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Electronic Supplementary Information

A Room-Temperature Synthesis of 2-2'-Bisoxazoles through Palladium-Catalyzed Oxidative Coupling of α-Isocyanoacetamides

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I. General Information

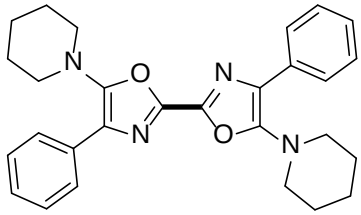
^{1}H NMR (400 MHz) and ^{13}C NMR (125 MHz) were registered on Bruker 400 M and 500 M spectrometers. Chemical shifts were reported in units (ppm) by assigning TMS resonance in the ^{1}H spectrum as 0.00 ppm, $CDCl_3$ resonance in the ^{13}C spectrumas 77.0 ppm. All coupling constants (*J* values) were reported in Hertz (Hz) unit. IR, GC, MS, and melting points were performed by the Analytical Center in GIBH. HRMS was performed by the Analytical Center in Jinan University.

II. Synthetic Procedures

General Procedure A*:* To an open tube was added $Pd(OAc)_2$ (0.02 mmol, 4.48 mg), PPh_3 (0.04 mmol, 10.5 mg), Cs_2CO_3 (0.22 mmol, 71.7 mg) and 1.0 mL of MeCN. After the mixture was stirred at room temperature for 0.5 h, 2-isocyano-2-phenyl-1-(piperidin-1-yl)ethanone (**1a**, 0.2 mmol) was dissolved in 1.0 mL of MeCN and the solution was added dropwise with a syringe pump within 0.5 h. The crude reaction mixture was extracted with DCM (20 mL × 3) and washed with brine (20 mL). The organic phase was concentrated in vacuo and the purified product **2a** was isolated as a white solid by flash chromatography using DCM and MeOH (100:1 to 50:1) as the eluent.

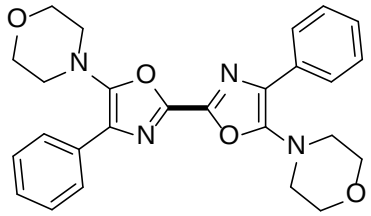
General Procedure B*:* To an open tube was added $Pd(OAc)_2$ (0.02 mmol, 4.48 mg), PPh_3 (0.04 mmol, 10.5 mg), Cs_2CO_3 (0.22 mmol, 71.7 mg) and 1.0 mL of MeCN. After the mixture was stirred at room temperature for 0.5 h, a solution of 2-isocyano-2-phenyl-1-(piperidin-1-yl)ethanone (**1a**, 0.2 mmol) and 2-isocyano-2-phenyl-1-(piperidin-1-yl)ethanone (**1b**, 0.6 mmol) in 4.0 mL of MeCN was added dropwise with a syringe pump over 1.0 h. The crude reaction mixture was extracted with DCM (20 mL × 3) and washed with brine (20 mL). The organic phase was concentrated in vacuo and the purified product **3a** was isolated as a white solid by flash chromatography using DCM and MeOH (100:1 to 50:1) as the eluent.

III. Product Characterization



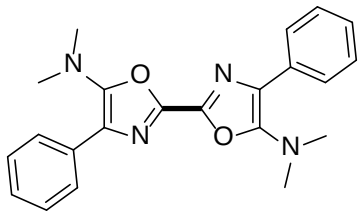
4,4'-diphenyl-5,5'-di(piperidin-1-yl)-2,2'-bioxazole (2a)

Prepared from 2-isocyano-2-phenyl-1-(piperidin-1-yl)ethanone (**1a**, 0.2 mmol) according to the general procedure A. Isolated as a white solid. Yield: 74%. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.90 (d, *J* = 7.6 Hz, 4H), 7.41 (t, *J* = 7.2 Hz, 4H), 7.25-7.29 (m, 2H), 3.19 (t, *J* = 4.8 Hz, 8H), 1.73-1.74 (m, 8H), 1.63-1.65 (m, 4H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 153.1, 145.0, 131.8, 128.3, 126.9, 126.2, 123.6, 50.9, 25.8, 23.8; FTIR: ν_{max} /cm^{-1} 2927, 2847, 1626, 1500, 1384, 1224, 1106, 981, 770, 705; HRMS: calcd for $C_{28}H_{31}N_4O_2$ (M^++H) 455.2447; found: 455.2442; mp: 158-160 ^{o}C.



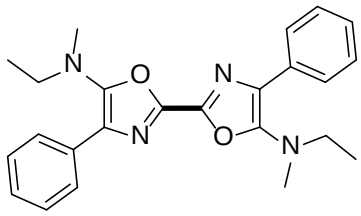
5,5'-dimorpholino-4,4'-diphenyl-2,2'-bioxazole (2b)

Prepared from 2-isocyano-1-morpholino-2-phenylethanone (**1b**, 0.2 mmol) according to the general procedure A. Isolated as a white solid. Yield: 70%. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.98 (d, *J* = 8.0 Hz, 4H), 7.43 (t, *J* = 7.6 Hz, 4H), 7.26-7.30 (m, 2H), 3.88 (t, *J* = 4.0 Hz, 8H), 3.26 (t, J = 3.6 Hz, 8H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 151.7, 145.4, 131.2, 128.4, 127.4, 126.4, 124.7, 66.7, 49.9; FTIR: ν_{max} /cm^{-1} 2891, 2845, 1625, 1601, 1486, 1447, 1385, 1115, 986, 707; HRMS: calcd for $C_{26}H_{27}N_4O_4$ (M^++H) 459.2032; found: 459.2024; mp: 277-279 ^{o}C.



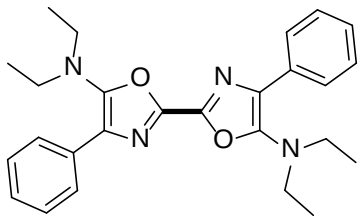
***N*5,*N*5,*N*5',*N*5'-tetramethyl-4,4'-diphenyl-[2,2'-bioxazole]-5,5'-diamine (2c)**

Prepared from 2-isocyano-*N*,*N*-dimethyl-2-phenylacetamide (**1c**, 0.2 mmol) according to the general procedure A. Isolated as a white solid. Yield: 78%. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.90 (d, *J* = 7.6 Hz, 4H), 7.41 (t, *J* = 7.6 Hz, 4H), 7.26-7.29 (m, 2H), 2.92 (s, 12H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 153.3, 144.5, 131.7, 128.3, 126.9, 126.6, 122.6, 41.8; FTIR: ν_{max} /cm^{-1} 2957, 2885, 1628, 1504, 1447, 1406, 1375, 1090, 974, 716; HRMS: calcd for $C_{22}H_{23}N_4O_2$ (M^++H) 375.1821; found: 375.1816; mp: 197-199 ^{o}C.



***N*5,*N*5'-diethyl-*N*5,*N*5'-dimethyl-4,4'-diphenyl-[2,2'-bioxazole]-5,5'-diamine (2d)**

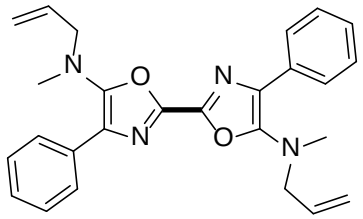
Prepared from *N*-ethyl-2-isocyano-*N*-methyl-2-phenylacetamide (**1d**, 0.2 mmol) according to the general procedure A. Isolated as a white solid. Yield: 81%.^{1}H NMR (400 MHz, $CDCl_3$): δ 7.96 (d, *J* = 7.6 Hz, 4H), 7.41 (t, *J* = 7.6 Hz, 4H), 7.25-7.27 (m, 2H), 3.21 (q, *J* = 7.2 Hz, 4H), 2.90 (s, 6H), 1.15 (t, J = 7.2 Hz, 6H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 152.5, 145.2, 131.7, 128.2, 127.0, 126.5, 124.6, 48.9, 39.2, 12.8; FTIR: ν_{max} /cm^{-1} 2968, 1614,1601, 1486, 1228, 1095, 989, 963, 789, 712; HRMS: calcd for $C_{24}H_{27}N_4O_2$ (M^++H) 403.2134; found: 403.2126; mp: 129-131 ^{o}C.



