One-Pot Synthesis of Novel Photochormic Oxazine Compounds

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Supplementary Material

General information:

All reactions were performed using oven dried glassware. All chemicals were purchased from Aldrich and Fluka, and used as received. For flash chromatography technical grade solvents were used, which were distilled prior to use. Chromatographic purification was performed as flash chromatography using Brunschwig silica 32-63, 60 Å, with 0.3-0.5 bar pressure.
^{1}H NMR spectra were recorded on a VARIAN Mercury 300 MHz spectrometer in chloroform-d, all signals are reported in ppm with the internal chloroform signal at 7.26 ppm. The data are reported as (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, coupling constant(s), integration). ^{13}C NMR spectra were recorded with ^{1}H decoupling on a VARIAN Mercury 75 MHz spectrometer in chloroform-d, all signals are reported in ppm with the internal chloroform signal at 77.0 ppm as standard.
Infrared spectra were recorded on a Perkin Elmer Spectrum RX-I FT-IR spectrophotometer as thin films or as solution in chloroform and are reported as cm^{-1}.
Mass spectrometric measurements were performed by the mass spectrometry service of the LOC at the ETHZ.
UV-Vis spectra were recorded in Varian Cary 50 UV-Vis spectrometer.
ReactIR spectra were recorded in ReactIR™ 4000 (Mettler Toledo).

General procedure: A mixture of disubstituted acrylic acid (1.0 mmol), DPPA (95%, 348 mg, 1.2 mmol), Et_3N (506 mg, 5.0 mmol), 9,10-phenanthrenequinone (146 mg, 0.7 mmol) and Ph_3AsO (16 mg, 0.05 mmol) in toluene (12 mL) was maintained at 60 °C over 3 h. Solvent was removed *in vacuo*. The photochromic oxazine compound was purified by chromatography (silica gel, dichloromethane-hexane as eluent) and recrystallization (from dichloromethane-hexane).

Characterization data for oxazine compounds:
2a: 2,2-diphenyl-phenanthro (9,10)-[2*H*]-[1,4]-oxazine
M.p. 185.7-186.5 ºC. ^{1}H NMR (300 MHz, $CDCl_3$): δ 8.66-8.53 (m, 3H), 8.52-8.43 (m, 1H), 8.12 (s, 1H), 7.70-7.46 (m, 8H), 7.29-7.24 ppm (m, 6H). ^{13}C NMR (75MHz, $CDCl_3$): δ 155.7, 141.4, 138.0, 131.3, 129.8, 128.6, 128.4, 127.6, 127.3, 127.1, 126.9, 126.9, 125.2, 125.1, 123.0, 122.8, 122.7, 122.5, 79.5 ppm. IR (KBr): ν = 3056 (w), 3028 (w), 1609 (w), 1577 (m), 1488 (m), 1448 (m), 1320 (m), 1230 (m), 1111 (m), 1018 (m), 753 (s), 723 (m), 697 (s) cm^{-1}. HRMS (MALDI) Calcd for $C_{28}H_{19}NO$: m/z (%): 386.1539 (MH^+). Found: 386.1535 (100%).

2b: 2-(*p*-methoxyphenyl)-2-phenyl-phenanthro (9,10)-[2*H*]-[1,4]-oxazine
M.p. 167.2-167.9 ºC. ^{1}H NMR (300 MHz, $CDCl_3$): δ 8.62-8.53 (m, 3H), 8.52-8.44 (m, 1H), 8.07 (s, 1H), 7.66-7.61 (m, 3H), 7.60-7.50 (m, 3H), 7.42-7.29 (m, 5H), 6.80-6.60 (m, 2H), 3.74 ppm (s, 3H). ^{13}C NMR (75MHz, $CDCl_3$): δ 159.7, 155.9, 141.6, 138.0, 133.3, 131.2, 129.8, 128.6, 128.3, 127.5, 127.3, 127.0, 126.9, 126.8, 125.2, 125.1, 122.9, 122.8, 122.8, 122.7, 122.5, 114.0, 79.4, 55.2 ppm. IR (KBr): ν = 3061 (w), 3031 (w), 2954 (w), 1608 (m), 1581 (m), 1511 (m), 1448 (m), 1254 (m), 1237 (m), 1016 (s), 830 (m), 752 (m), 701 (m) cm^{-1}. HRMS (MALDI) Calcd for $C_{29}H_{20}NO_2$: m/z (%): 416.1645 (MH^+). Found: 416.1642 (100%).

2c: 2-(*p*-fluorophenyl)-2-phenyl-phenanthro (9,10)-[2*H*]-[1,4]-oxazine
M.p. 140.8-141.8 ºC. ^{1}H NMR (300 MHz, $CDCl_3$): δ 8.64-8.55 (m, 3H), 8.46-8.44 (m, 1H), 8.01 (s, 1H), 7.71-7.60 (m, 3H), 7.60-7.52 (m, 1H), 7.52-7.44 (m, 2H), 7.42-7.37 (m, 2H), 7.08-6.98 (m, 2H), 6.88-6.82 (m, 2H), 3.76 ppm (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 162.3 (^{1}J = 247.2 Hz), 160.7, 159.5, 155.2, 137.6, 137.1, 132.7, 131.0, 129.4, 128.8 (^{3}J = 7.9 Hz), 128.3, 127.5, 127.2, 126.7, 125.0, 124.9, 122.8, 122.6, 122.5 (^{2}J = 24.8 Hz), 122.5, 115.5, 115.2, 113.9, 79.0, 55.3 ppm. IR (KBr): ν = 3065 (w), 2933 (w), 2841 (w), 1608 (m), 1581 (m), 1510 (s), 1258 (m), 1240 (s), 1160 (m), 1016 (m), 830 (m), 751 (m), 721 (m) cm^{-1}. HRMS (MALDI) Calcd for $C_{29}H_{20}FNO_2$: m/z (%): 434.1551 (MH^+). Found: 434.1548 (100%).

2d: 2,2-di-(*p*-methoxyphenyl)-phenanthro (9,10)-[2*H*]-[1,4]-oxazine
M.p. 152.4-153.4 ºC. ^{1}H NMR (300 MHz, $CDCl_3$): δ 8.63-8.54 (m, 3H), 8.47-8.43 (m, 1H), 8.03 (s, 1H), 7.68 -7.62 (m, 3H), 7.60-7.52 (m, 1H), 7.45-7.35 (m, 4H), 6.90-6.80 (m, 4H), 3.80-3.70 (m, 4H), 3.07-2.98 (m, 4H), 3.75 ppm (s, 6H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 159.7, 156.1, 133.5, 131.2, 129.8, 128.5, 127,5, 127.3, 126.8, 125.1, 122.9, 122.8, 122.7, 122.5, 114.0, 79.3, 55.2 ppm; IR (KBr): ν = 3073 (w), 3002 (w), 2983 (w), 2831 (w), 1608 (m), 1586 (m), 1509 (s), 1450 (m), 1300 (m), 1252 (s), 1172 (m), 1116 (m), 1023 (m), 831 (m), 757 (m), 725 (m) cm^{-1}. HRMS (MALDI) Calcd for $C_{30}H_{23}NO_3$: m/z (%): 446.1751 (MH^+). Found: 446.1747 (100%).

2e: 2-(*p*-methoxyphenyl)-2-(*p*-morphlinophenyl)-phenanthro (9,10)-[2*H*]-[1,4]-oxazine
M.p. 135.0-136.0 ºC. ^{1}H NMR (300 MHz, $CDCl_3$): δ 8.69-8.52 (m, 3H), 8.53-8.43 (m, 1H), 8.04 (s, 1H), 7.71 -7.60 (m, 3H), 7.60-7.50 (m, 1H), 7.46-7.38 (m, 2H), 7.38-7.30 (m, 2H), 6.89-6.80 (m, 2H), 6.77-6.68 (m, 2H), 3.75 ppm (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 159.5, 156.2, 150.9, 138.1, 133.5, 132.0, 129.8, 128.5, 128.1, 127.4, 127.3, 126.8, 125.3, 125.0, 122.8, 122.7, 122.6, 122.5, 114.9, 113.8, 79.2, 66.6, 55.1, 48.4, 29.6 ppm. IR (KBr): ν = 3069 (w), 2956 (w), 2823 (w), 1608 (m), 1512 (s), 1450 (m), 1253 (m), 1228 (m), 1120 (m), 927 (m), 821 (m), 756 (m) cm^{-1}. HRMS (MALDI) Calcd for $C_{33}H_{28}N_2O_3$: m/z (%): 499.2016 ($M\text{-}H_2^+$). Found: 499.2013 (100%).

