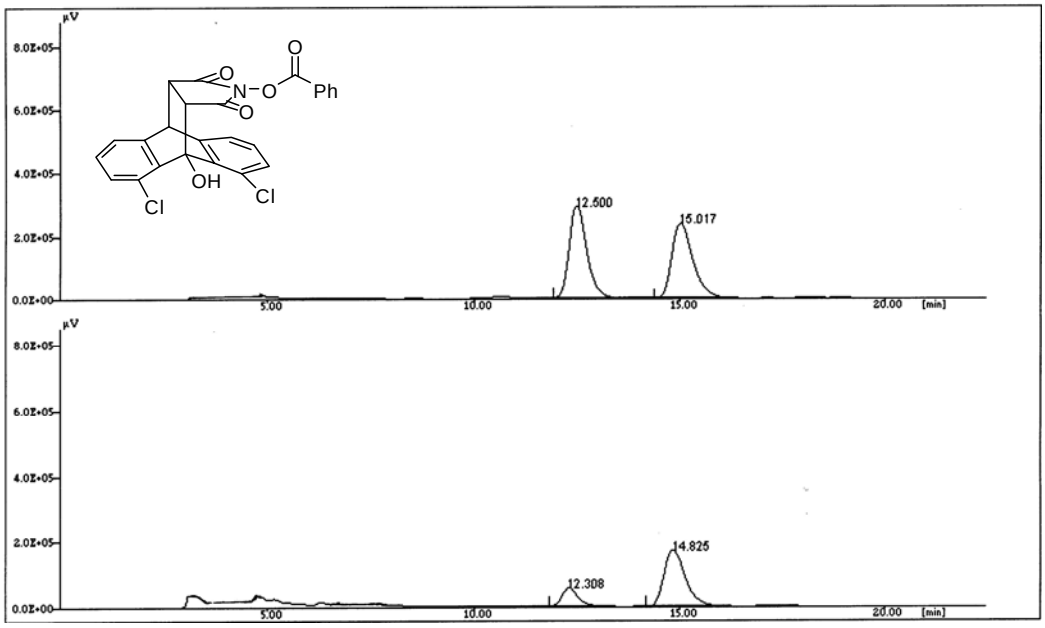


(**4c**) 2-benzoyloxy-5, 13-dichloro-4-hydroxy-3a, 4, 9, 9a-tetrahydro-4, 9-[1', 2'] benzeno -1*H*-benz[*f*]isoindole-1, 3(2*H*)-dione

White solid. mp 176.5 -177.2 ^{o}C. 83% yield, 64% ee. ^{1}H NMR (500 MHz, $CDCl_3$, ppm)： δ 3.40 (dd, 1H, J = 3.1, 8.8Hz), 3.51 (d, 1H, J = 8.8Hz), 4.74 (d, 1H, J = 3.2Hz), 4.90 (s, 1H), 7.14-7.97 (m, 11H). ^{13}C NMR (75 MHz, $CDCl_3$, ppm): δ 41.8, 43.6, 47.4, 80.0, 121.4, 122.9, 123.4, 127.2, 127.4, 127.8, 128.6, 128.7, 129.2, 129.9,

130.0, 133.5, 137.3, 140.2, 167.2, 167.5. IR (film): 1216.0, 1641.9, 3018.7 cm^{-1}. LRMS(FAB) m/z 479.9 (M^+), HRMS(FAB) m/z 480.0400 ($M+H^+$), calc. for $C_{20}H_{13}Cl_2NO_5$ 480.0406.

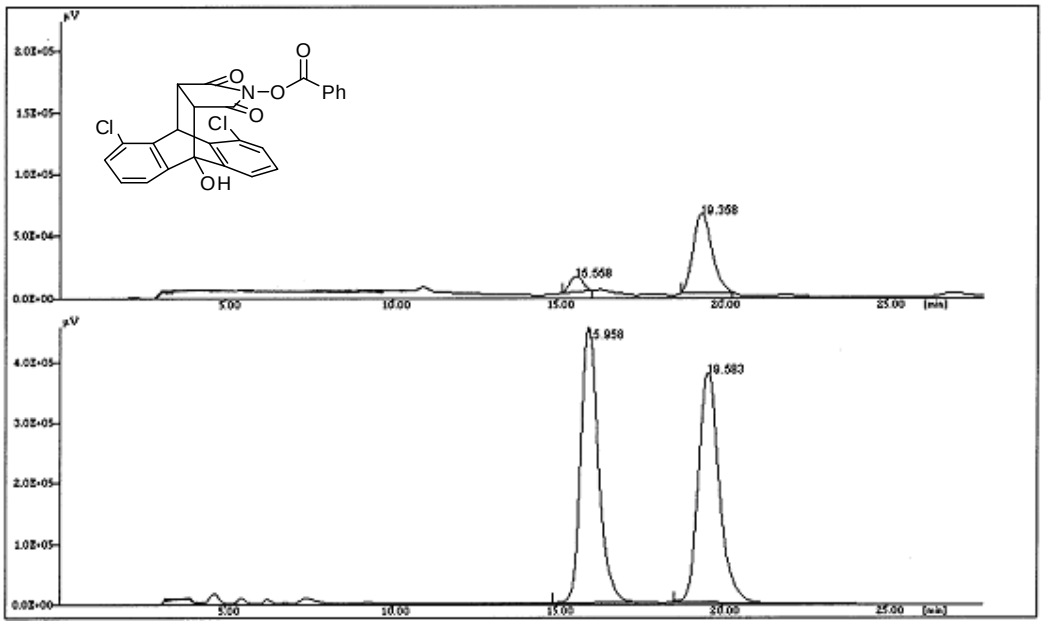
The ee determined by chiral HPLC; CHIRALCEL AD-H (4.6 mm i.d. x 250 mm); hexane/2-propanol 80/20; flow rate 1.0 ml/min; temp 25 °C; detection UV 230 nm; retention time: 12.3 min and 14.8 min.



(**4d**) 8, 10-Dichloro-2-benzoyloxy-4-hydroxy-3a, 4, 9, 9a-tetrahydro-4, 9-[1', 2'] benzeno-1*H*-benz[*f*]isoindole-1, 3(2*H*)-dione

White solid. mp 253.3-254.2 °C. 84% yield, 87% ee. ^1H NMR (500 MHz, $CDCl_3$, ppm)：δ 3.26 (d, 1H, J = 8.8Hz), 3.49 (dd, 1H, J = 3.7, 8.8Hz), 4.35 (s, 1H), 5.92 (d, 1H, J = 3.2Hz), 7.22-7.97 (m, 11H). ^{13}C NMR (125 MHz, $CDCl_3$, ppm): δ 37.0, 43.6, 47.2, 77.2, 119.5, 120.0, 124.7, 127.7, 128.3, 128.6 (two peaks), 128.7, 130.1, 130.5, 131.2, 133.4, 134.9, 142.5, 144.5, 167.8, 170.1. IR (film): 1216.0, 1735.4, 3018.2 cm^{-1}. LRMS (FAB) 480.0 m/z (M^+), HRMS (FAB) m/z 480.0378 ($M+H^+$), calc. for $C_{25}H_{16}Cl_2NO_5$ 480.0406.

The ee determined by chiral HPLC; CHIRALCEL AD-H (4.6 mm i.d. x 250 mm); hexane/2-propanol 80/20; flow rate 1.0 ml/min; temp 25 °C; detection UV 230 nm; retention time: 15.6 min and 19.4 min.



(**5**) 2-Hydroxy-5, 13-dichloro-4-hydroxy-3a, 4, 9, 9a-tetrahydro-4, 9-[1', 2'] benzeno -1*H*-benz[*f*]isoindole-1, 3(2*H*)-dione

White solid. mp 191.0 -191.8 ^{o}C. 92% yield. ^{1}H NMR (500 MHz, $(CD_3)_2CO$, ppm)：δ 3.40 (dd, 1H, J = 3.1, 8.8Hz), 3.43 (d, 1H, J = 8.8Hz), 4.82 (d, 1H, J = 2.5Hz), 4.97 (s, 1H), 7.16-7.49 (m, 6H). ^{13}C NMR (125 MHz, $(CD_3)_2CO$, ppm): δ 42.7, 44.8, 48.3, 81.2, 123.3, 124.2, 128.5, 128.8, 129.3, 129.4, 130.6 (two peaks), 135.9, 137.5, 140.2, 142.7, 170.6, 172.2. IR (film): 1215.9, 1642.0, 3018.2, 3433.0 cm^{-1}. LRMS(ESI) m/z 376.3 (M^+), HRMS(ESI) m/z 397.9957 ($M+Na^+$), calc. for $C_{18}H_{11}Cl_2NO_4Na$ 397.9963.

(**7a**) 1-Acenaphthenol

Yellow solid. 42% yield. ^{1}H NMR (300 MHz, $CDCl_3$, ppm)：δ 3.27 (d, 1H, J = 17.8Hz), 3.82 (dd, 1H, J = 7.4, 17.8Hz), 5.75 (dd, 1H, J = 2.4, 7.3Hz), 7.31-7.78 (m, 6H). LRMS (FAB) m/z 170.1 (M^+).