***N*5,*N*5,*N*5',*N*5'-tetraethyl-4,4'-diphenyl-[2,2'-bioxazole]-5,5'-diamine (2e)**

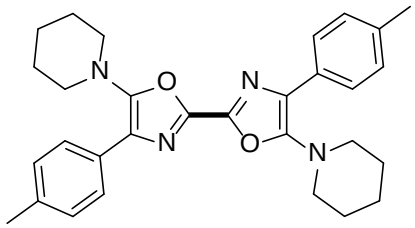
Prepared from *N*,*N*-diethyl-2-isocyano-2-phenylacetamide (**1e**, 0.2 mmol) according to the general procedure A. Isolated as a white solid. Yield: 77%. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.07 (d, *J* = 7.6 Hz, 4H), 7.40 (t, *J* = 7.6 Hz, 4H), 7.26-7.30 (m, 2H), 3.22 (q, *J* = 7.2 Hz, 4H),1.12(t, J = 7.2 Hz, 6H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 151.2, 146.2, 131.7, 128.2, 127.7, 127.2, 126.4, 47.0, 13.3; FTIR: ν_{max} /cm^{-1} 2975, 2844, 1626, 1560, 1502, 1484, 1340, 1196, 1068, 772; HRMS: calcd for $C_{26}H_{31}N_4O_2$

(M^++H) 431.2447; found: 431.2443; mp: 163-165 ^oC.



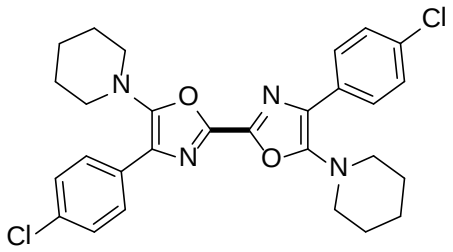
_N_5,_N_5'-diallyl-_N_5,_N_5'-dimethyl-4,4'-diphenyl-[2,2'-bioxazole]-5,5'-diamine (2f)

Prepared from *N*-allyl-2-isocyano-N-methyl-2-phenylacetamide (**1f**, 0.2 mmol) according to general procedure A. Isolated as a white solid. Yield: 82%. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.94 (d, *J* = 7.6 Hz, 4H), 7.41 (t, *J* = 7.6 Hz, 4H), 7.26-7.30 (m, 2H), 5.84-5.94 (m, 2H), 5.18-5.27 (m, 4H), 3.75 (d, *J* = 6.4 Hz, 4H), 2.89 (s, 6H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 152.6, 145.0, 133.7, 131.7, 128.3, 127.1, 126.6, 124.1, 118.6, 57.3, 38.9; FTIR: ν_{max} /cm^{-1} 2916, 2842, 1629, 1601, 1481, 1378, 1220, 1064, 995, 728; HRMS: calcd for $C_{26}H_{27}N_4O_2$ (M^++H) 427.2134; found: 427.2128; mp: 98-100 ^oC.



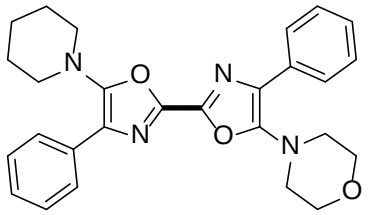
5,5'-di(piperidin-1-yl)-4,4'-di-*p*-tolyl-2,2'-bioxazole (2g)

Prepared from 2-isocyano-1-(piperidin-1-yl)-2-(*p*-tolyl)ethanone (**1g**, 0.2 mmol) according to the general procedure A. Isolated as a white solid. Yield: 56%. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.88 (d, *J* = 8.0 Hz, 4H), 7.23 (d, *J* = 8.0 Hz, 4H), 3.18 (t, *J* = 4.8 Hz, 8H), 2.38 (s, 6H), 1.71-1.76 (m, 8H), 1.61-1.65 (m, 4H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 152.7, 145.1, 136.6, 128.9, 129.0, 126.2, 123.9, 50.9, 29.7, 25.8, 23.8, 21.2; FTIR: ν_{max} /cm^{-1} 2936, 2849, 1624, 1514, 1385, 1180, 1112, 987, 822, 611; HRMS: calcd for $C_{30}H_{35}N_4O_2$ (M^++H) 483.2760; found: 483.2757; mp: 228-230 ^oC.



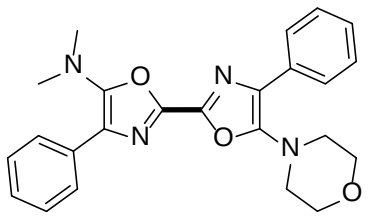
4,4'-bis(4-chlorophenyl)-5,5'-di(piperidin-1-yl)-2,2'-bioxazole (2h)

Prepared from 2-(4-chlorophenyl)-2-isocyano-1-(piperidin-1-yl)ethanone (**1h**, 0.2 mmol) according to the general procedure A. Isolated as a white solid. Yield: 72%. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.92 (d, *J* = 8.0 Hz, 4H), 7.38 (t, *J* = 8.0 Hz, 4H), 3.17 (t, *J* = 4.8Hz, 8H), 1.73-1.74 (m, 8H), 1.64-1.65 (m, 4H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 153.3, 145.0, 132.6, 130.3, 128.5, 127.4, 122.7, 50.9, 25.7, 23.7; FTIR: ν_{max} /cm^{-1} 2937, 2822, 1620, 1593, 1470, 1382, 1109, 985, 883, 730; HRMS: calcd for $C_{28}H_{29}Cl_2N_4O_2$ (M^++H) 523.1668; found: 523.1663; mp: 233-235 ^{o}C.



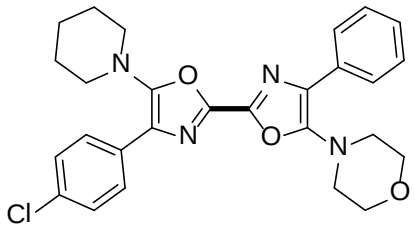
5-morpholino-4,4'-diphenyl-5'-(piperidin-1-yl)-2,2'-bioxazole (3a)

Prepared from 2-isocyano-2-phenyl-1-(piperidin-1-yl)ethanone (**1a**, 0.2 mmol) and 2-isocyano-1-morpholino-2-phenylethanone (**1b**, 0.6 mmol) according to the general procedure B. Isolated as a white solid. Yield: 51%.^{1}H NMR (400 MHz, $CDCl_3$): δ 7.98 (t, *J* = 7.2 Hz, 4H), 7.42 (t, *J* = 7.2 Hz, 4H), 7.26-7.31 (m, 2H), 3.88-3.89 (m, 4H), 3.25-3.26 (m, 4H), 3.20 (t, *J* = 4.8Hz, 4H), 1.74-1.75 (m, 4H), 1.64-1.65 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 153.3, 151.6, 145.6, 144.7, 131.7, 131.3, 128.4, 128.3, 127.3, 127.0, 126.4, 126.2, 124.7, 123.5, 66.8, 50.9, 50.0, 25.7, 23.8; FTIR: ν_{max} /cm^{-1} 2955, 2846, 1625, 1600, 1500, 1448, 1384, 1115, 984, 706; HRMS: calcd for $C_{27}H_{29}N_4O_3$ (M^++H) 457.2240; found: 457.2233; mp: 199-201 ^{o}C.



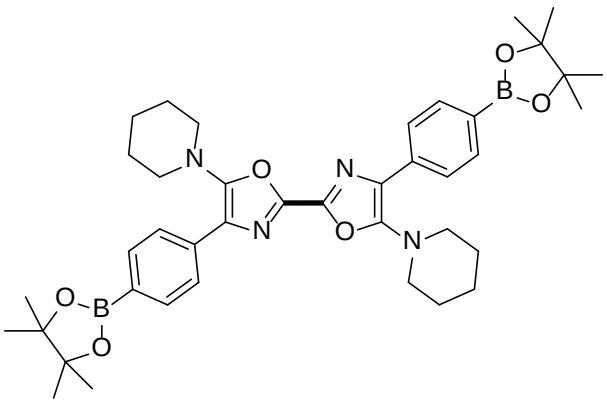
***N*,*N*-dimethyl-5'-morpholino-4,4'-diphenyl-[2,2'-bioxazol]-5-amine (3b)**

Prepared from 2-isocyano-*N*,*N*-dimethyl-2-phenylacetamide (**1c**, 0.2 mmol) and 2-isocyano-1-morpholino-2-phenylethanone (**1b**, 0.6 mmol) according to the general procedure B. Isolated as a white solid. Yield: 63%. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.99 (d, *J* = 7.6 Hz, 2H), 7.88 (d, *J* = 7.6 Hz, 2H), 7.40-7.43 (m, 4H), 7.26-7.30 (m, 2H), 3.88 (br s, 4H), 3.25 (br s, 4H), 2.93 (s, 6H); ^{13}C NMR (125 MHz, $CDCl_3$): δ153.4, 151.6, 145.6, 144.2, 131.7, 131.3, 128.4, 128.3, 127.4, 127.0, 126.7, 126.4, 124.7, 122.5, 66.8, 50.0, 41.7; FTIR: ν_{max} /cm^{-1} 2990, 2846, 1626, 1600, 1502, 1380, 1262, 1115, 1096, 710; HRMS: calcd for $C_{24}H_{25}N_4O_3$ (M^++H) 417.1927; found: 417.1922; mp: 207-208 ^{o}C.