2f: 2-(*p*-methoxyphenyl)-2-(*p*-piperidinophenyl)-phenanthro (9,10)-[2*H*]-[1,4]-oxazine
M.p. 134.5-136.0 ºC. ^{1}H NMR (300 MHz, $CDCl_3$): δ 8.63-8.54 (m, 3H), 8.50-8.40 (m, 1H), 8.02 (s, 1H), 7.70 -7.50 (m, 4H), 7.45-7.38 (m, 2H), 7.36-7.29 (m, 2H), 6.89-6.80 (m, 4H), 3.76 (s, 3H), 3.18-3.08 (m, 4H), 1.72-1.60 (m, 4H), 1.60-1.48 ppm (m, 2H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 159.4, 156.2, 151.7, 138.1, 133.6, 131.0, 128.5, 128.0, 127.3, 127.1, 126.7, 126.6, 125.3, 124.8, 122.8, 122.6, 122.5, 122.4, 115.5, 113.7, 79.4, 55.2, 49.8, 25.7, 24.2 ppm. IR (KBr): ν = 2929 (m), 2889 (w), 2838 (w), 1605 (m), 1510 (s), 1379 (m), 1255 (m), 1174 (m), 1114 (m), 1020 (m), 836 (m), 816 (m), 759 (m), 726 (m) cm^{-1}. HRMS (MALDI) Calcd for $C_{34}H_{30}N_2O_2$: m/z (%): 497.2224 ($M\text{-}H^+$). Found: 497.2216 (100%).

2g: 2-cyclopropyl-2-phenyl-phenanthro (9,10)-[2*H*]-[1,4]-oxazine
M.p. 108.0-109.0 ºC. ^{1}H NMR (300 MHz, $CDCl_3$): δ 8.70-8.47 (m, 3H), 8.47-8.36 (m, 1H), 8.00 (s, 1H), 7.78 -7.45 (m, 6H), 7.38-7.19 (m, 3H), 1.66-1.54 (m, 1H), 0.94-0.60

ppm (m, 4H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 156.0, 141.0, 138.7, 131.1, 129.9, 128.4, 128.3, 127.5, 127.3, 126.8, 126.7, 125.8, 125.0, 122.9, 122.6, 122.6, 122.5, 76.6, 19.5, 1.4, 0.8 ppm. IR (KBr): 3074 (m), 3009(m), 1611 (w), 1578 (m), 1496 (m), 1447 (m), 1319 (m), 1242 (m), 1166 (m), 1111 (m), 1047 (m), 754 (m), 722 (m), 705(m) cm^{-1}. HRMS (MALDI) Calcd for $C_{25}H_{19}NO$: m/z (%): 350.1539 (MH^+). Found: 350.1540 (100%).

2h: 2-methyl-2-phenyl-phenanthro (9,10)-[2*H*]-[1,4]-oxazine
M.p. 119.0-120.0 ºC. ^{1}H NMR (300 MHz, $CDCl_3$): δ 8.70-8.54 (m, 3H), 8.54-8.45 (m, 1H), 7.94 (s, 1H), 7.74 -7.60 (m, 4H), 7.60-7.46 (m, 4H), 7.38-7.20 (m, 4H), 1.96 ppm (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 156.6, 142.0, 138.5, 131.0, 129.7, 128.6, 128.0, 127.4, 127.2, 126.7, 126.7, 125.1, 124.9, 124.9, 122.7, 122.6, 122.5, 122.5, 122.4, 75.5, 26.0 ppm. IR (KBr): ν = 3074 (w), 3025 (w), 3001 (w), 2940 (w), 1608 (w), 1580 (m), 1494 (m), 1443 (m), 1322 (m), 1167 (m), 1140 (m), 1015 (m), 764 (m), 726 (m), 708 (m) cm^{-1}. HRMS (MALDI) Calcd for $C_{23}H_{17}NO$: m/z (%): 324.1383 (MH^+). Found: 324.1384 (100%).

Reaction spectra:

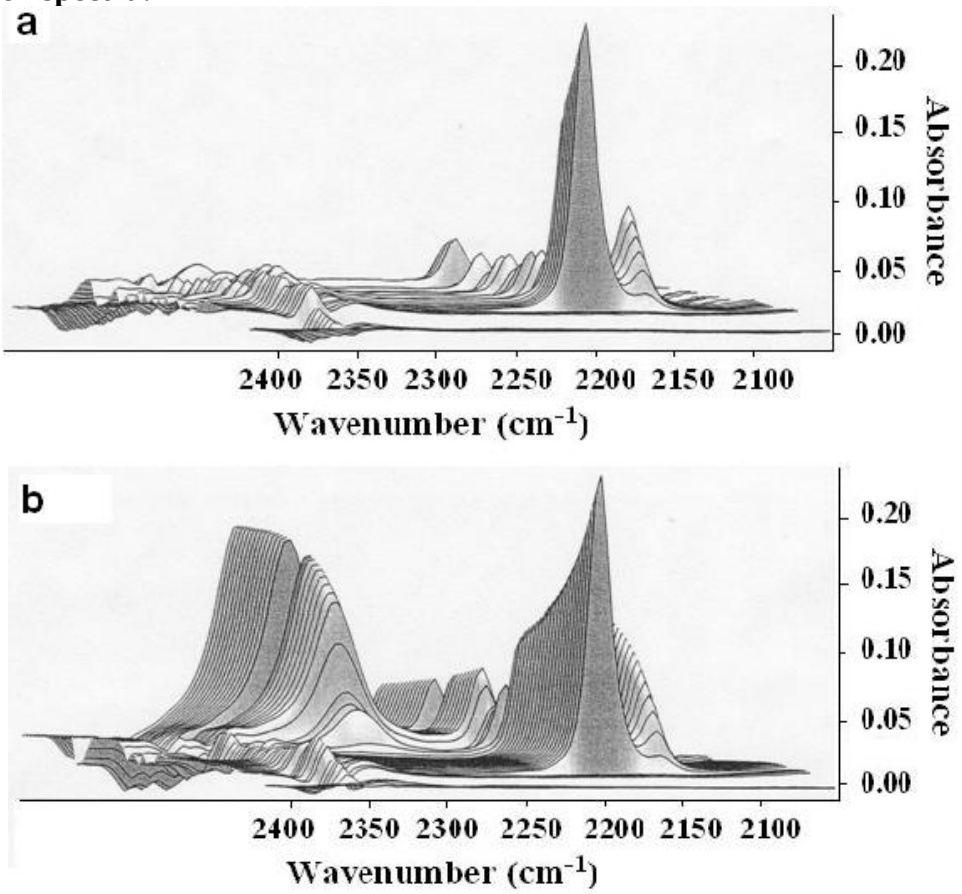


Figure 1. ReactIR spectra of one-pot reaction of 3,3-diphenyl acrylic acid (0.2 mmol scale), DPPA, Et_3N, Ph_3AsO, and 9,10-phenanthrenequinone in argon in degassed toluene (2 mL) from ambient temperature to 70 °C over 1 h (a), and the control

experiment without Ph_3AsO and 9,10-phenanthrenequinone (b). The spectra were taken in 1 min interval.