The ee determined by chiral HPLC; CHIRALPAK IA (4.6 mm i.d. x 250 mm); hexane/2-propanol 95/5; flow rate 0.5 ml/min; temp 25 °C; detection UV 230 nm; retention time: 21.8 min and 23.2 min.

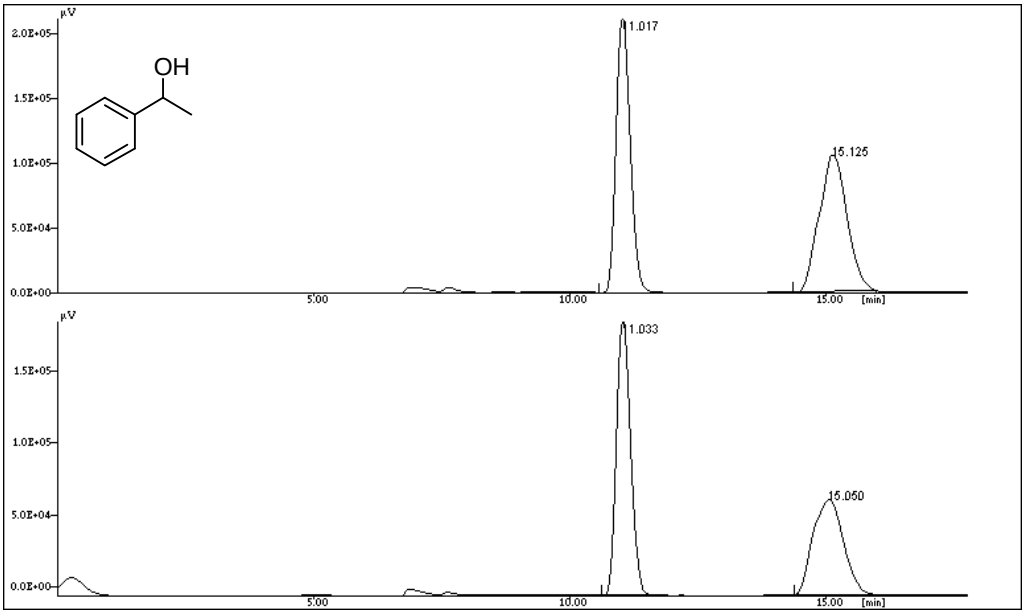
(**9**) 2-Methoxy-2-phenylindan-1-one

White solid. 55% yield. ^{1}H NMR (300 MHz, $CDCl_3$, ppm)：δ 3.34 (s, 3H), 3.59 (s, 2H), 7.29-7.79 (m, 9H). LRMS (FAB) m/z 237.0 ($M-H^+$).

The ee determined by chiral HPLC; CHIRALPAK IA (4.6 mm i.d. x 250 mm); hexane/2-propanol 97/3; flow rate 0.3 ml/min; temp 25 °C; detection UV 230 nm; retention time: 27.5 min and 28.9 min.

(**11**) 1-Phenylethanol

The ee determined by chiral HPLC; CHIRALPAK IA (4.6 mm i.d. x 250 mm); hexane/2-propanol 95/5; flow rate 0.5 ml/min; temp 25 °C; detection UV 230 nm; retention time: 11.0 min and 15.1 min.



(**13**) Benzoin

White solid. 35% yield. ^{1}H NMR (300 MHz, $CDCl_3$, ppm) ：δ 4.57 (d, 1H, J = 6.0Hz), 5.96 (d, 1H, J = 6.3Hz), 7.26-7.93 (m, 10H). LRMS (FAB) m/z 213.0 ($M+H^+$).

The ee determined by chiral HPLC; CHIRALPAK IA (4.6 mm i.d. x 250 mm); hexane/2-propanol 95/5; flow rate 1.0 ml/min; temp 25 °C; detection UV 230 nm; retention time: 40.0 min and 46.5 min.

(**14**) 4-Hydroxy-2-hydroxy -3a, 4, 9, 9a-tetrahydro-4, 9[1', 2']-benzeno-1*H*-benz[*f*]isoindole-1, 3(2*H*)-dione

White solid. mp 225.0 -225.9 ^{o}C. 92% yield. ^{1}H NMR (500 MHz, $(CD_3)_2CO$, ppm)：δ 3.14 (d, 1H, J = 8.8Hz), 3.34 (dd, 1H, J = 3.2, 8.2Hz), 4.76 (d, 1H, J = 3.2Hz), 7.13-7.66 (m, 8H). ^{13}C NMR (125 MHz, $(CD_3)_2CO$, ppm): δ 44.0, 44.7, 47.8, 76.9, 120.5, 121.0, 123.7, 124.5, 126.3, 126.5, 126.6, 126.8, 137.4, 139.9, 141.3, 144.0, 170.9, 171.2. IR (film): 1215.7, 1638.2, 3018.0, 3429.3 cm^{-1}. LRMS(ESI) m/z 306.2 ($M-H^+$), HRMS(ESI) m/z 330.0734 ($M+Na^+$), calc. for $C_{18}H_{13}NO_4Na$ 330.0742.

(**17a**) Methyl 2-hydroxy-3-(2-methyl-1,3-dioxolan-2-yl)propanoate

Colorless liquid. 60% yield. ^{1}H NMR (300 MHz, $CDCl_3$, ppm) ： δ 1.38 (s, 3H), 2.14 (dd, 1H, J = 7.6, 14.8Hz), 2.27 (dd, 1H, J = 3.3, 14.8Hz), 3.77 (s, 3H), 3.99 (m, 4H), 4.40 (m, 1H). LRMS(ESI) m/z 213.0 ($M+Na^+$)

(**17b**) Methyl 3-(1,3-dioxolan-2-yl)-2-hydroxypropanoate

Colorless liquid. 56% yield. ^{1}H NMR (300 MHz, $CDCl_3$, ppm)：δ 2.85 (dd, 1H, J = 6.3, 16.4Hz), 2.92 (dd, 1H, J = 4.4, 16.4Hz), 3.66 (s, 3H), 3.81 (m, 4H), 4.26 (m, 1H), 4.52 (t, 1H, J = 5.4Hz). LRMS(FAB) m/z 177.0 ($M+H^+$)

(**17c**) 2-Hydroxy-3-(2-methyl-1,3-dioxolan-2-yl)propanenitrile

Colorless liquid. 58% yield. ^{1}H NMR (500 MHz, $CDCl_3$, ppm)：δ 1.39 (s, 3H), 2.26 (dd, 1H, J = 5.7, 15.1Hz), 2.44 (dd, 1H, J = 7.6, 15.1Hz), 4.01 (m, 4H), 4.92 (dd, 1H, J = 5.6, 7.6Hz). LRMS(ESI) m/z 176.1 ($M+NH_4^+$)

(**17d**) 3-(1,3-Dioxolan-2-yl)-2-hydroxypropanenitrile

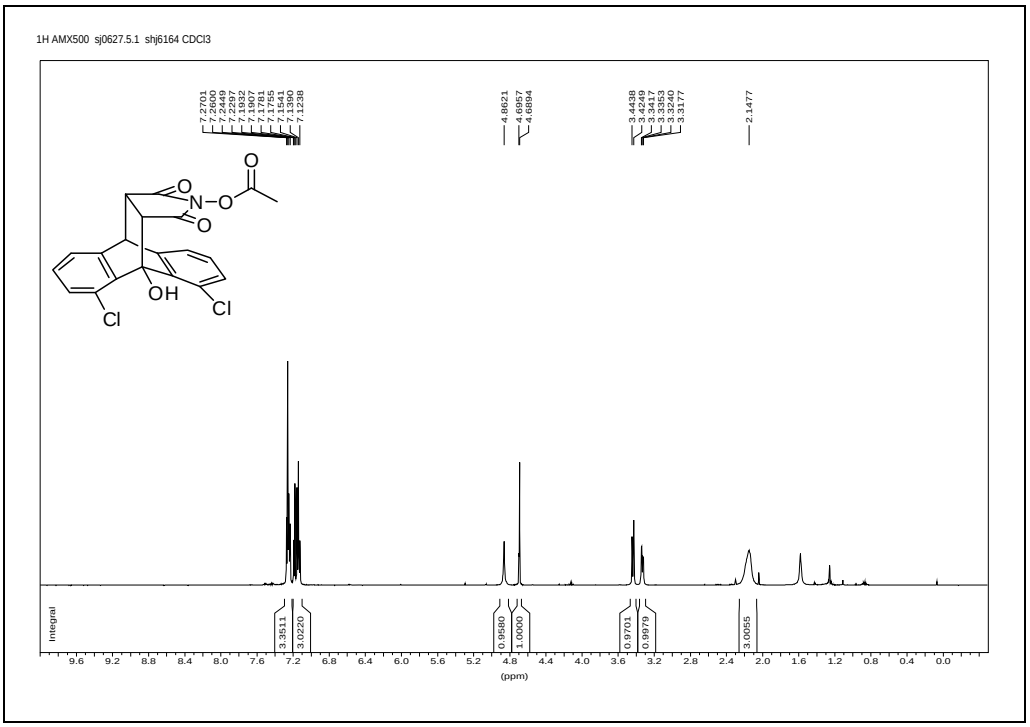
Colorless liquid. 52% yield. ^{1}H NMR (300 MHz, $CDCl_3$, ppm)：δ 2.21 (m, 1H), 2.32 (m, 1H), 3.67 (1H, d, 1H, J = 6.6Hz), 3.92-4.09 (m, 4H), 4.74 (dd, 1H, J = 3.8, 6.6Hz), 5.18 (dd, 1H, J = 3.5, 5.2Hz). LRMS(EI) m/z 142.1 ($M-H^+$)