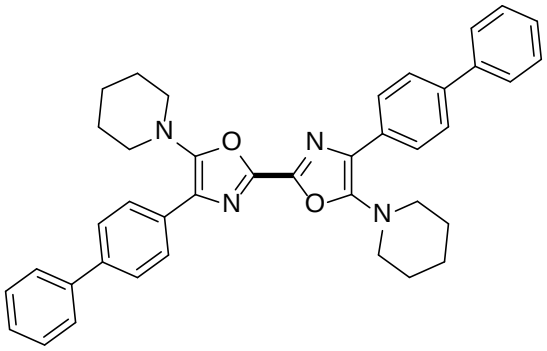
4-(4-chlorophenyl)-5'-morpholino-4'-phenyl-5-(piperidin-1-yl)-2,2'-bioxazole (3c)

Prepared from 2-(4-chlorophenyl)-2-isocyano-1-(piperidin-1-yl)ethanone (**1g**, 0.2 mmol) and 2-isocyano-1-morpholino-2-phenylethanone (**1b**, 0.6 mmol) according to the general procedure B. Isolated as a white solid. Yield: 57%. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.98 (d, *J* = 7.6 Hz, 2H), 7.92 (d, *J* = 7.2 Hz, 2H), 7.38-7.43 (m, 4H), 7.26-7.30 (m, 1H), 3.88-3.89 (m, 4H), 3.25-3.26 (m, 4H), 3.18 (t, *J* = 4.8Hz, 4H), 1.74 (m, 4H), 1.64-1.65 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 153.4, 151.6, 145.4, 144.8, 132.6, 131.2, 130.2, 128.5, 128.4, 127.4, 127.4, 126.4, 124.7, 122.7, 66.8, 50.9, 49.9, 25.7, 23.7; FTIR: ν_{max} /cm^{-1} 2922, 2843, 1622, 1599, 1498, 1384, 1262, 1157, 987, 707; HRMS: calcd for $C_{27}H_{28}ClN_4O_3$ (M^++H) 491.1850; found: 491.1842; mp: 177-179 ^{o}C.



5,5'-di(piperidin-1-yl)-4,4'-bis(4-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)phenyl)-2,2'-bioxazole (4)

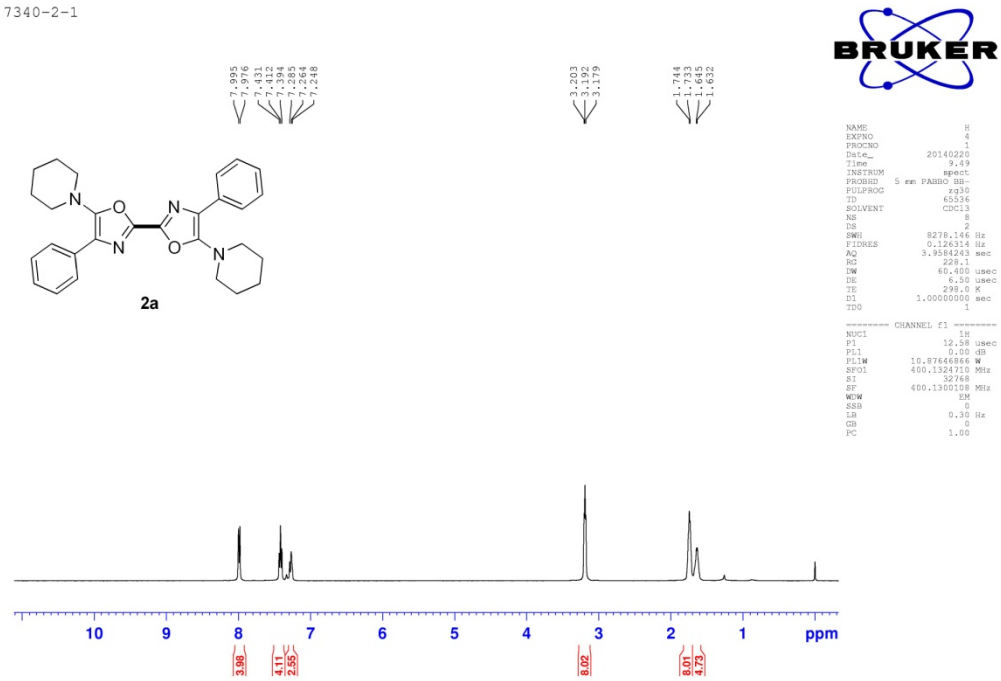
Prepared from 4,4'-bis(4-chlorophenyl)-5,5'-di(piperidin-1-yl)-2,2'-bioxazole (**2h**, 0.1 mmol). ***Procedure:*** An oven-dried Schlenk tube was charged with 4,4'-bis(4-chlorophenyl)-5,5'-di(piperidin-1-yl)-2,2'-bioxazole (**2h**, 52.2 mg, 0.1 mmol), $Pd_2(dba)_3$ (9.2 mg, 0.01 mmol), XPhos (19.1 mg, 0.04 mmol), bis(pinacolato)diboron (203 mg, 0.8 mmol) and KOAc (58.8 mg, 0.6 mmol). The Schlenk tube was capped with a rubber septum and then evacuated and backfilled with argon (this sequence was carried out twice). 1,4-Dioxane (1.0 mL) was added via syringe. The septum was then replaced with a Teflon screwcap and the Schlenk tube was sealed. The reaction mixture was heated to 110 °C until **2h** had been completely consumed. The crude reaction mixture was extracted with DCM (20 mL × 3) and washed with brine (20 mL). The organic phase was concentrated in vacuo and the purified product **4** was isolated as a white solid by flash chromatography. Yield: 89%. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.97 (d, *J* = 8.0 Hz, 4H), 7.86 (d, *J* = 8.0 Hz, 4H), 3.19 (t, J = 4.8Hz, 8H), 1.71-1.73 (m, 8H), 1.63-1.64 (m, 4H), 1.36 (s, 24H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 153.6, 144.8, 134.7, 134.5, 127.0, 125.3, 123.0, 83.7, 50.7, 25.7, 24.9, 23.8; FTIR: ν_{max} /cm^{-1} 2939, 2824, 1610, 1451, 1362, 1288, 1141, 1091, 850, 657; HRMS: calcd for $C_{40}H_{53}B_2N_4O_6$ (M^++H) 707.4151; found: 707.4375; mp: >300 ^{o}C.

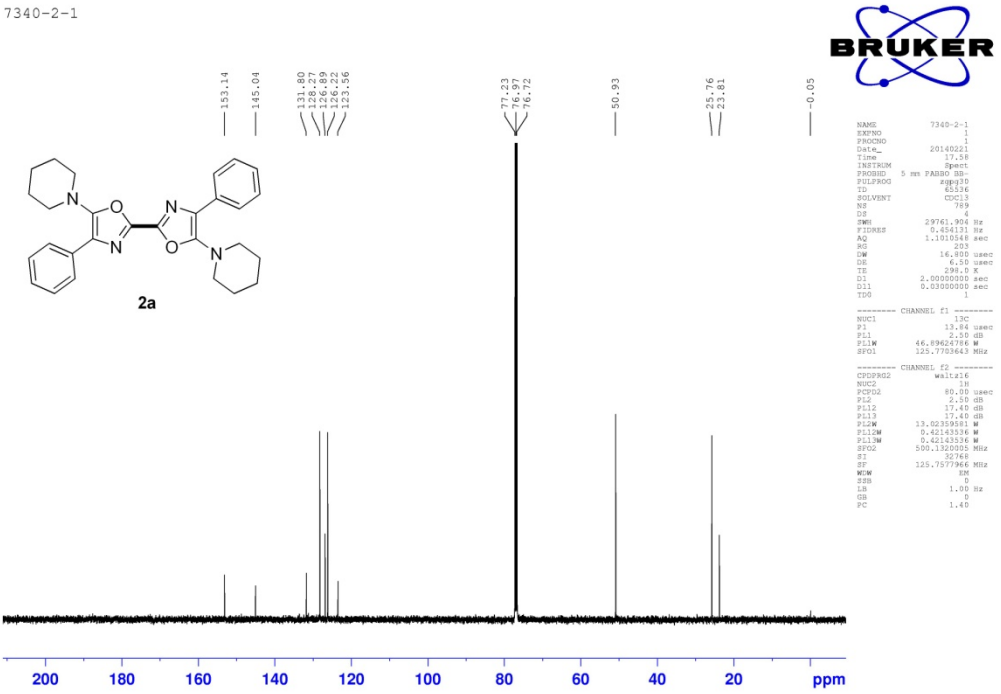


4,4'-di([1,1'-biphenyl]-4-yl)-5,5'-di(piperidin-1-yl)-2,2'-bioxazole (5)