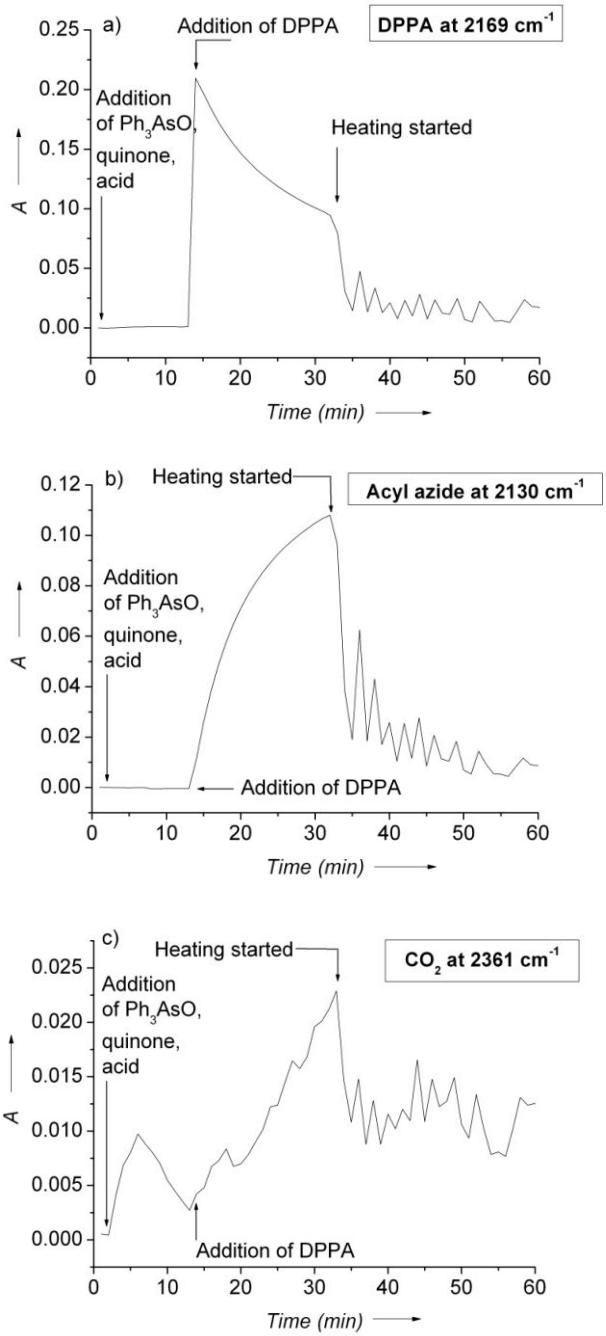


Figure 2. ReactIR profiles of Figure 1 (a). The ReactIR spectra of the one-pot formation of 2,2-diphenyl- phenanthro (9,10)-[2*H*]-[1,4]-oxazine compound (**1a**) in Figure 1 (a) and the profiles in Figure 2 (b) clearly indicated the fast formation of acyl azide at 2130 cm^{-1} and concomitantly consumption of DPPA at 2169 cm^{-1}. The isocynate intermediate observed in the control experiment Figure 1 (b) at 2264 cm^{-1} could not be seen in Figure 1 (a), which indicates such an intermediate to be captured immediately once it was formed. The profile 2 (c) indicated the generation of CO_2 at 2361 cm^{-1}.

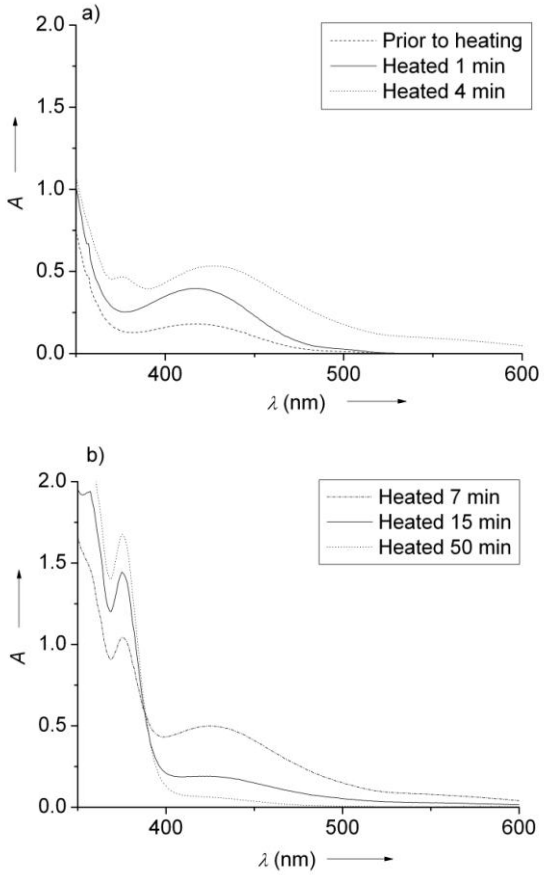
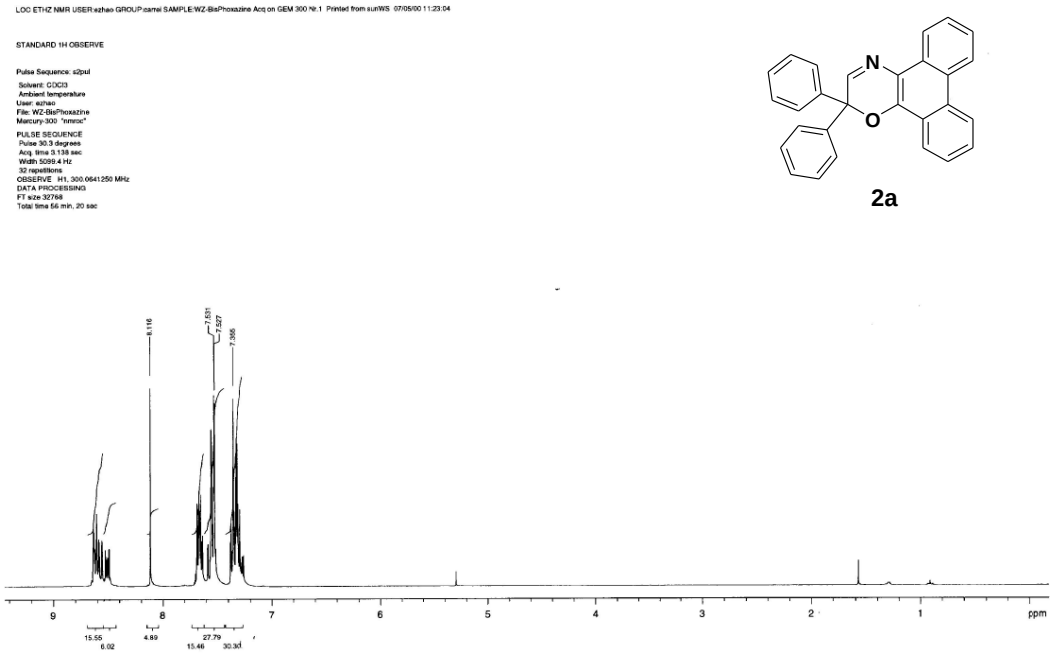
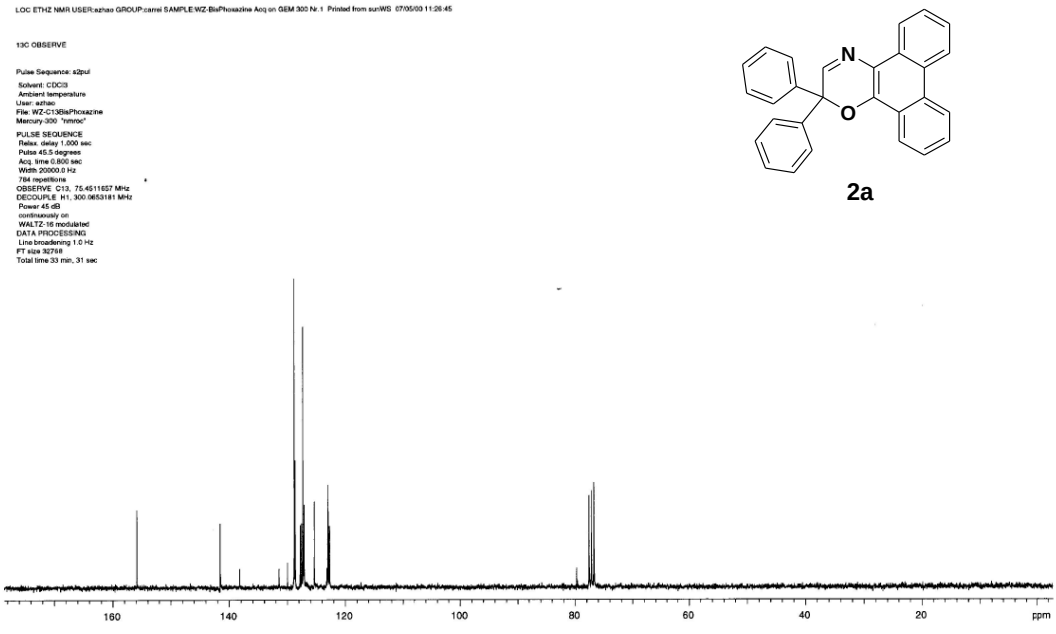