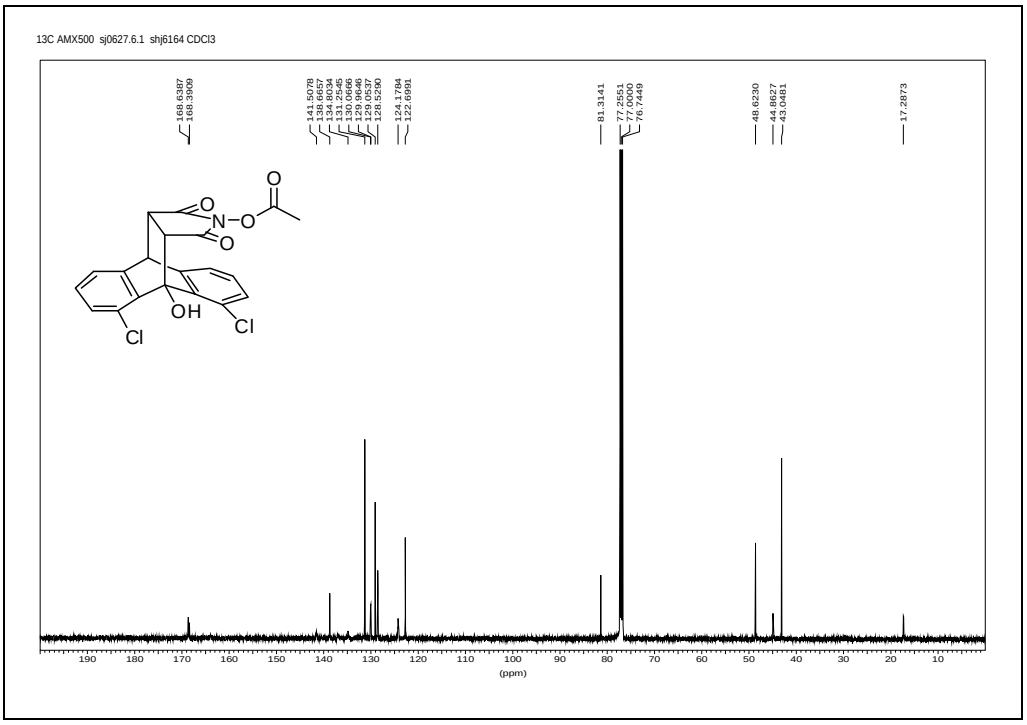
(**19**) Dihydro-3-hydroxy-5,5-dimethyl-2(3*H*)-furanone

Colorless liquid. 65% yield. ^{1}H NMR (300 MHz, $CDCl_3$, ppm)：δ 1.39 (s, 3H), 1.50 (s, 3H), 2.04 (dd, 2H, J = 2.8, 9.1Hz), 2.50(dd, 1H, J = 8.7, 12.9Hz), 4.65 (t, 4H, J = 9.1). LRMS(FAB) m/z 147.1 ($M+NH_4^+$)

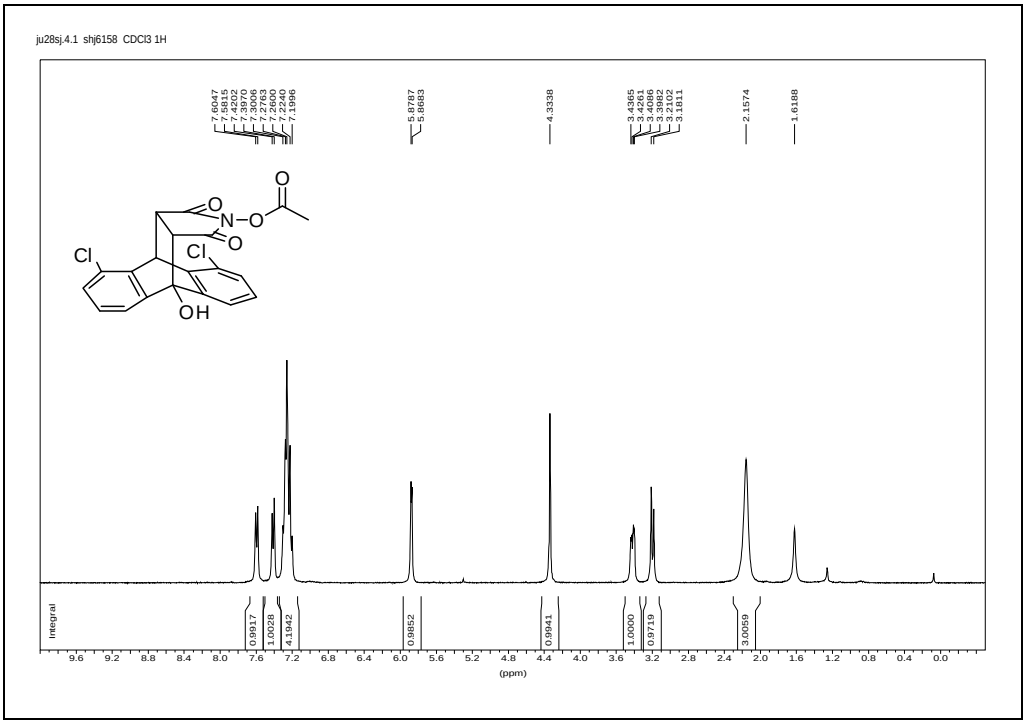
4. Copies of NMR spectrum

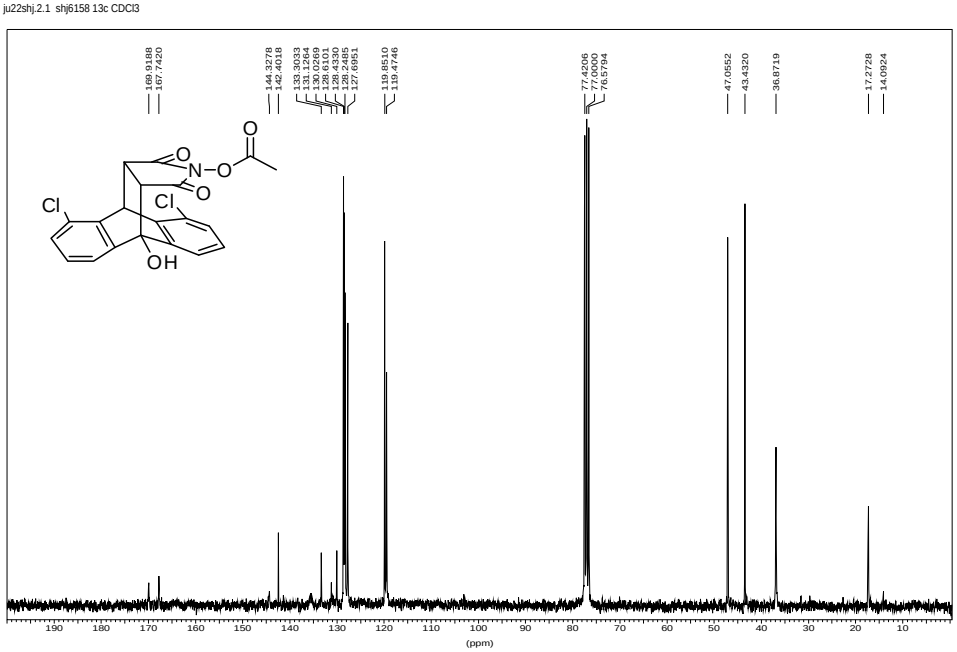
(4a)