Prepared from 4,4'-bis(4-chlorophenyl)-5,5'-di(piperidin-1-yl)-2,2'-bioxazole (**2h**, 0.1 mmol). ***Procedure:*** An oven-dried Schlenk tube was charged with 4,4'-bis(4-chlorophenyl)-5,5'-di(piperidin-1-yl)-2,2'-bioxazole (**2h**, 52.2 mg, 0.1 mmol), $Pd(OAc)_2$ (4.5 mg, 0.02 mmol), JohnPhos (11.9 mg, 0.04 mmol), phenylboronic acid (73 mg, 0.6 mmol) and KF (34.8 mg, 0.6 mmol). The Schlenk tube was capped with a rubber septum and then evacuated and backfilled with argon (this sequence was carried out two times). THF (1.0 mL) was added via syringe. The septum was then replaced with a Teflon screwcap and the Schlenk tube was sealed. The reaction mixture was heated to 65 °C until **2h** had been completely consumed. The crude reaction mixture was extracted with DCM (20 mL × 3) and washed with brine (20 mL). The organic phase was concentrated in vacuo and the purified product **5** was isolated as a white solid by flash chromatography. Yield: 93%. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.07 (d, *J* = 8.4 Hz, 4H), 7.65-7.68 (m, 8H), 7.46 (t, *J* = 8.4 Hz, 4H), 7.33-7.37 (m, 2H), 3.24 (t, *J* = 5.2Hz, 8H), 1.75-1.81 (m, 8H), 1.63-1.69 (m, 4H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 153.3, 145.1, 140.8, 139.5, 130.8, 128.7, 127.2, 127.0, 126.9, 126.6, 123.3, 51.0, 25.8, 23.8; FTIR: ν_{max} /cm^{-1} 2934, 2852, 1600, 1486, 1449, 1380, 1260, 1109, 856, 697; HRMS: calcd for $C_{40}H_{39}N_4O_2$ (M^++H) 607.3073; found: 607.3064; mp: 115-117 ^{o}C.

IV. Copies of ^{1}H and ^{13}C NMR Spectra





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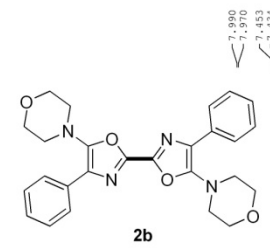

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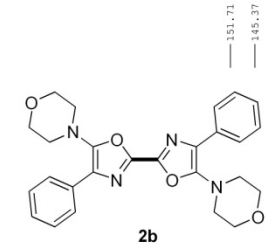
PROCNO 1
Date_ 20140219
Time 9.53
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 362
DW 60.400 usec
DE 6.50 usec
TE 298.2 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 12.58 usec
PL1 0.00 dB
PL1W 10.87646866 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300090 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

7340-3

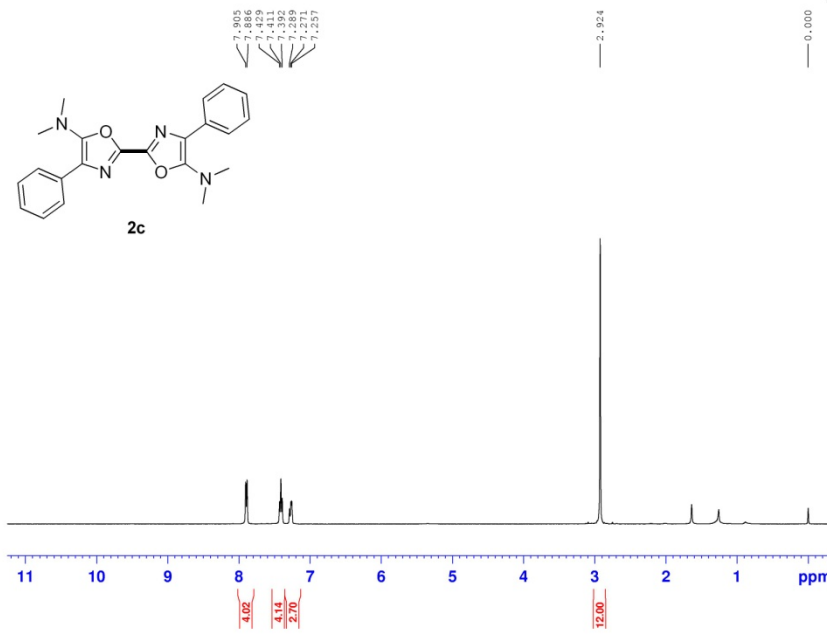


NAME 7340-3
EXPNO 1
PROCNO 1
Date_ 20140219
Time 19.36
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 2048
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 203
DW 16.800 usec
DE 6.50 usec
TE 299.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 13.84 usec
PL1 2.50 dB
PL1W 46.89624786 W
SFO1 125.7703643 MHz

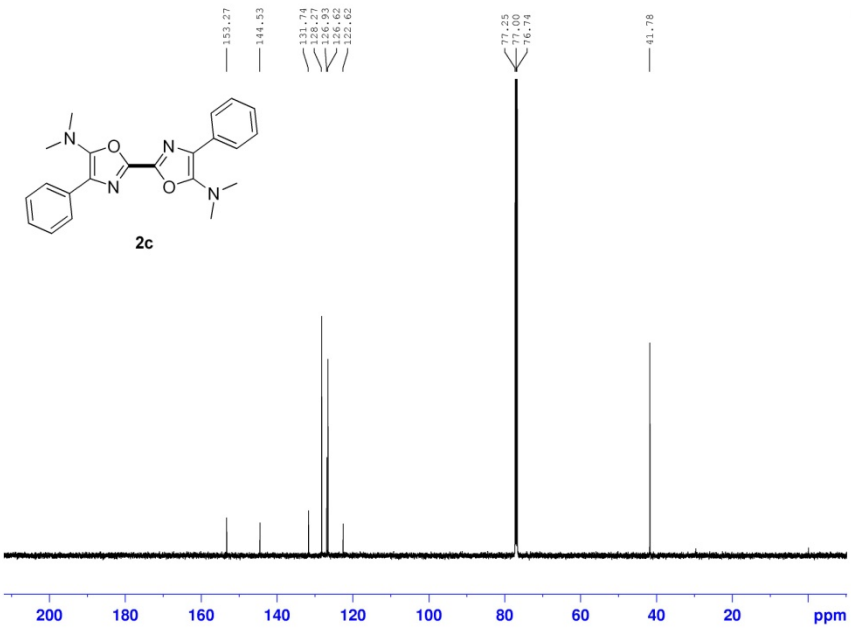
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.50 dB
PL12 17.40 dB
PL13 17.40 dB
PL2W 13.02359581 W
PL12W 0.42143536 W
PL13W 0.42143536 W
SFO2 500.1320005 MHz
SI 32768
SF 125.7577966 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40

7299-2-2



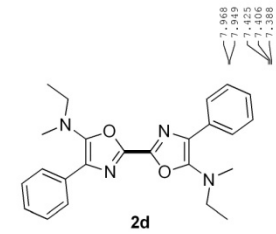
```
NAME                 H
EXPNO              729922
PROCNO                  1
Date_            20131211
Time                9.59
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     8
DS                     2
SWH             8278.146 Hz
FIDRES          0.126314 Hz
AQ              3.9584243 sec
RG                 322.5
DW                60.400 usec
DE                 6.50 usec
TE                298.0 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 12.58 usec
PL1                0.00 dB
PL1W        10.87646866 W
SFO1         400.1324710 MHz
SI                 32768
SF           400.1300101 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

7299-2-2



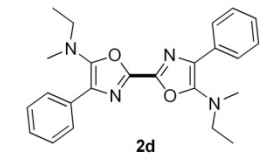
```
NAME             7299-2-2
EXPNO                   1
PROCNO                  1
Date_            20131214
Time                22.24
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   1024
DS                     4
SWH             29761.904 Hz
FIDRES          0.454131 Hz
AQ              1.1010548 sec
RG                   203
DW                16.800 usec
DE                 6.50 usec
TE                295.9 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                 13.84 usec
PL1                2.50 dB
PL1W        46.89624786 W
SFO1         125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2              80.00 usec
PL2                2.50 dB
PL12              17.40 dB
PL13              17.40 dB
PL2W        13.02359581 W
PL12W        0.42143536 W
PL13W        0.42143536 W
SFO2         500.1320005 MHz
SI                 32768
SF           125.7577950 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