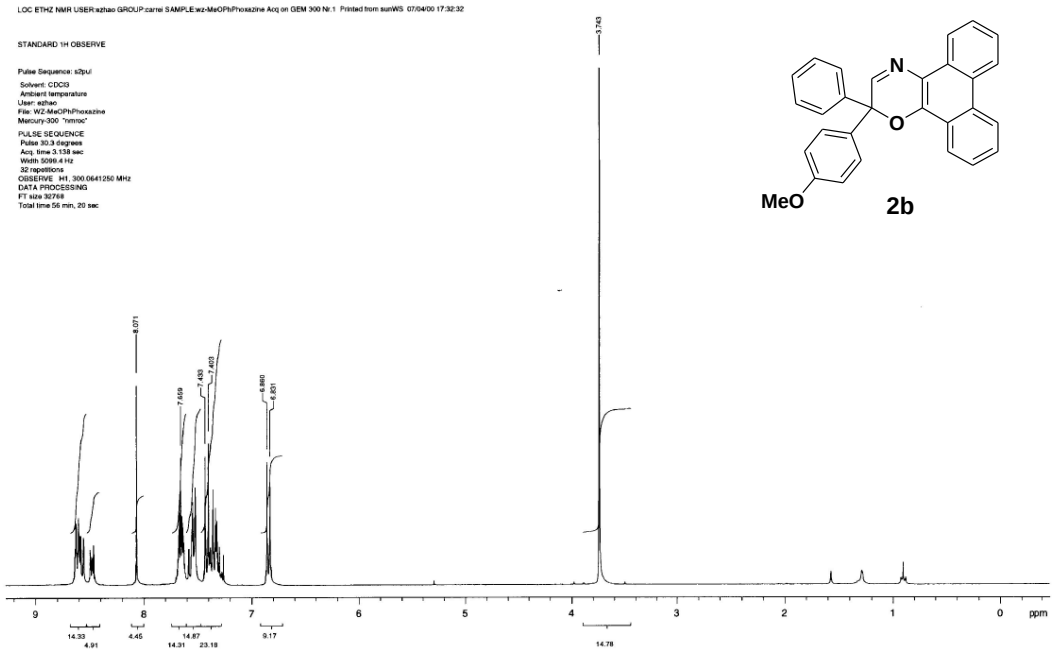
Figure 3. UV-Vis spectra of one-pot reaction of 3,3-diphenyl acrylic acid (0.2 mmol scale), DPPA, Et_3N, Ph_3AsO, and 9,10-phenanthrenequinone in toluene (2 mL) from ambient temperature to 70 °C over 1 h. Each spectrum was obtained by taken 40 μL of the reaction mixture and diluted in 2.5 mL toluene.

The formation of quinone-imine intermediate and final product could be easily captured by UV-Vis spectrometer (Figure 3). The starting 9,10-phenanthrenequinone absorbs at 415 nm. New colored species absorb at longer wavelength was generated upon heating, which was transformed to the oxazine (absorbs at much shorter wavelength) with extended time of heating indicated by the increased absorption at 360 nm.

Copies of NMR spectra:






LOC ETHZ NMR USER:ezhao GROUP:carreI SAMPLE:wz-MeOPhPhoxazine Acq on GEM 300 Nr.1 Printed from sunWS 07/04/00 17:32:32
STANDARD 1H OBSERVE
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Ambient temperature
User: ezhao
File: WZ-MeOPhPhoxazine
Mercury-300 "nmroc"
PULSE SEQUENCE
Pulse 30.3 degrees
Acq. time 3.138 sec
Width 5099.4 Hz
32 repetitions
OBSERVE H1, 300.0641250 MHz
DATA PROCESSING
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7.459
7.483
7.503
6.880
6.851
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14.31
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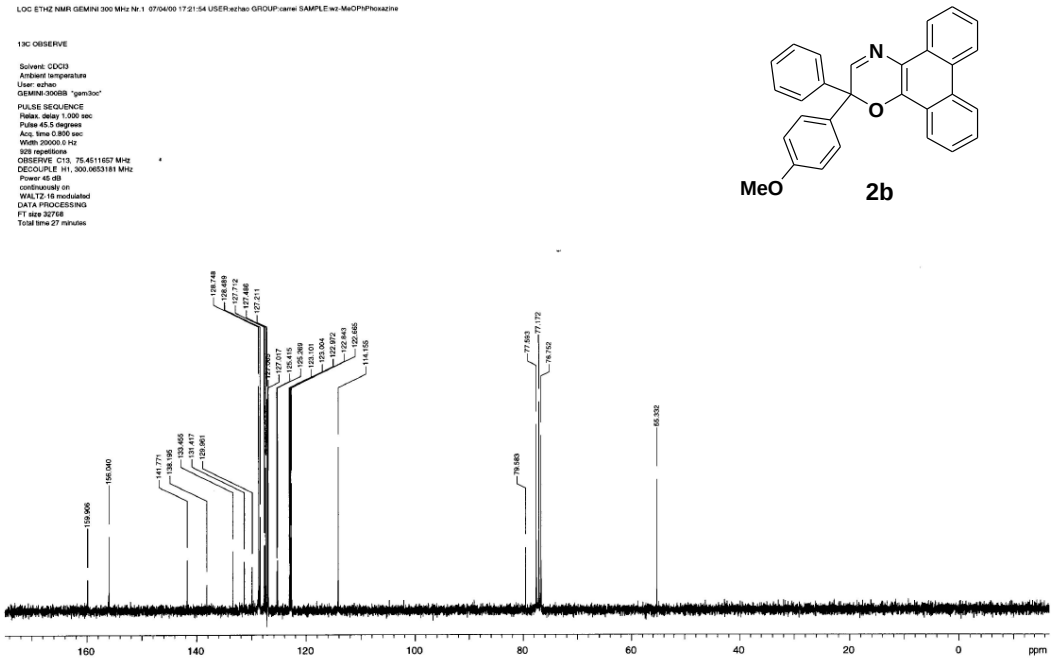
9.17
14.78
ppm
N
O
MeO
2b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13C OBSERVE
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Ambient temperature
User: ezhao
GEMINI-300BB "gem3oc"
PULSE SEQUENCE
Relax. delay 1.000 sec
Pulse 45.5 degrees
Acq. time 0.800 sec
Width 20000.0 Hz
928 repetitions
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DECOUPLE H1, 300.0653181 MHz
Power 45 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
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Total time 27 minutes
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133.455
131.417
130.801
126.748
126.489
127.712
127.486
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122.643
122.065
114.155
77.553
77.172
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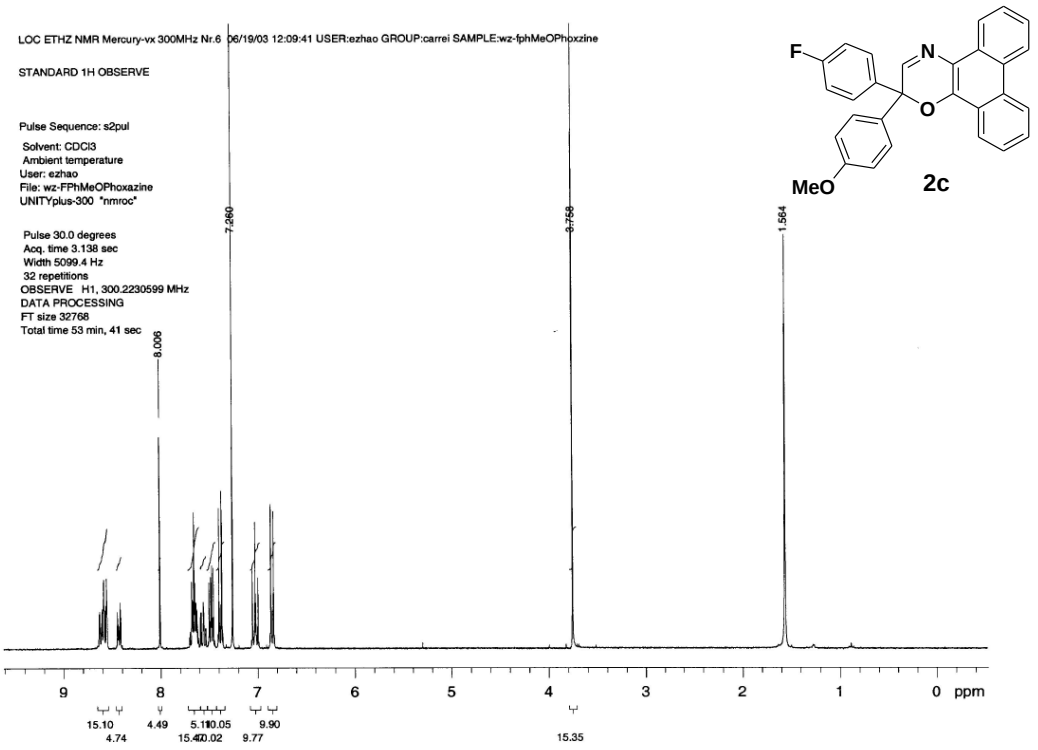
120
100
80
60
40
20
0
ppm
N
O
MeO
2b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Solvent: CDCl3
Ambient temperature
User: ezhao