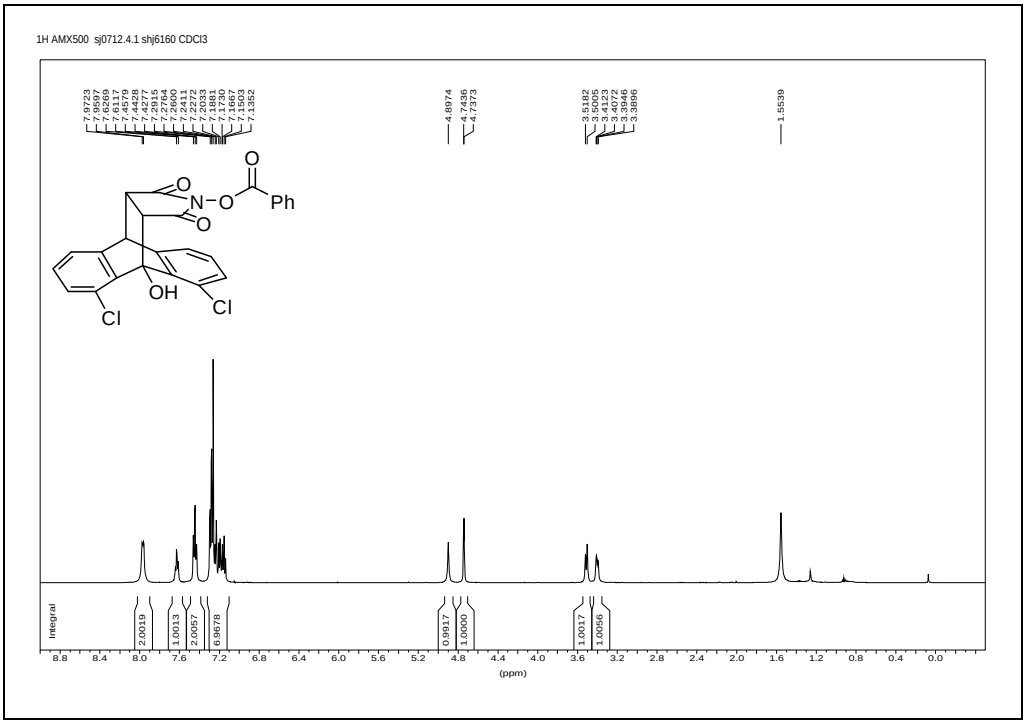


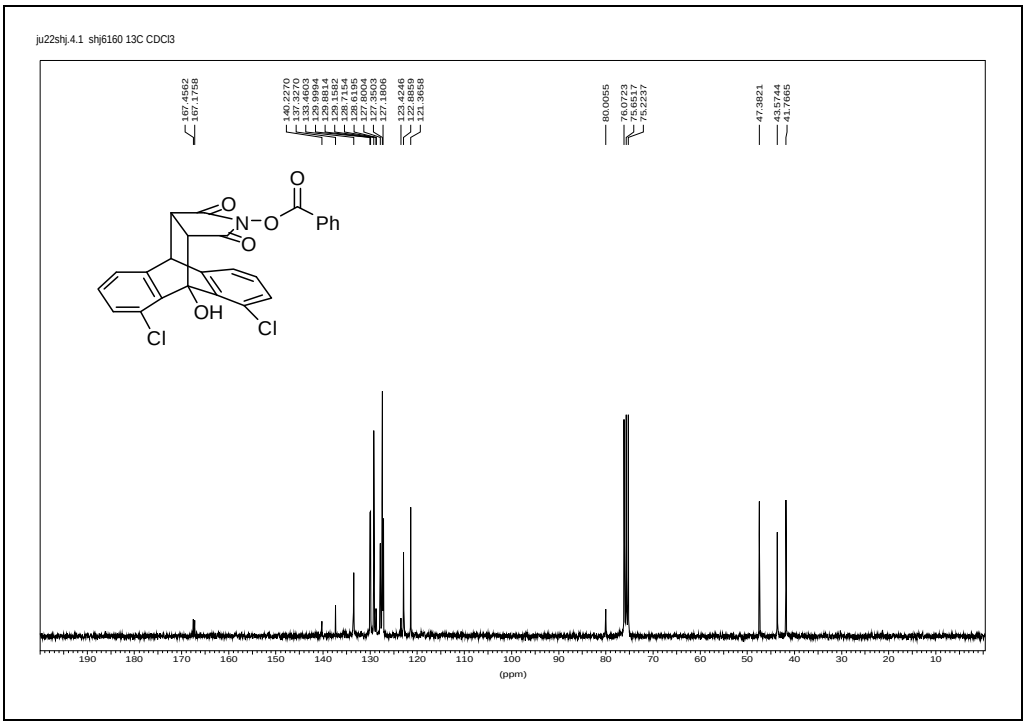
(**4b**)


ju28sj.4.1 shj6158 CDCl3 1H
7.6047
7.5815
7.4202
7.3970
7.3096
7.2763
7.2600
7.2240
7.1996
5.8787
5.8683
4.3338
3.4365
3.4261
3.4086
3.3982
3.2102
3.1811
2.1574
1.6188
Integral
0.9917
1.0028
4.1942
0.9852
0.9941
1.0000
0.9719
3.0059
(ppm)

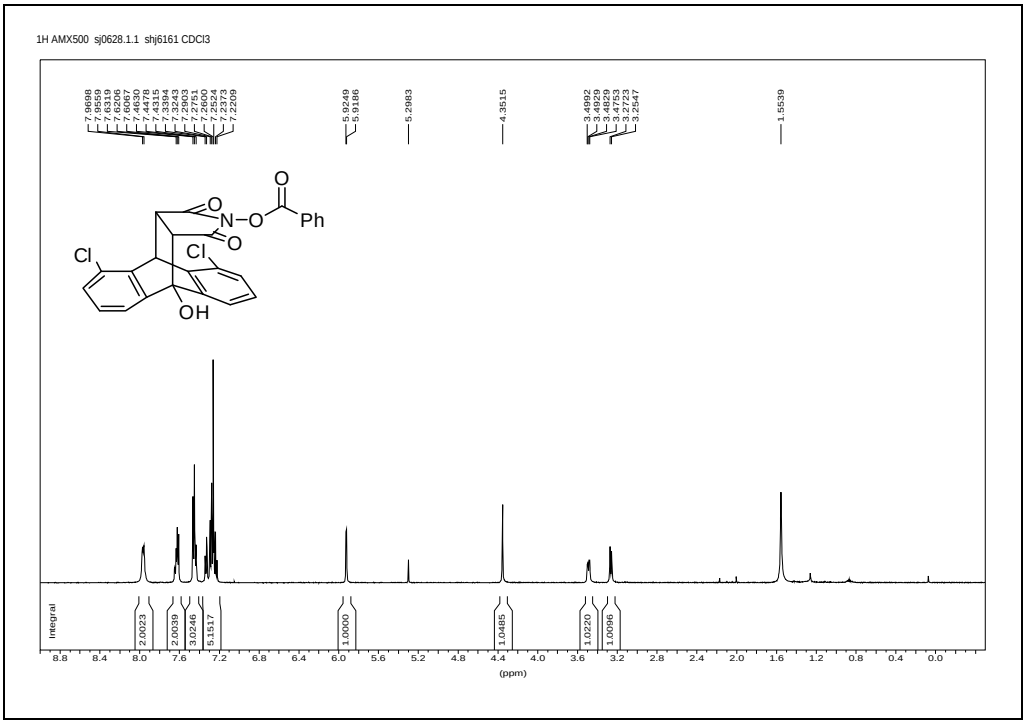

ju22shj.2.1 shj6158 13c CDCl3
169.9188
167.7420
144.3278
142.4018
133.3033
131.3764
130.0269
128.6101
128.4340
128.2485
127.6951
119.8510
119.4746
77.4206
77.0000
76.5794
47.0552
43.4320
36.8719
17.2728
14.0924
(ppm)

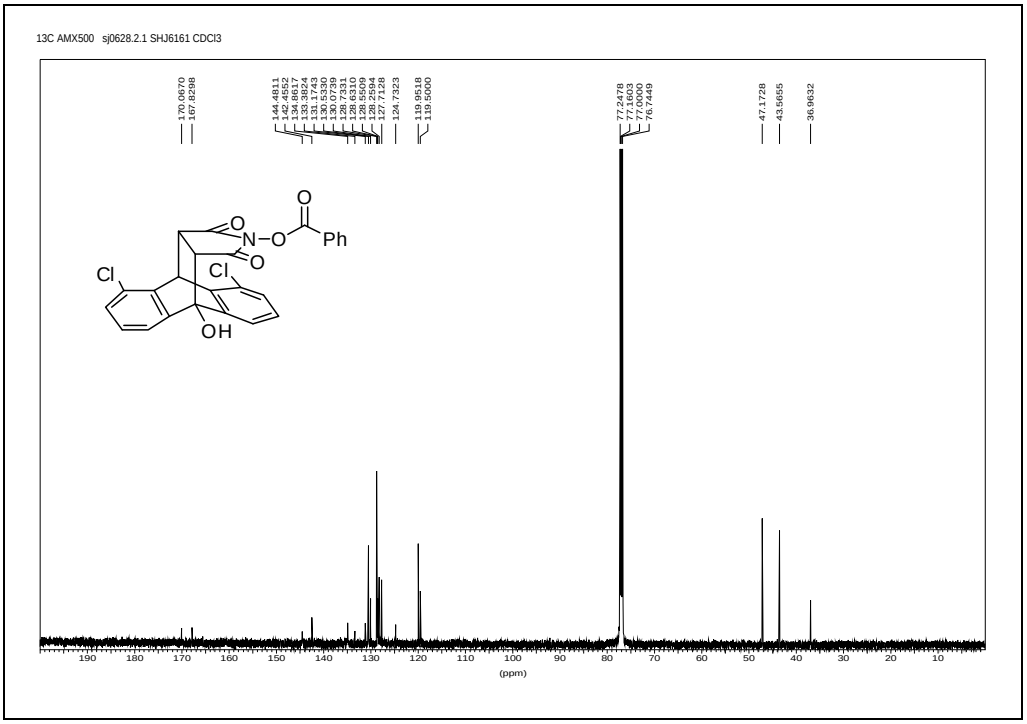
(**4c**)