7299-4-2



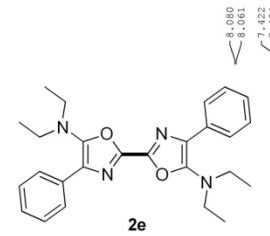

```
NAME                 H
EXPNO             729942
PROCNO                 1
Date_          20131211
Time                10.14
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     8
DS                     2
SWH             8278.146 Hz
FIDRES          0.126314 Hz
AQ              3.9584243 sec
RG                 161.3
DW                60.400 usec
DE                 6.50 usec
TE                298.0 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 12.58 usec
PL1                0.00 dB
PL1W          10.87646866 W
SFO1          400.1324710 MHz
SI                 32768
SF            400.1300113 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

7299-4-2



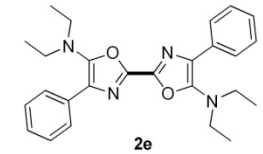

```
NAME              7299-4-2
EXPNO                    1
PROCNO                   1
Date_          20131218
Time                22.17
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  1447
DS                     4
SWH            29761.904 Hz
FIDRES          0.454131 Hz
AQ              1.1010548 sec
RG                   203
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                 13.84 usec
PL1                2.50 dB
PL1W          46.89624786 W
SFO1          125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                  1H
PCPD2              80.00 usec
PL2                2.50 dB
PL12              17.40 dB
PL13              17.40 dB
PL2W          13.02359581 W
PL12W          0.42143536 W
PL13W          0.42143536 W
SFO2          500.1320005 MHz
SI                 32768
SF            125.7577966 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

7299-5


2e

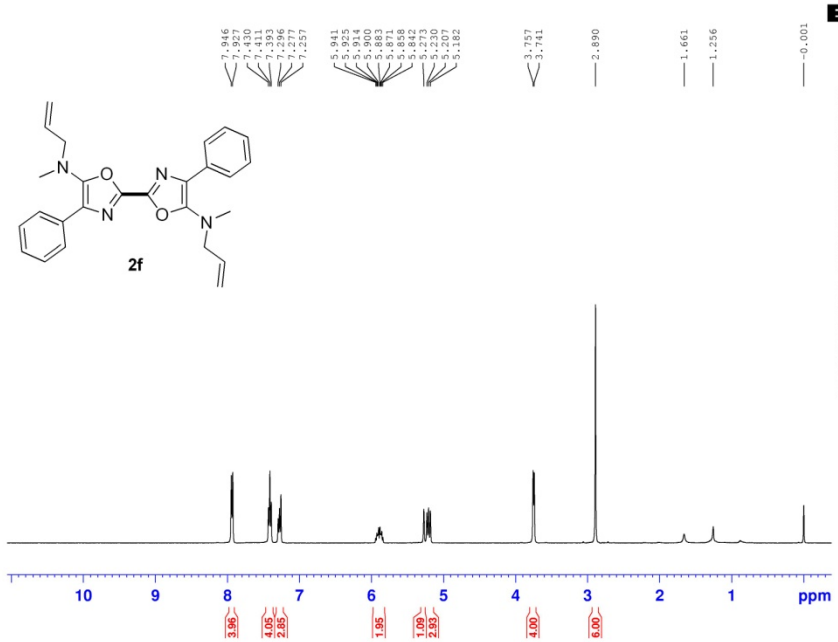
8.080
8.061
7.402
7.384
7.385
7.295
7.277
7.257
3.250
3.232
3.215
3.197
1.137
1.120
1.102
11
10
9
8
7
6
5
4
3
2
1
ppm
3.84
4.05
2.48
8.00
12.03

7299-5


2e

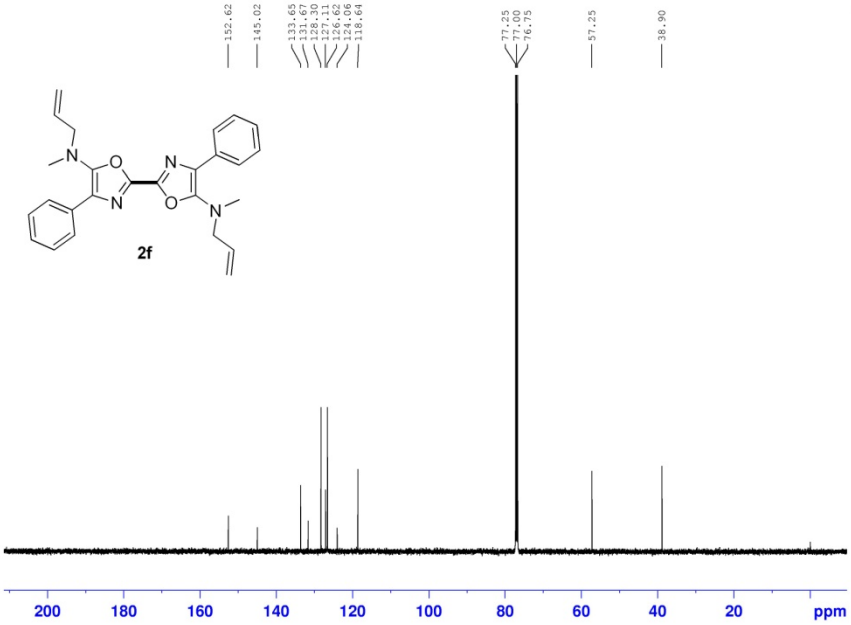
151.16
146.22
131.67
128.81
128.11
127.74
127.19
126.45
77.25
77.00
76.75
46.99
13.13
200
180
160
140
120
100
80
60
40
20
ppm

7299-6



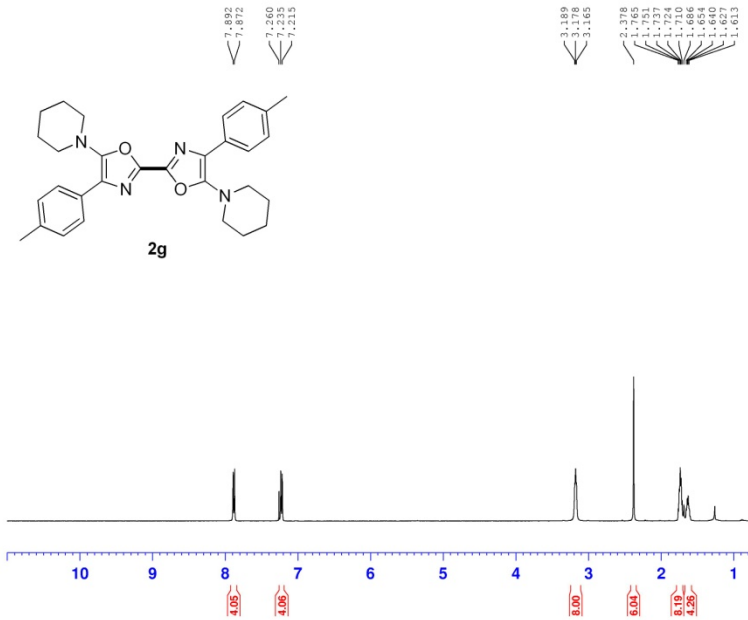

```
NAME                  H
EXPNO             72996
PROCNO                1
Date_          20131206
Time              12.14
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG           zg30
TD                65536
SOLVENT           CDCl3
NS                   8
DS                   2
SWH            8278.146 Hz
FIDRES         0.126314 Hz
AQ             3.9584243 sec
RG                 362
DW                60.400 usec
DE                 6.50 usec
TE               298.0 K
D1            1.00000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1                 1H
P1                12.58 usec
PL1                0.00 dB
PL1W        10.87646866 W
SFO1        400.1324710 MHz
SI                32768
SF          400.1300109 MHz
WDW                 EM
SSB                  0
LB                0.30 Hz
GB                   0
PC                1.00
```