File: wz-FPhMeOPhoxazine
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Pulse 30.0 degrees
Acq. time 3.138 sec
Width 5099.4 Hz
32 repetitions
OBSERVE H1, 300.2230599 MHz
DATA PROCESSING
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Total time 53 min, 41 sec
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7.260
3.758
1.564
9 8 7 6 5 4 3 2 1 0 ppm
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15.40
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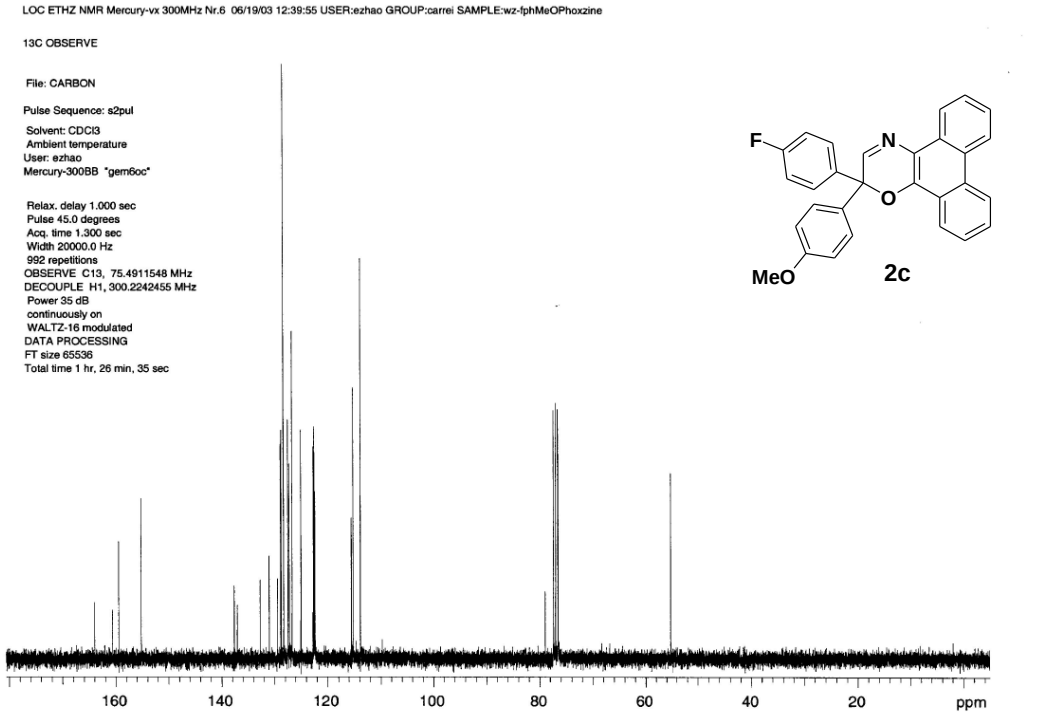
10.02
9.90
9.77
15.35
F
N
O
MeO
2c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13C OBSERVE
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Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: ezhao
Mercury-300BB *gem6oc*
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 20000.0 Hz
992 repetitions
OBSERVE C13, 75.4911548 MHz
DECOUPLE H1, 300.2242455 MHz
Power 35 dB
continuously on
WALTZ-16 modulated
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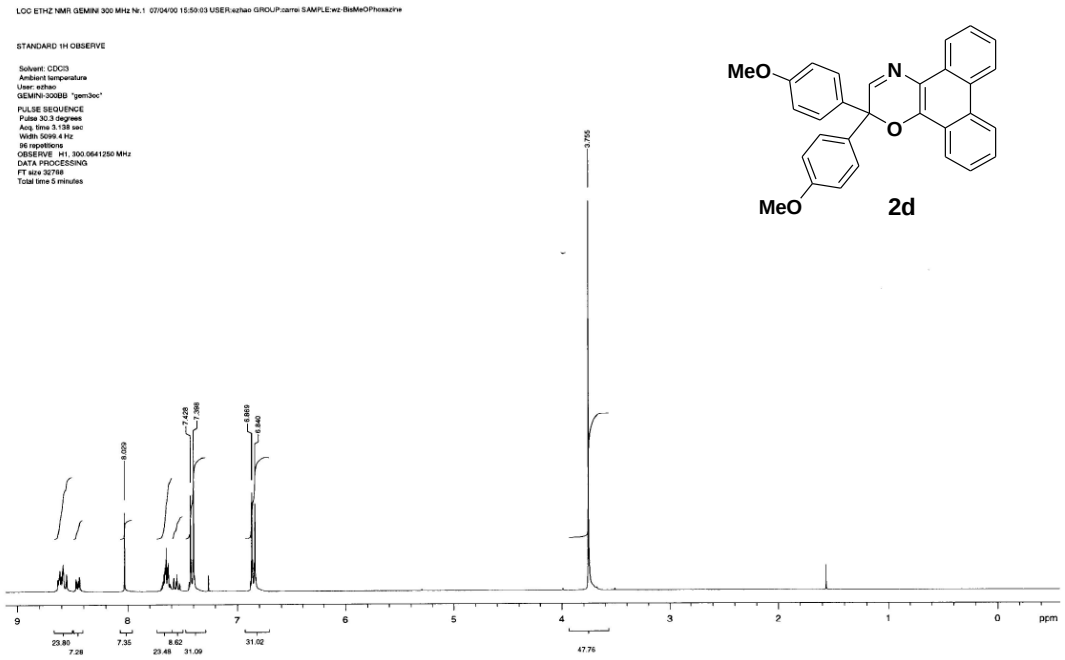
F
N
O
MeO
2c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STANDARD 1H OBSERVE
Solvent: CDCl3
Ambient temperature
User: ezhao
GEMINI-300BB "gem3oc"
PULSE SEQUENCE
Pulse 30.3 degrees
Acq. time 3.138 sec
Width 5099.4 Hz
96 repetitions
OBSERVE H1, 300.0641250 MHz
DATA PROCESSING
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Total time 5 minutes
MeO
N
O
MeO
2d
9.059
7.428
7.399
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3.755
ppm
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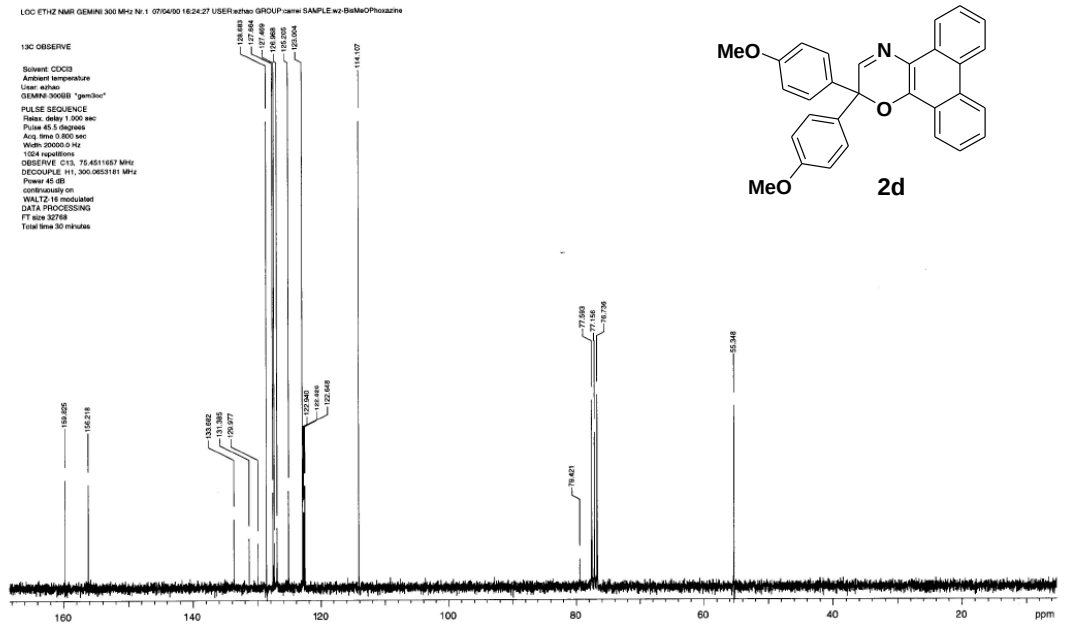
7.28
7.35
8.62
23.48
31.09