1H AMX500 sj0712.4.1 shj6160 CDCl3
7.9723
7.9597
7.6269
7.6117
7.4579
7.4428
7.4277
7.2915
7.2764
7.2600
7.2411
7.2272
7.2033
7.1881
7.1730
7.1667
7.1553
7.1382
4.8974
4.7436
4.7373
3.5182
3.5005
3.4823
3.4072
3.3946
3.3896
1.5539
O
N−O
Ph
O
O
OH
Cl
Cl
Integral
2.0019
1.0013
2.0057
6.9678
0.9917
1.0000
1.0017
1.0056
(ppm)

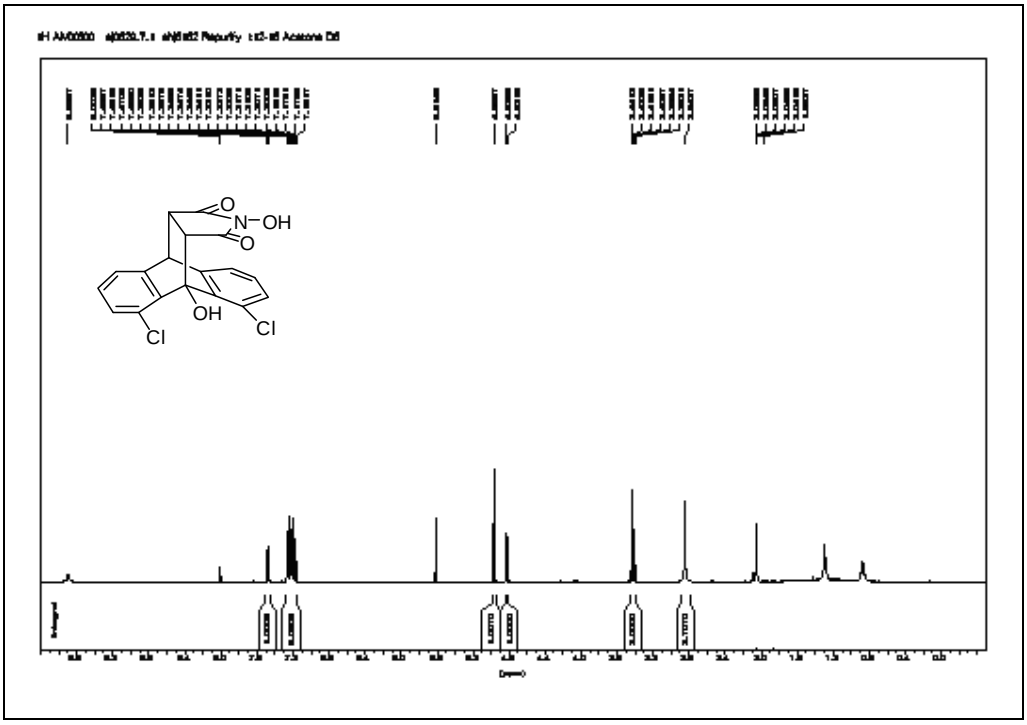

ju22shj.4.1 shj6160 13C CDCl3
167.4562
167.1758
140.2270
137.2370
133.4603
129.9994
129.4814
129.1852
128.7154
128.6135
127.9064
127.3503
127.1806
123.4246
122.8859
121.3868
80.0055
76.0723
75.6537
75.2537
47.3821
43.5744
41.7665
O
N−O
Ph
O
O
OH
Cl
Cl
(ppm)

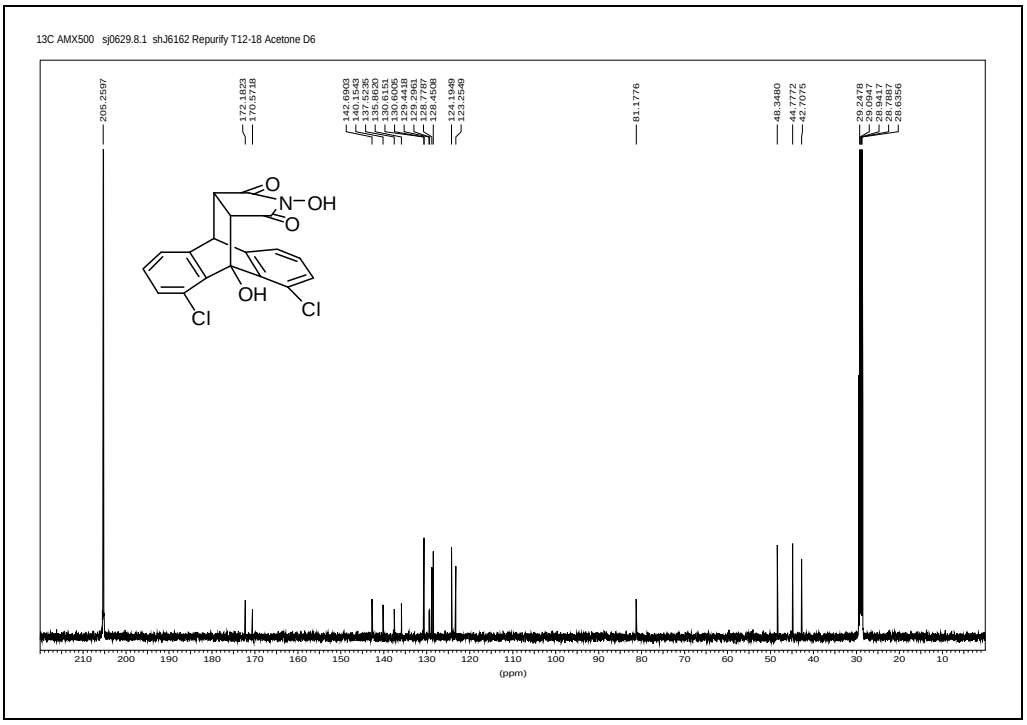
(**4d**)


1H AMX500 sj0628.1.1 shj6161 CDCl3
7.9698
7.9569
7.6319
7.6206
7.6057
7.4630
7.4478
7.4315
7.3394
7.3243
7.2903
7.2751
7.2600
7.2524
7.2373
7.2209
5.9249
5.9186
5.2983
4.3515
3.4992
3.4929
3.4829
3.4763
3.2723
3.2547
1.5539
O
N–O
Ph
O
O
Cl
Cl
OH
Integral
2.0023
2.0039
3.0246
5.1517
1.0000
1.0485
1.0220
1.0096
8.8 8.4 8.0 7.6 7.2 6.8 6.4 6.0 5.6 5.2 4.8 4.4 4.0 3.6 3.2 2.8 2.4 2.0 1.6 1.2 0.8 0.4 0.0
(ppm)

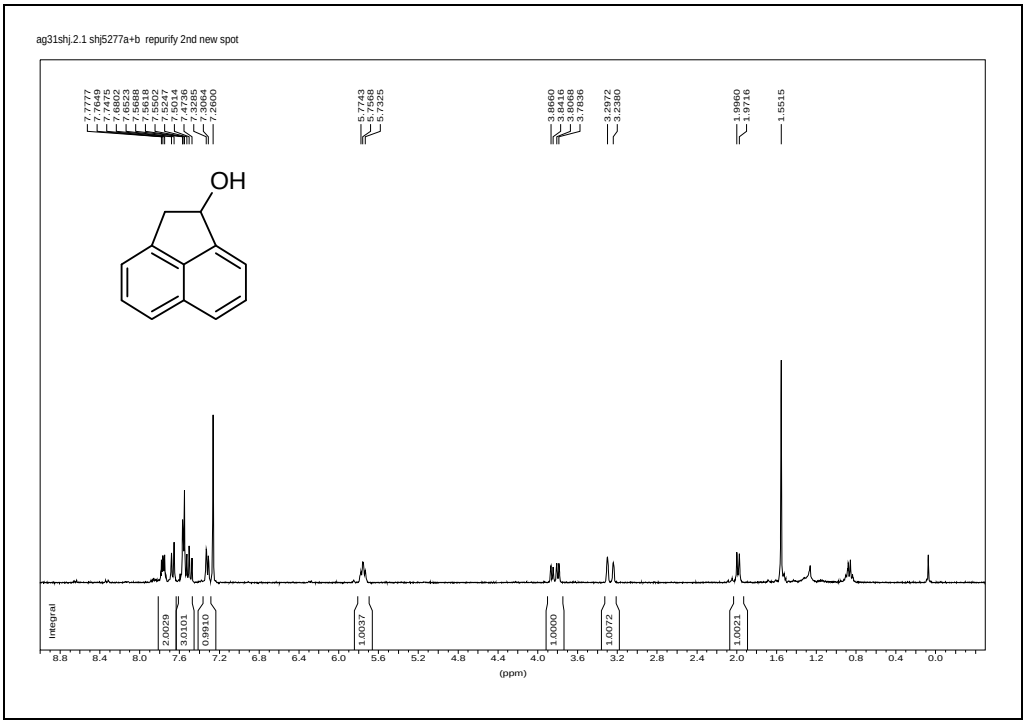

13C AMX500 sj0628.2.1 SHJ6161 CDCl3
170.0670
167.8298
144.4811
142.4552
134.1657
133.3824
131.1743
130.9589
130.0739
128.7331
128.5910
128.5509
128.2594
127.7128
124.7323
119.9518
119.5000
77.2478
77.1663
77.0000
76.7449
47.1728
43.5655
36.9632
O
N–O
Ph
O
O
Cl
Cl
OH
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
(ppm)