7299-6

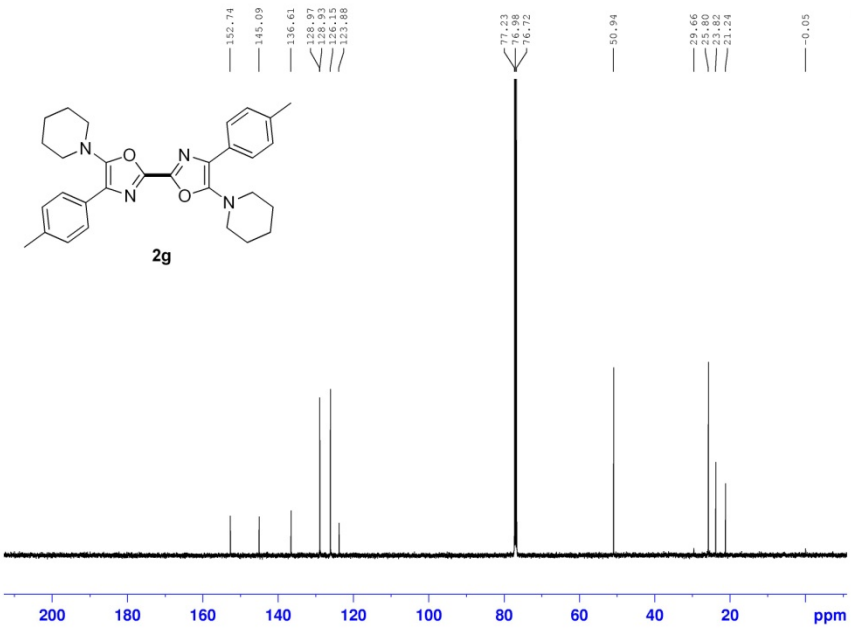



```
NAME              7299-6
EXPNO                 1
PROCNO                1
Date_          20131207
Time              22.21
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG         zgpg30
TD                65536
SOLVENT           CDCl3
NE                 1024
DS                   4
SWH           29761.904 Hz
FIDRES         0.454131 Hz
AQ             1.1010548 sec
RG                 203
DW                16.800 usec
DE                 6.50 usec
TE               297.9 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1                13C
P1                13.84 usec
PL1                2.50 dB
PL1W        46.89624786 W
SFO1       125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                1H
PCPD2             80.00 usec
PL2                2.50 dB
PL12              17.40 dB
PL13              17.40 dB
PL2W        13.02359581 W
PL12W        0.42143536 W
PL13W        0.42143536 W
SFO2       500.1320005 MHz
SI                32768
SF         125.7577931 MHz
WDW                 EM
SSB                  0
LB                1.00 Hz
GB                   0
PC                1.40
```

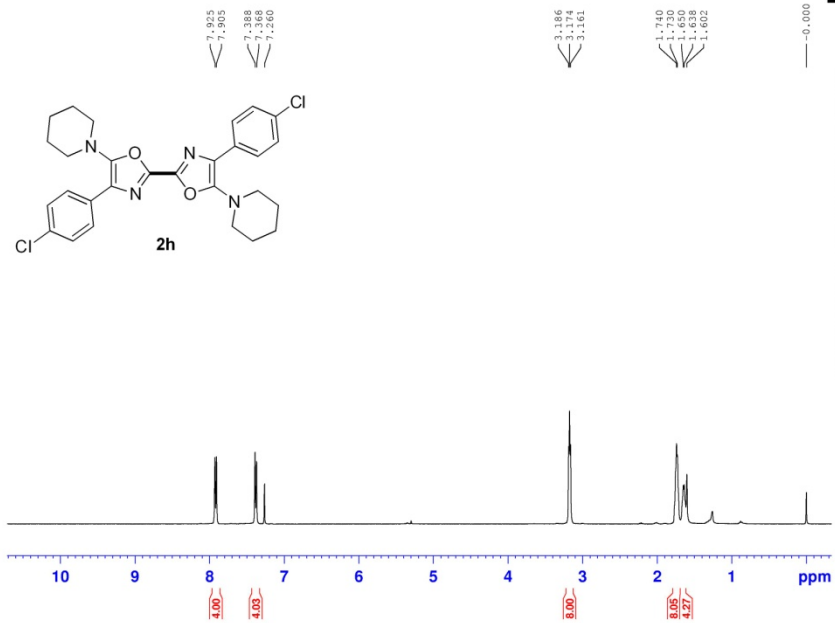
90074-1



90074

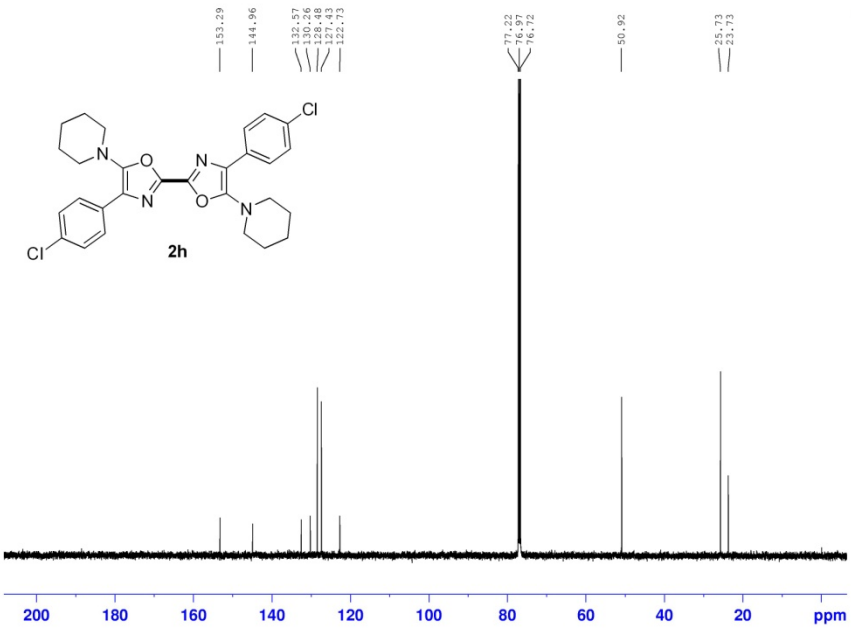


7313-1



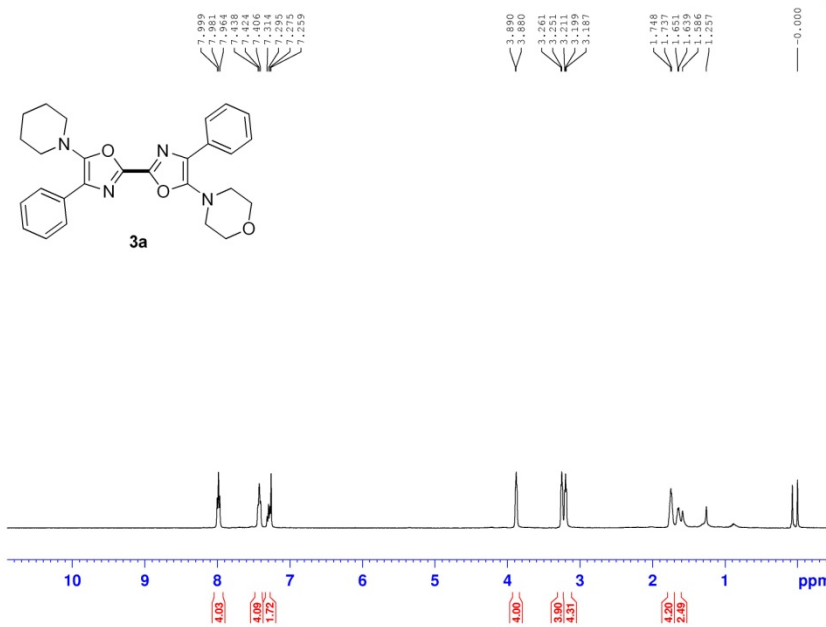

```
NAME                  H
EXPNO             73131
PROCNO                1
Date_          20140103
Time              15.20
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG           zg30
TD                65536
SOLVENT           CDCl3
NS                    8
DS                    2
SWH            8278.146 Hz
FIDRES         0.126314 Hz
AQ             3.9584243 sec
RG                  362
DW                60.400 usec
DE                 6.50 usec
TE               298.0 K
D1             1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                  1H
P1                12.58 usec
PL1                0.00 dB
PL1W          10.87646866 W
SFO1          400.1324710 MHz
SI                 32768
SF            400.1300092 MHz
WDW                   EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```

7313-1




```
NAME               7313-1
EXPNO                   1
PROCNO                  1
Date_           20140106
Time               17.23
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  948
DS                    4
SWH            29761.904 Hz
FIDRES         0.454131 Hz
AQ             1.1010548 sec
RG                  203
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                13.84 usec
PL1                2.50 dB
PL1W          46.89624786 W
SFO1          125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             80.00 usec
PL2                2.50 dB
PL12              17.40 dB
PL13              17.40 dB
PL2W          13.02359581 W
PL12W          0.42143536 W
PL13W          0.42143536 W
SFO2          500.1320005 MHz
SI                 32768
SF            125.7577966 MHz
WDW                   EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