31.02
47.76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13C OBSERVE
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Ambient temperature
User: ezhao
GEMINI-300BB "gem3oc"
PULSE SEQUENCE
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Pulse 45.5 degrees
Acq. time 0.800 sec
Width 20000.0 Hz
1024 repetitions
OBSERVE C13, 75.4511657 MHz
DECOUPLE H1, 300.0653181 MHz
Power 45 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
FT size 32768
Total time 30 minutes
MeO
N
O
MeO
2d
159.825
156.218
133.662
131.365
129.977
128.689
127.664
127.468
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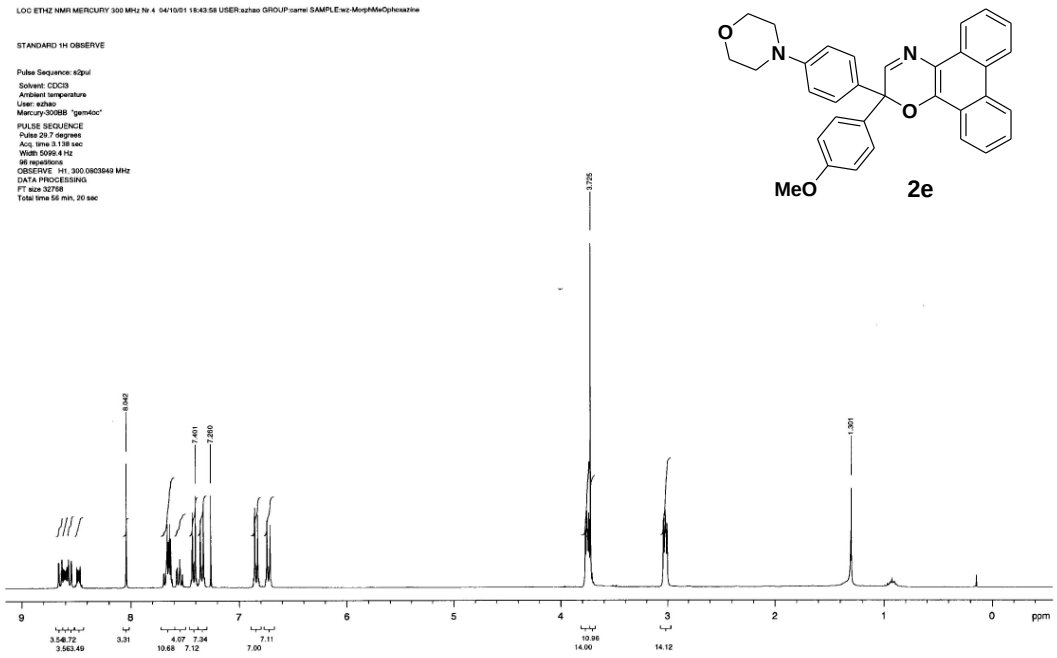
77.156
76.726
55.348
ppm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Solvent: CDCl3
Ambient temperature
User: azhao
Mercury-300BB "gem4oc"
PULSE SEQUENCE
Pulse 29.7 degrees
Acq. time 3.138 sec
Width 5099.4 Hz
96 repetitions
OBSERVE H1, 300.0803949 MHz
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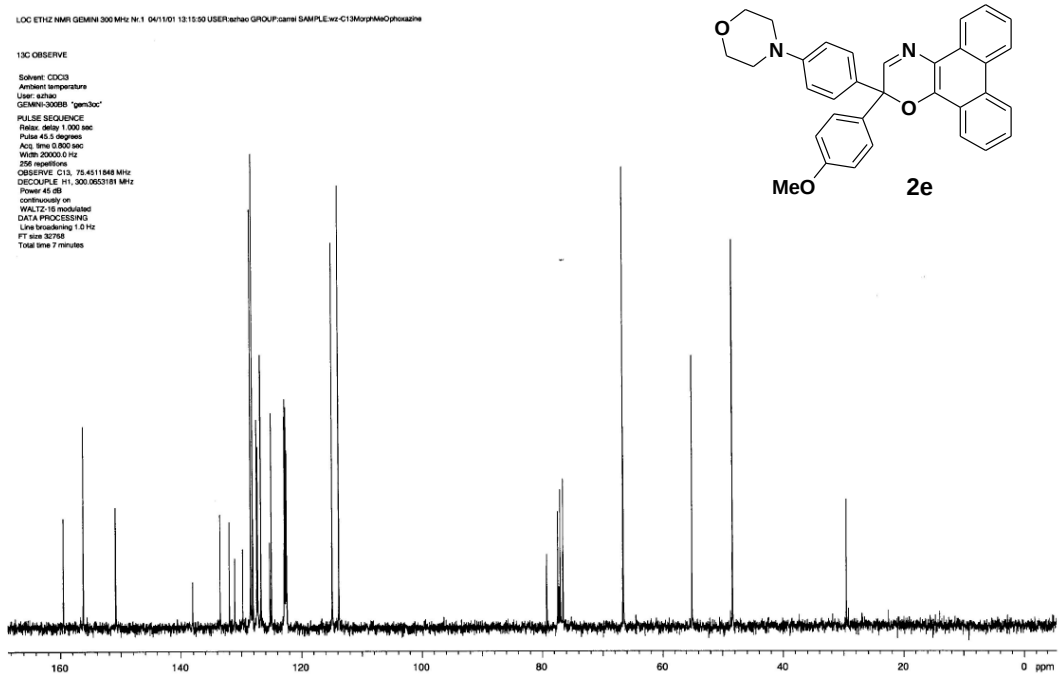
1.361
ppm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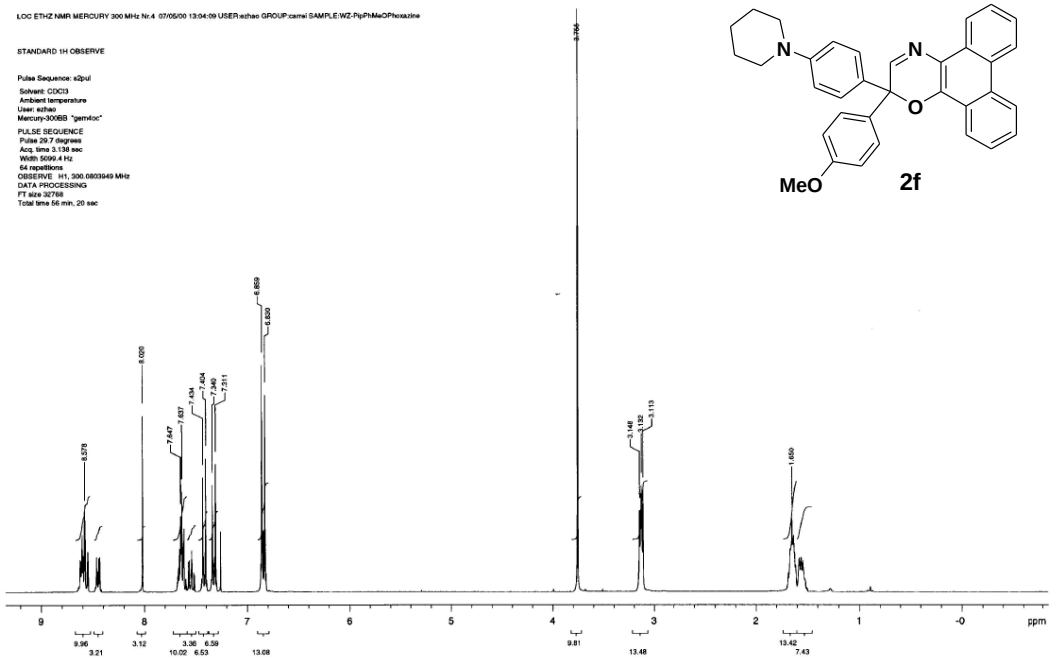
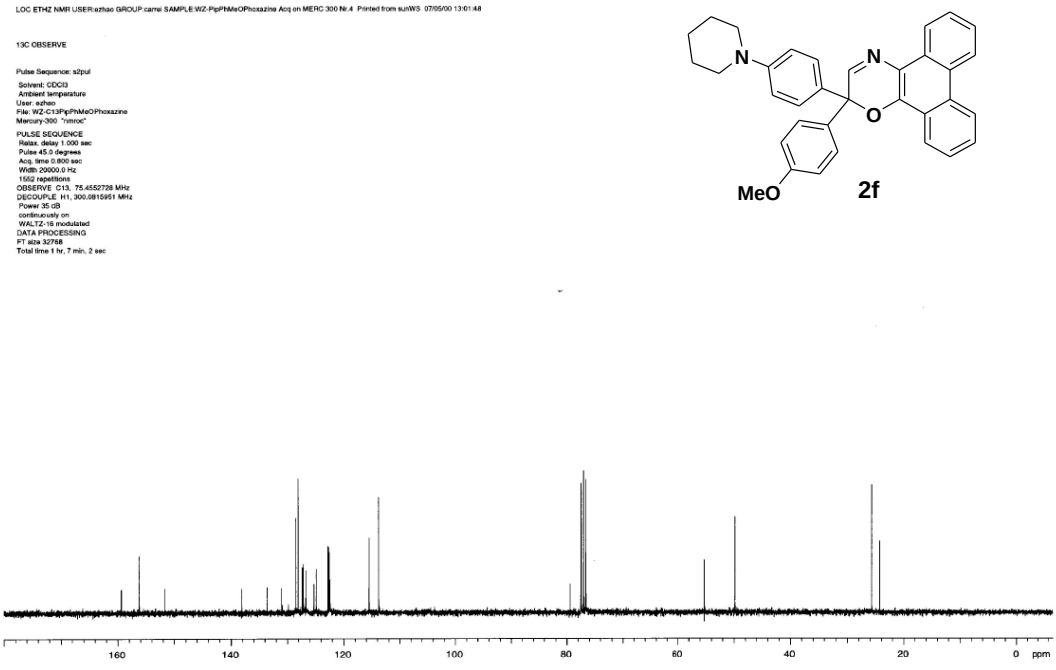
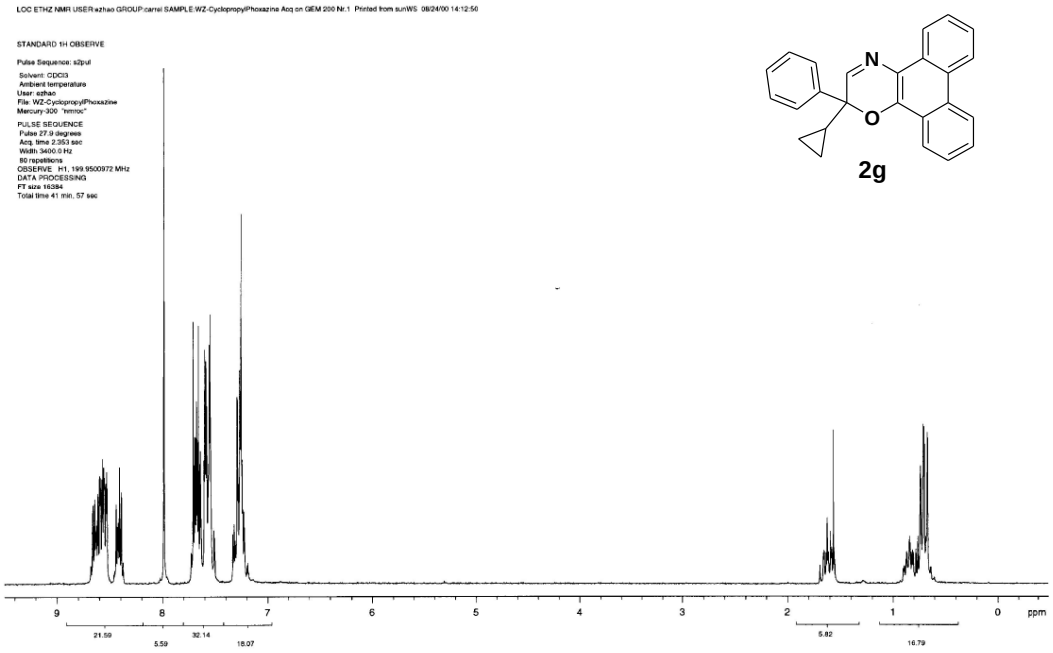
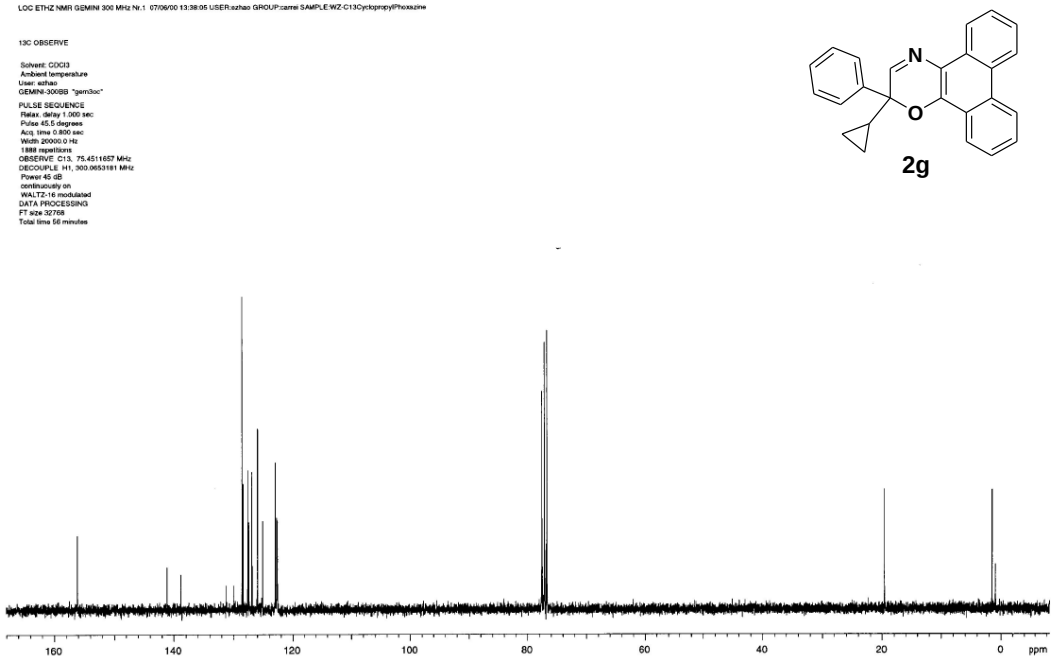
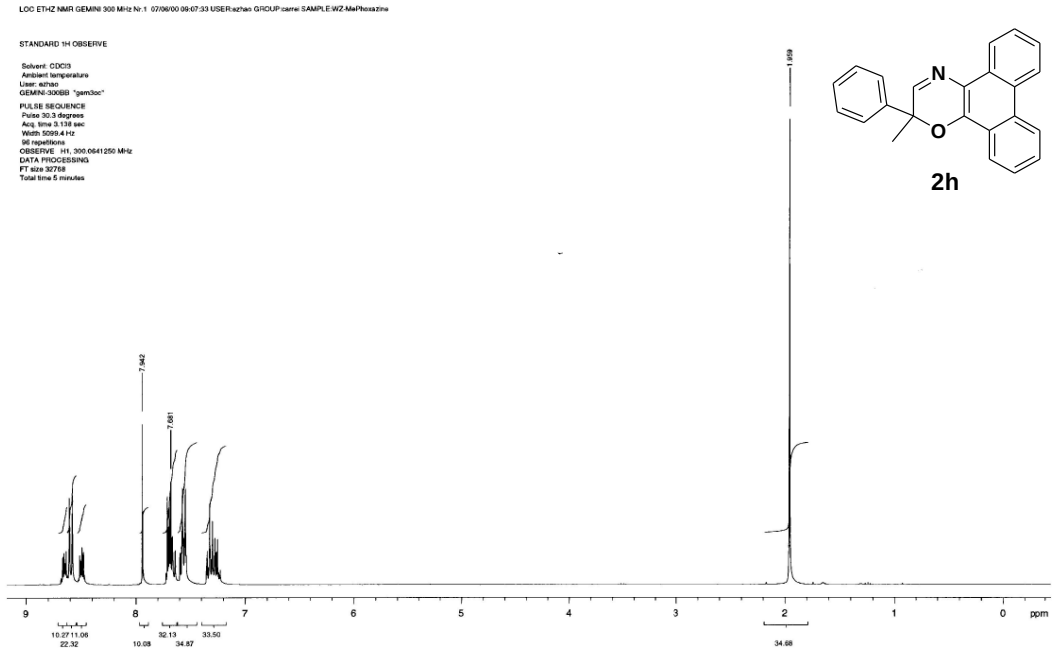
Ambient temperature
User: azhao
GEMINI-300BB "gem3oc"
PULSE SEQUENCE
Relax. delay 1.000 sec
Pulse 45.5 degrees
Acq. time 0.800 sec
Width 20000.0 Hz
256 repetitions
OBSERVE C13, 75.4511848 MHz
DECOUPLE H1, 300.0653181 MHz
Power 45 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 32768
Total time 7 minutes
MeO
2e
ppm