(5)

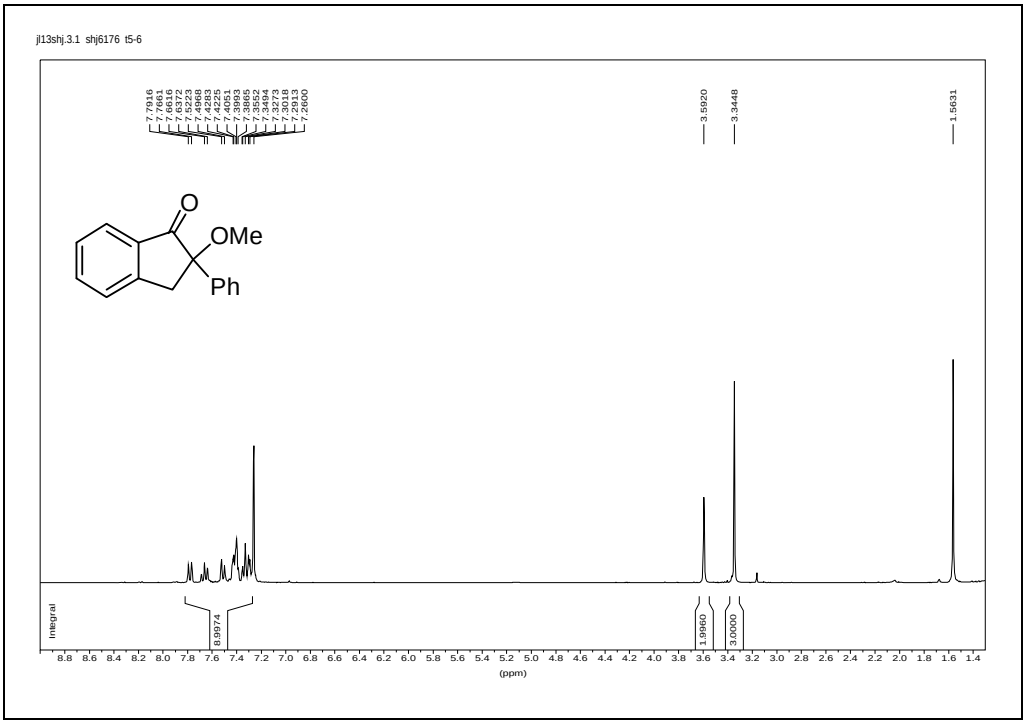

O
N–OH
O
OH
Cl
Cl


13C AMX500 sj0629.8.1 shJ6162 Repurify T12-18 Acetone D6
205.2597
172.1823
170.5738
142.6903
140.1543
137.7835
135.8620
130.6151
130.0095
129.4418
129.2961
128.7787
128.4548
124.1049
123.2548
81.1776
48.3480
44.7772
42.7075
29.2478
29.0647
28.9417
28.7887
28.6356
O
N–OH
O
OH
Cl
Cl
(ppm)

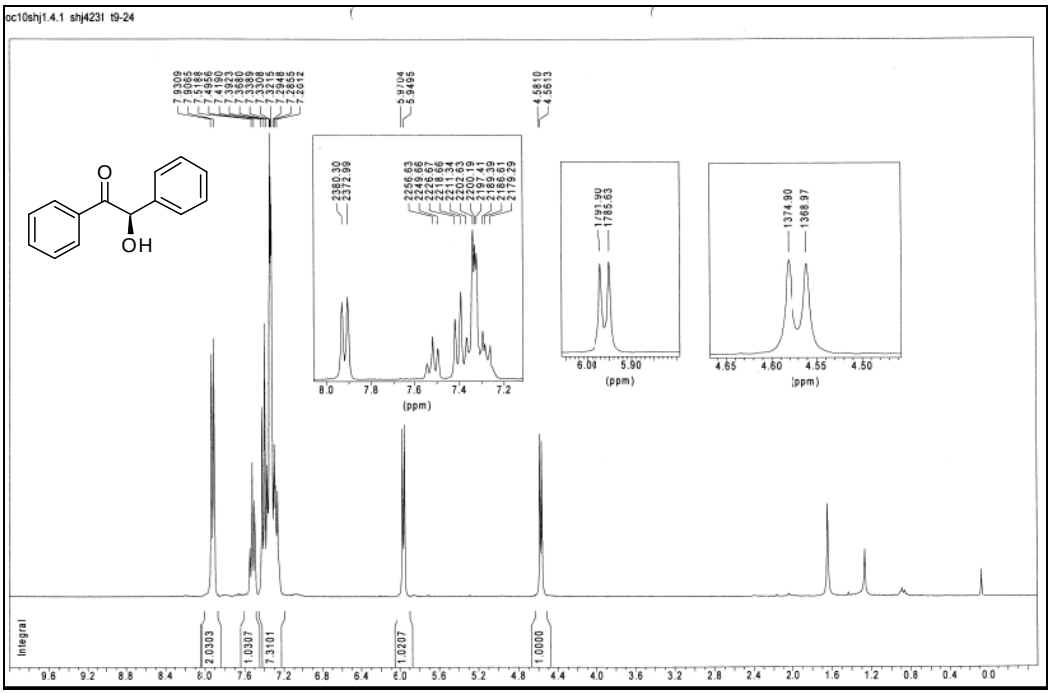
(**7**)


ag31shj.2.1 shj5277a+b repurify 2nd new spot
7.7777
7.7649
7.7475
7.6802
7.6653
7.5688
7.5618
7.5502
7.5247
7.5014
7.4786
7.3285
7.3064
7.2600
5.7743
5.7568
5.7325
3.8660
3.8515
3.8068
3.7836
3.2972
3.2380
1.9960
1.9716
1.5515
OH
Integral
2.0029
3.0101
0.9910
1.0037
1.0000
1.0072
1.0021
(ppm)

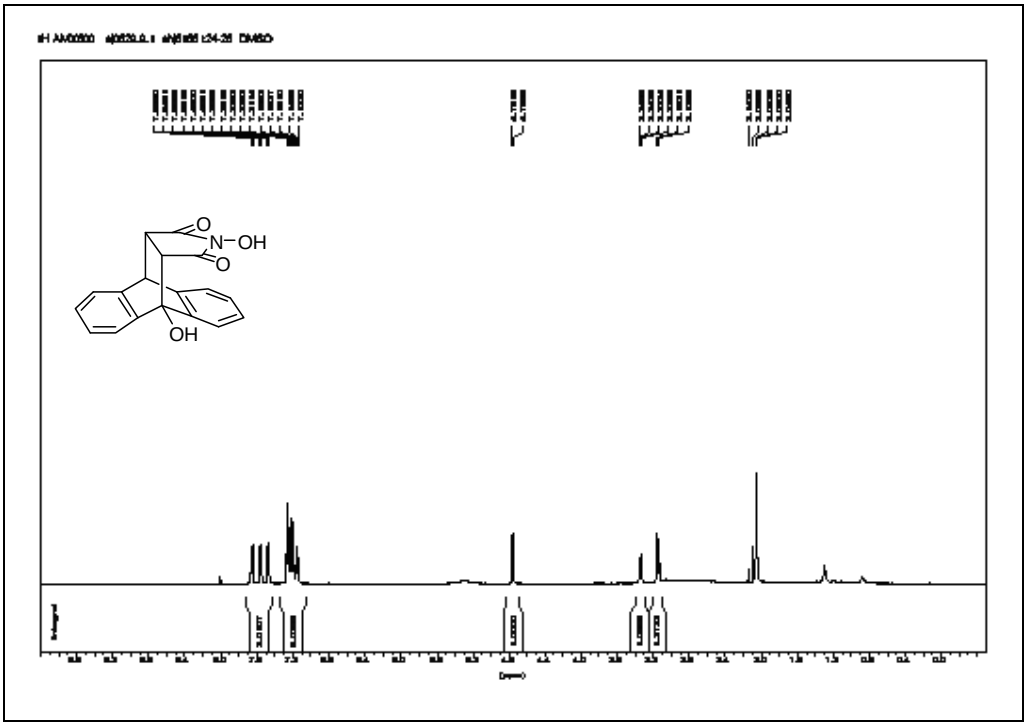
(**9**)