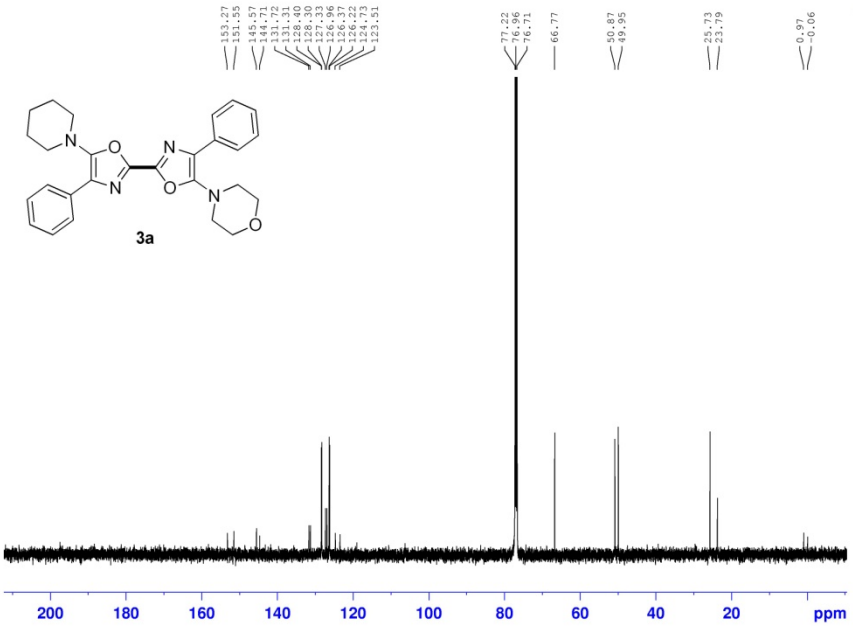
7340-1-1



NAME H
EXPNO 39
PROCNO 1
Date_ 20140219
Time 15.50
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 362
DW 60.400 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 12.58 usec
PL1 0.00 dB
PL1W 10.87646866 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300099 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

7340-1-1

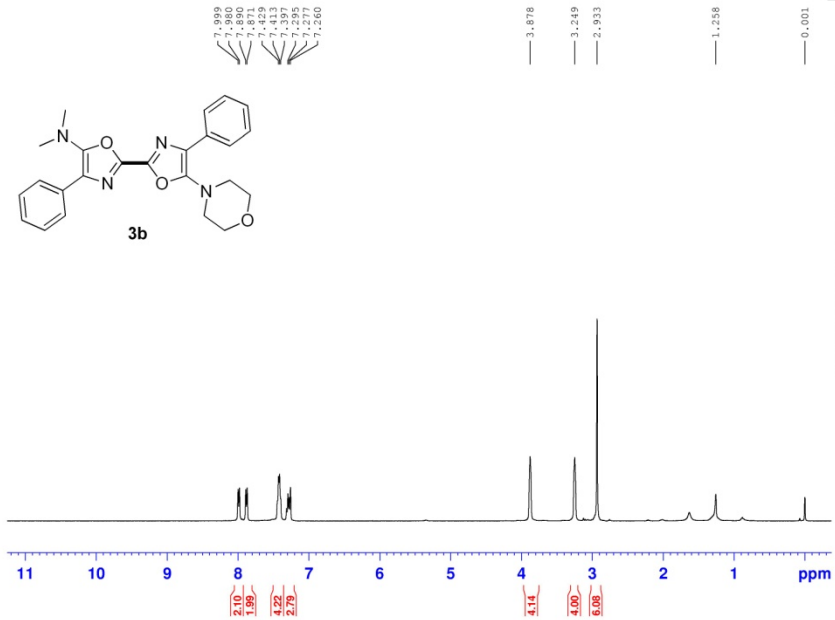


NAME 7340-1-1
EXPNO 1
PROCNO 1
Date_ 20140220
Time 19.00
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1810
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 203
DW 16.800 usec
DE 6.50 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 13.84 usec
PL1 2.30 dB
PL1W 46.89624786 W
SFO1 125.7703643 MHz

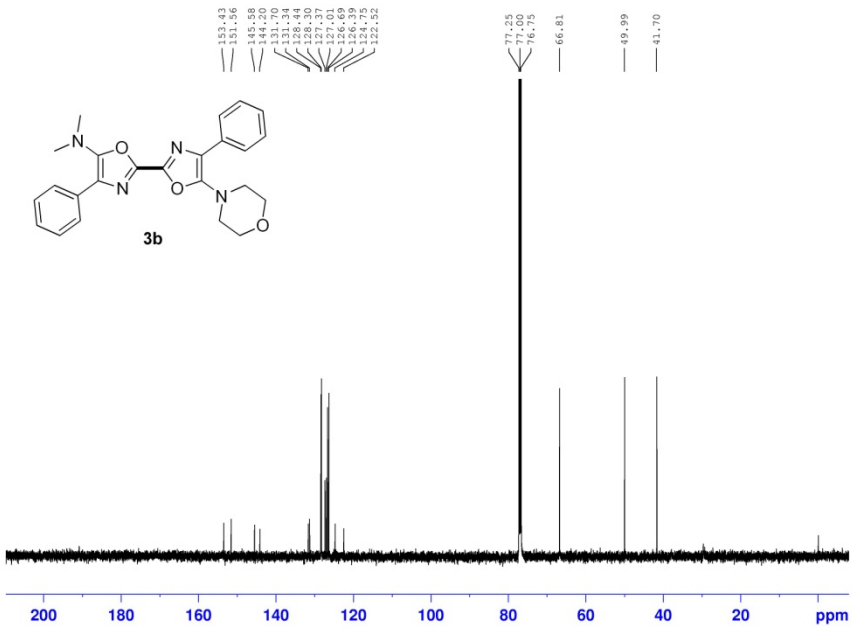
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.50 dB
PL12 17.40 dB
PL13 17.40 dB
PL2W 13.02359581 W
PL12W 0.42143536 W
PL13W 0.42143536 W
SFO2 500.1320005 MHz
SI 32768
SF 125.7577966 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

7321-2


7.999
7.980
7.890
7.871
7.429
7.413
7.397
7.295
7.277
7.260
3.878
3.249
2.933
1.258
0.001
3b
11
10
9
8
7
6
5
4
3
2
1
ppm
2.10
1.99
4.22
2.79
4.14
4.00
6.08

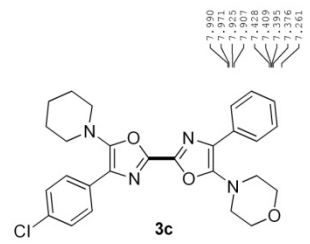

BRUKER
NAME H
EXPNO 7321
PROCNO 1
Date_ 20131230
Time 10.13
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 322.5
DW 60.400 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.58 usec
PL1 0.00 dB
PL1W 10.87646866 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300100 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

7321-2

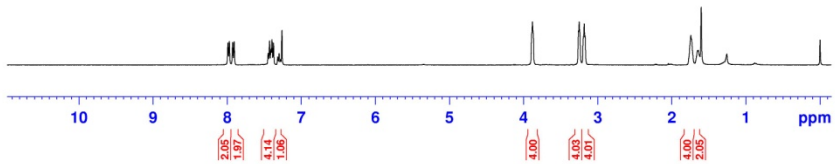

153.43
151.96
145.58
144.20
131.70
131.34
128.44
128.30
127.37
127.01
126.69
126.39
124.75
122.52
77.25
77.00
76.75
66.81
49.99
41.70
3b
200
180
160
140
120
100
80
60
40
20
ppm


BRUKER
NAME 7321-2
EXPNO 1
PROCNO 1
Date_ 20131230
Time 18.16
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1412
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 203
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 13.84 usec
PL1 2.50 dB
PL1W 46.89624786 W
SFO1 125.7703643 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.50 dB
PL12 17.40 dB
PL13 17.40 dB
PL2W 13.02359581 W
PL12W 0.42143536 W
PL13W 0.42143536 W
SFO2 500.1320005 MHz
SI 32768
SF 125.7577931 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