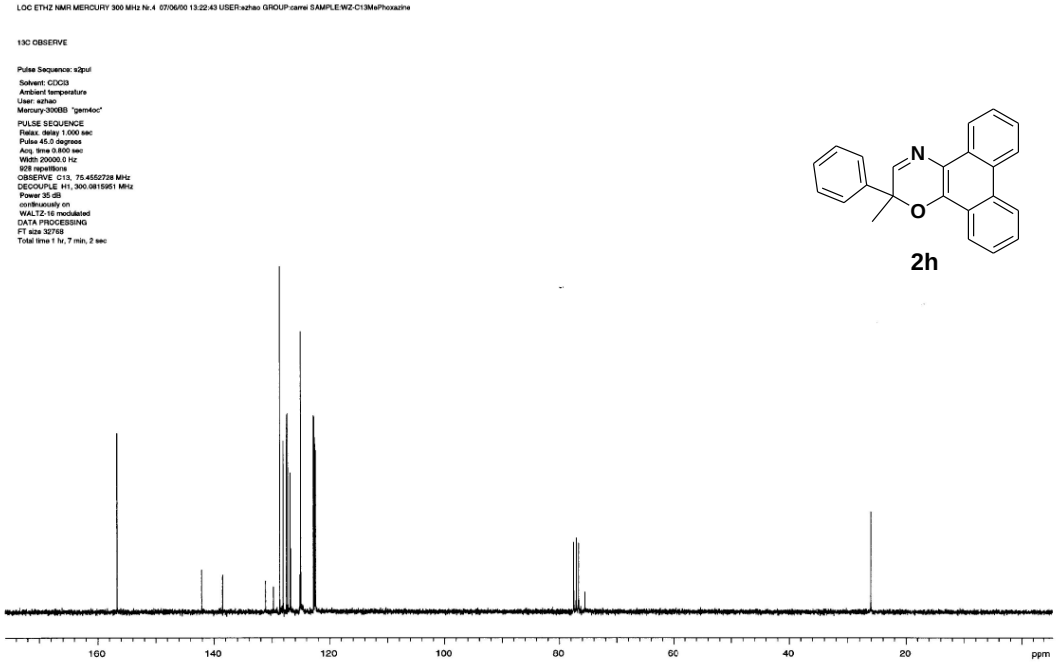

LOC ETHZ NMR MERCURY 300 MHz Nr.4 07/05/00 13:04:09 USER:ezhao GROUP:camsl SAMPLE:WZ-PipPhMeOPhoxazine
STANDARD 1H OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: ezhao
Mercury-300BB "gemtoc"
PULSE SEQUENCE
Pulse 29.7 degrees
Acq. time 3.138 sec
Width 5099.4 Hz
64 repetitions
OBSERVE H1, 300.0803949 MHz
DATA PROCESSING
FT size 32768
Total time 56 min, 20 sec
MeO
2f
ppm


LOC ETHZ NMR USER:ezhao GROUP:camsl SAMPLE:WZ-PipPhMeOPhoxazine Acq on MERC 300 Nr.4 Printed from sunWS 07/05/00 13:01:48
13C OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: ezhao
File: WZ-C13PipPhMeOPhoxazine
Mercury-300 "nmroc"
PULSE SEQUENCE
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 0.800 sec
Width 20000.0 Hz
1552 repetitions
OBSERVE C13, 75.4552728 MHz
DECOUPLE H1, 300.0815951 MHz
Power 35 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
FT size 32768
Total time 1 hr, 7 min, 2 sec
MeO
2f
ppm


LOC ETHZ NMR USER:azhao GROUP:carrel SAMPLE:WZ-Cyclopropylphoxazine Acq on GEM 200 Nr.1 Printed from sunWS 08/24/00 14:12:50
STANDARD 1H OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: azhao
File: WZ-CyclopropylPhoxazine
Mercury-300 "nmroc"
PULSE SEQUENCE
Pulse 27.9 degrees
Acq. time 2.353 sec
Width 3400.0 Hz
80 repetitions
OBSERVE H1, 199.9500972 MHz
DATA PROCESSING
FT size 16384
Total time 41 min, 57 sec
N
O
2g
9
8
7
6
5
4
3
2
1
0
ppm
21.59
5.59
32.14
18.07
5.82
16.79


LOC ETHZ NMR GEMINI 300 MHz Nr.1 07/06/00 13:38:05 USER:azhao GROUP:carrel SAMPLE:WZ-C13CyclopropylPhoxazine
13C OBSERVE
Solvent: CDCl3
Ambient temperature
User: azhao
GEMINI-300BB "gem3oc"
PULSE SEQUENCE
Relax. delay 1.000 sec
Pulse 45.5 degrees
Acq. time 0.800 sec
Width 20000.0 Hz
1888 repetitions
OBSERVE C13, 75.4511657 MHz
DECOUPLE H1, 300.0653181 MHz
Power 45 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
FT size 32768
Total time 56 minutes
N
O
2g
160
140
120
100
80
60
40
20
0
ppm


LOC ETHZ NMR GEMINI 300 MHz Nr.1 07/06/00 09:07:33 USER:azhao GROUP:carrei SAMPLE:WZ-MePhoxazine
STANDARD 1H OBSERVE
Solvent: CDCl3
Ambient temperature
User: azhao
GEMINI-300BB "gem3oc"
PULSE SEQUENCE
Pulse 30.3 degrees
Acq. time 3.138 sec
Width 5099.4 Hz
96 repetitions
OBSERVE H1, 300.0641250 MHz
DATA PROCESSING
FT size 32768
Total time 5 minutes
7.942
7.681
1.659
10.27 11.06
22.32
10.08
32.13 33.50
34.87
34.68
9
8
7
6
5
4
3
2
1
0
ppm
N
O
2h


LOC ETHZ NMR MERCURY 300 MHz Nr.4 07/06/00 13:22:43 USER:azhao GROUP:carrei SAMPLE:WZ-C13MePhoxazine
13C OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: azhao
Mercury-300BB "gem4oc"
PULSE SEQUENCE
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 0.800 sec
Width 20000.0 Hz
928 repetitions
OBSERVE C13, 75.4552728 MHz
DECOUPLE H1, 300.0815951 MHz
Power 35 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
FT size 32768
Total time 1 hr, 7 min, 2 sec
160
140
120
100
80
60
40
20
ppm
N
O
2h