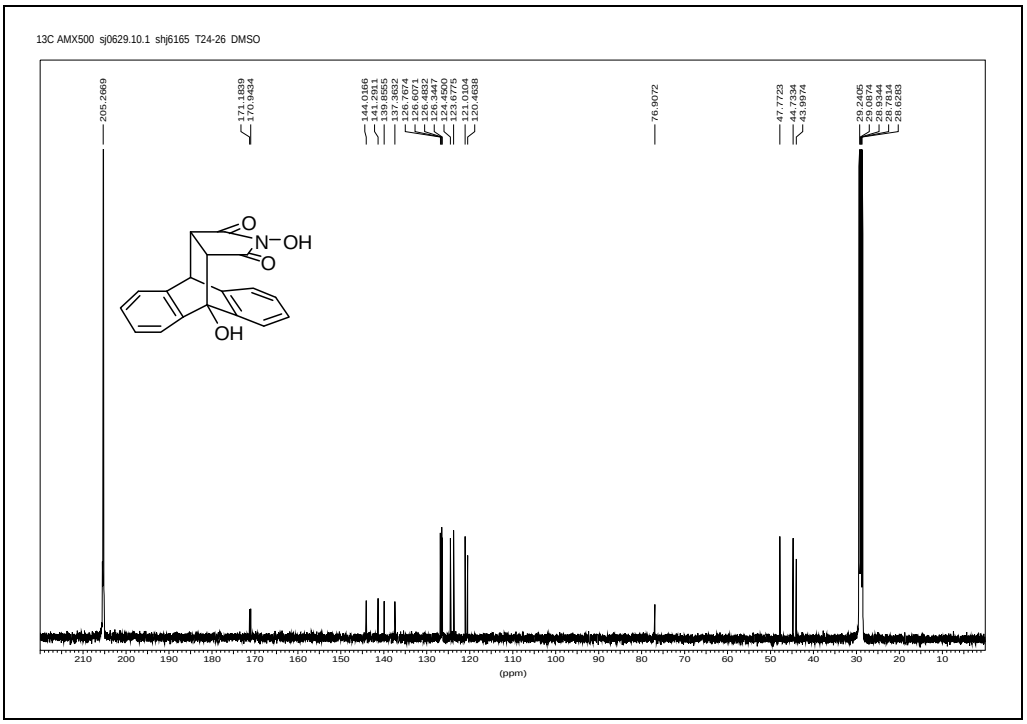

jl13shj.3.1 shj6176 t5-6
7.7916
7.7661
7.6516
7.6372
7.5223
7.4968
7.4283
7.4225
7.4051
7.3993
7.3865
7.3552
7.3464
7.3273
7.3018
7.2913
7.2600
3.5920
3.3448
1.5631
O
OMe
Ph
Integral
8.9974
1.9960
3.0000
(ppm)

(**13**)

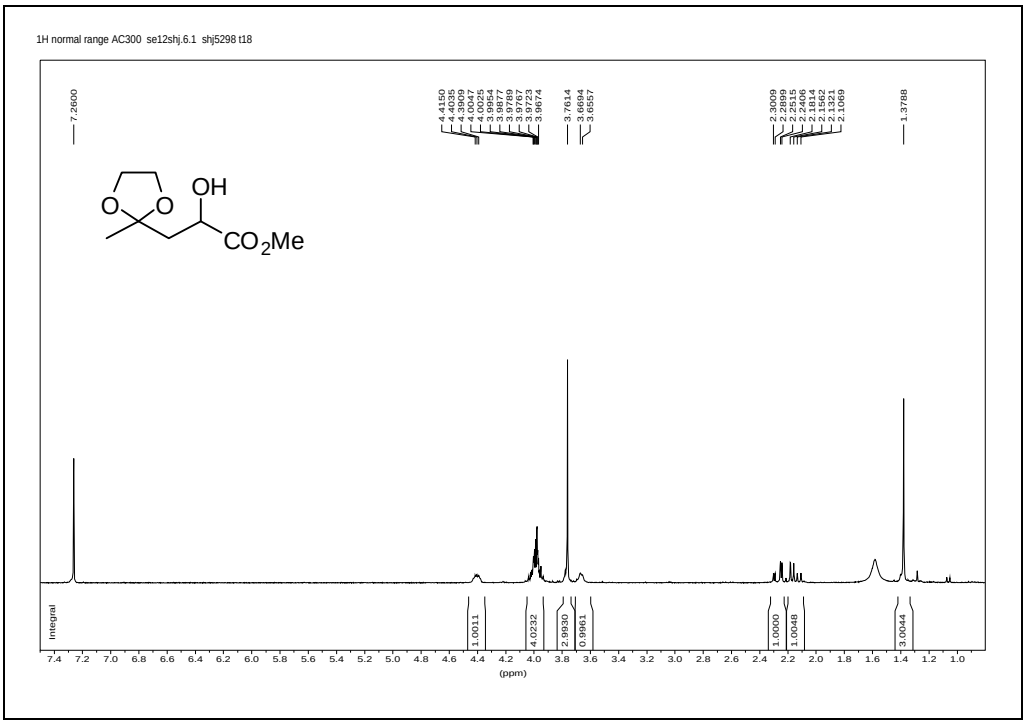

oc10shj1.4.1 shj4231 t9-24
O
OH
Integral
2.0303
1.0307
7.3101
1.0207
1.0000
(ppm)

(**14**)

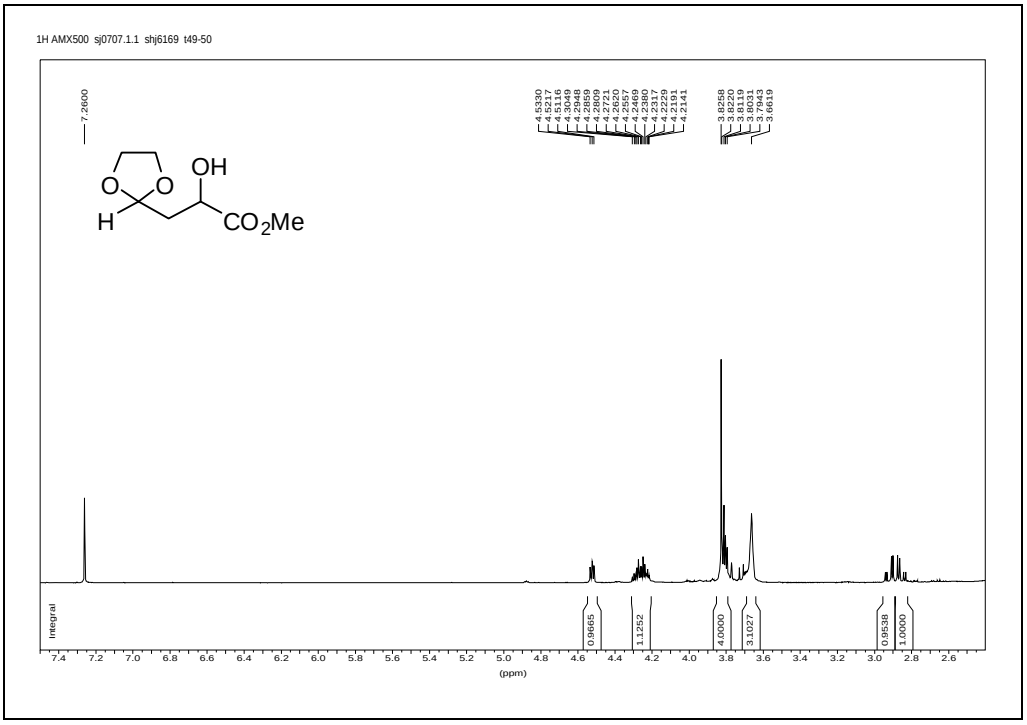

O
N–OH
O
OH


13C AMX500 sj0629.10.1 shj6165 T24-26 DMSO
205.2669
171.1899
170.9434
144.0166
141.2911
139.8555
137.7693
136.7674
126.6071
126.4832
125.9447
124.4500
123.6775
121.7034
120.4658
76.9072
47.7723
44.7334
43.9974
29.2405
29.0694
28.9344
28.7814
28.0283
O
N−OH
O
OH
(ppm)

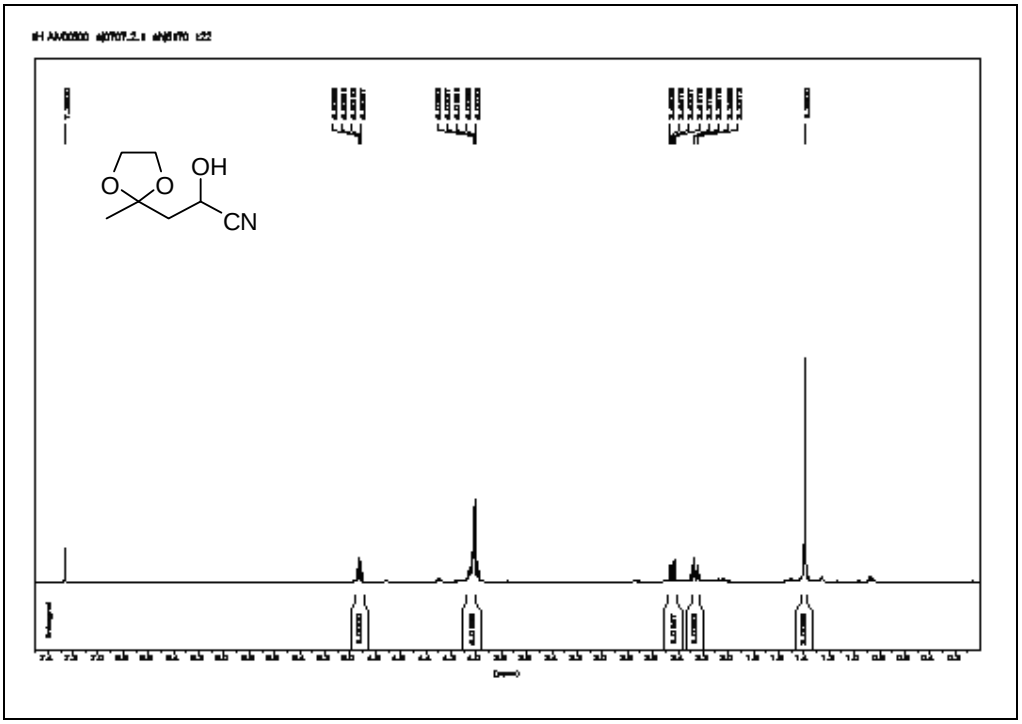
(**17a**)


1H normal range AC300 se12shj.6.1 shj5298 t18
7.2600
4.4150
4.4035
4.3938
4.0047
4.0025
3.9864
3.9877
3.9789
3.9657
3.9723
3.9674
3.7614
3.6694
3.6557
2.3009
2.2899
2.2515
2.2406
2.2114
2.1562
2.1321
2.1069
1.3788
OH
O
O
CO2Me
integral
1.0011
4.0232
2.9930
0.9961
1.0000
1.0048
3.0044
(ppm)

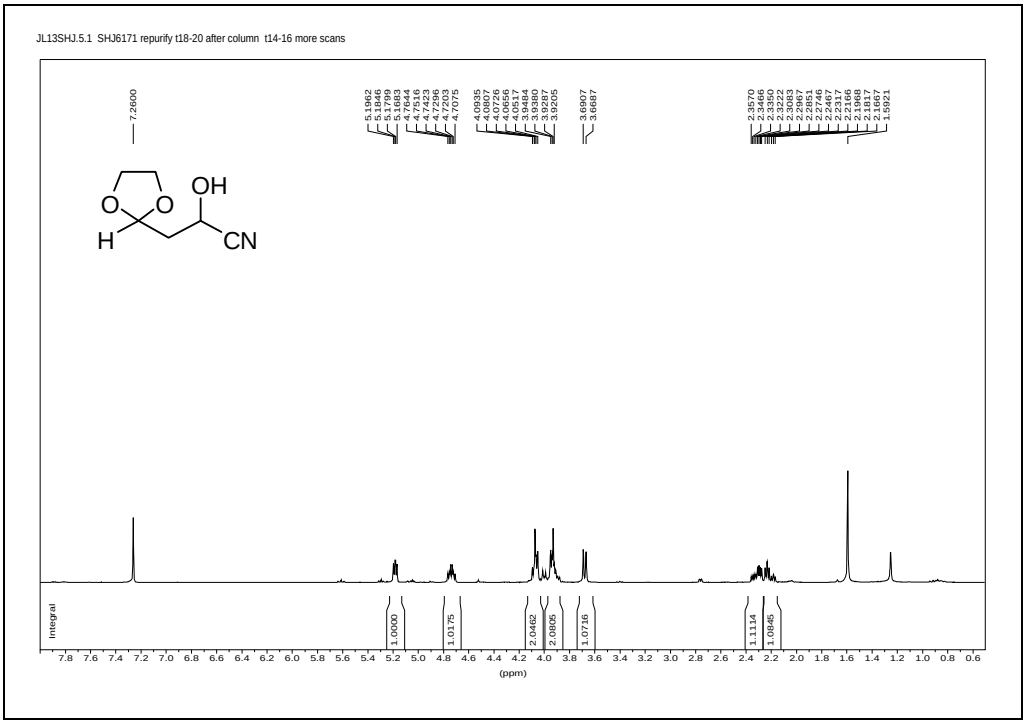
(**17b**)


1H AMX500 sj0707.1.1 shj6169 t49-50
7.2600
4.5330
4.5177
4.5116
4.3049
4.2948
4.2869
4.2809
4.2721
4.2650
4.2557
4.2469
4.2380
4.2317
4.2229
4.2141
4.2141
3.8258
3.8170
3.8119
3.8031
3.7943
3.6618
OH
O
O
H
CO2Me
integral
0.9665
1.1252
4.0000
3.1027
0.9538
1.0000
(ppm)

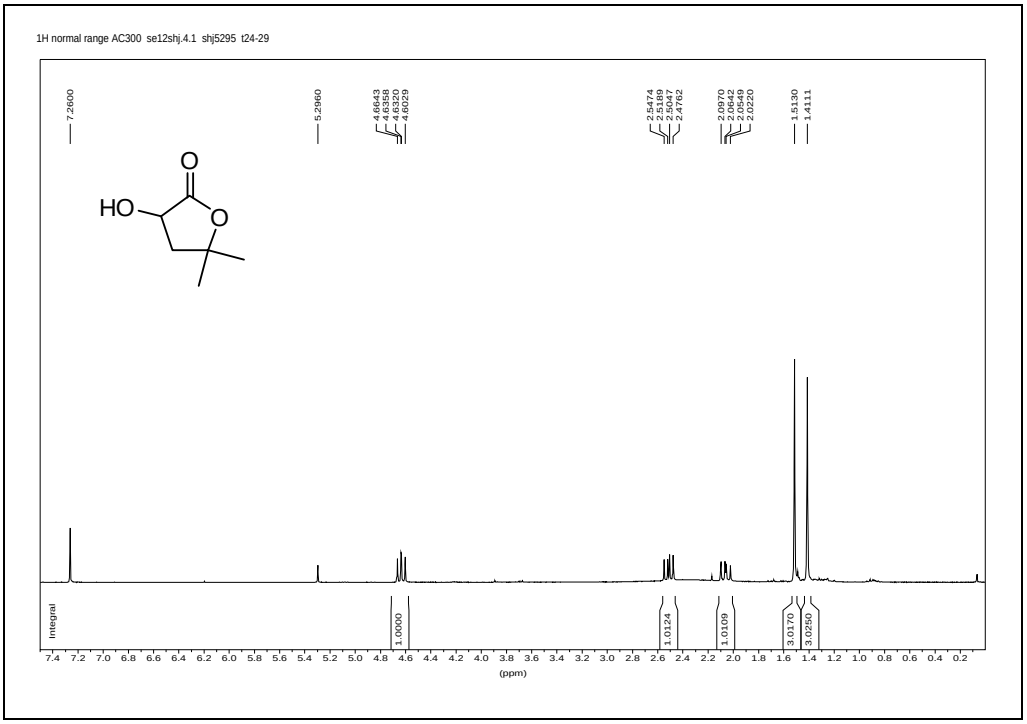
(**17c**)


OH
O
O
CN

(**17d**)


JL13SHJ.5.1 SHJ6171 repurify t18-20 after column t14-16 more scans
7.2600
5.1962
5.1646
5.1799
5.1683
4.7964
4.7516
4.7423
4.7296
4.7203
4.7075
4.0935
4.0807
4.0726
4.0646
4.0517
3.9484
3.9367
3.9287
3.9205
3.6907
3.6687
2.3570
2.3466
2.3350
2.3222
2.3083
2.2987
2.2851
2.2746
2.2647
2.2317
2.2166
2.1969
2.1817
2.1667
1.6921
OH
O
O
H
CN
integral
1.0000
1.0175
2.0462
2.0805
1.0716
1.1114
1.0845
(ppm)

(**19**)


1H normal range AC300 se12shj.4.1 shj5295 t24-29
7.2600
5.2960
4.6643
4.6358
4.6320
4.6020
2.5474
2.5330
2.5047
2.4762
2.0970
2.0842
2.0548
2.0220
1.5130
1.4111
O
HO
O
integral
1.0000
1.0124
1.0109
3.0170
3.0250
(ppm)