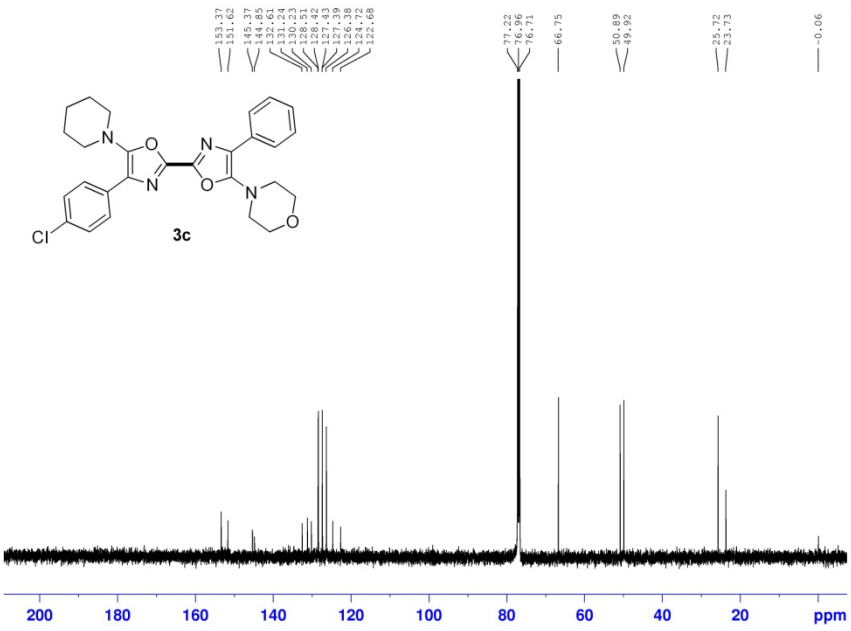
7324-2


7.990
7.971
7.965
7.907
7.428
7.409
7.395
7.376
7.261
3.890
3.881
3.259
3.250
3.183
3.182
3.170
1.744
1.654
1.642
1.603
-0.001
3c


NAME H
EXPNO 73242
PROCNO 1
Date_ 20140108
Time 10.10
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 362
DW 60.400 usec
DE 6.50 usec
TE 296.5 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.58 usec
PL1 0.00 dB
PL1W 10.87646866 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300092 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

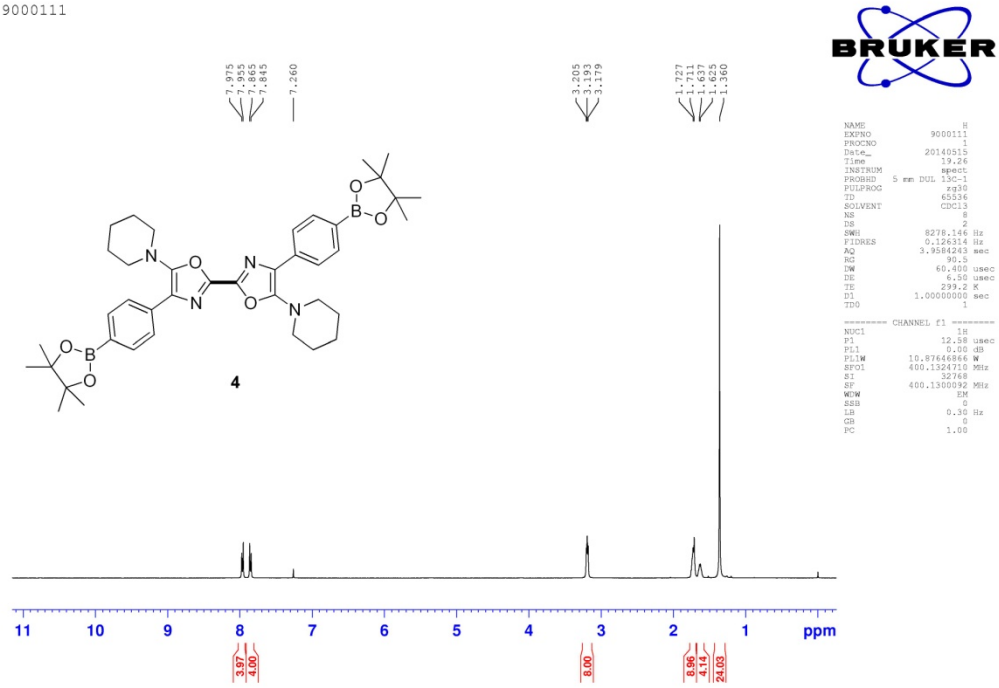

10
9
8
7
6
5
4
3
2
1
ppm
2.05
1.97
4.14
1.06
4.00
4.03
4.01
4.00
2.05

7324-2


153.37
151.62
145.37
144.85
132.61
132.14
130.23
128.51
128.42
128.07
127.43
127.39
126.38
124.72
122.68
77.22
76.96
76.71
66.75
50.89
49.92
25.72
23.73
-0.06
3c
200
180
160
140
120
100
80
60
40
20
ppm


NAME 7324-2
EXPNO 1
PROCNO 1
Date_ 20140108
Time 18.16
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1910
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 203
DW 16.800 usec
DE 6.50 usec
TE 298.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 13.84 usec
PL1 2.50 dB
PL1W 46.89624786 W
SFO1 125.7703643 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.50 dB
PL12 17.40 dB
PL13 17.40 dB
PL2W 13.02359581 W
PL12W 0.42143536 W
PL13W 0.42143536 W
SFO2 500.1320005 MHz
SI 32768
SF 125.7577966 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

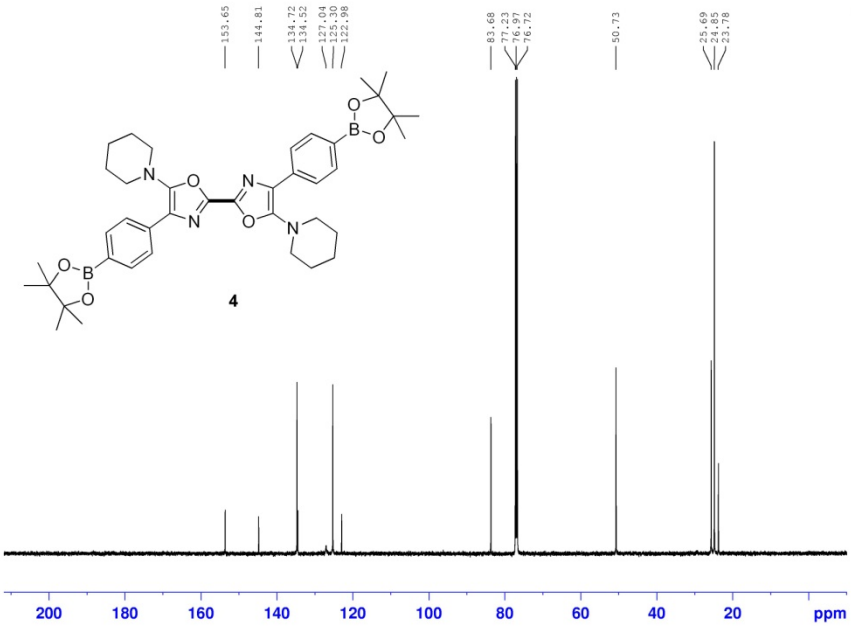
9000111



NAME H
EXPNO 9000111
PROCNO 1
Date_ 20140515
Time 19.26
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 90.5
DW 60.400 usec
DE 6.50 usec
TE 299.2 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 12.58 usec
PL1 0.00 dB
PL1W 10.87646866 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300092 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

9000111

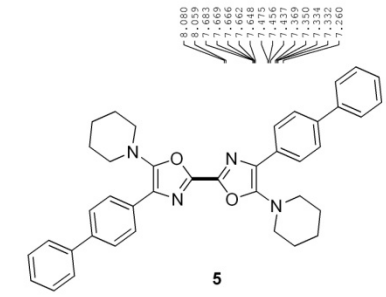


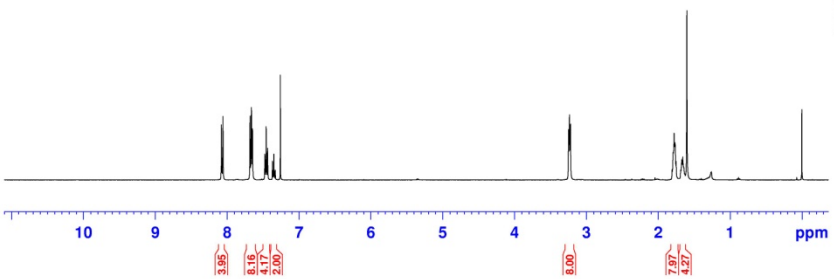
NAME 9000111
EXPNO 3
PROCNO 1
Date_ 20140520
Time 23.57
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 4316
DS 4
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 0.5505524 sec
RG 203
DW 16.800 usec
DE 6.50 usec
TE 299.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 13.84 usec
PL1 2.50 dB
PL1W 46.89624786 W
SFO1 125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.50 dB
PL12 17.40 dB
PL13 17.40 dB
PL2W 13.02359581 W
PL12W 0.42143536 W
PL13W 0.42143536 W
SFO2 500.1320005 MHz
SI 32768
SF 125.7577949 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

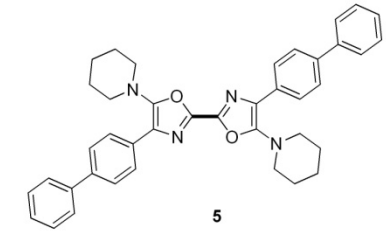
0004-3

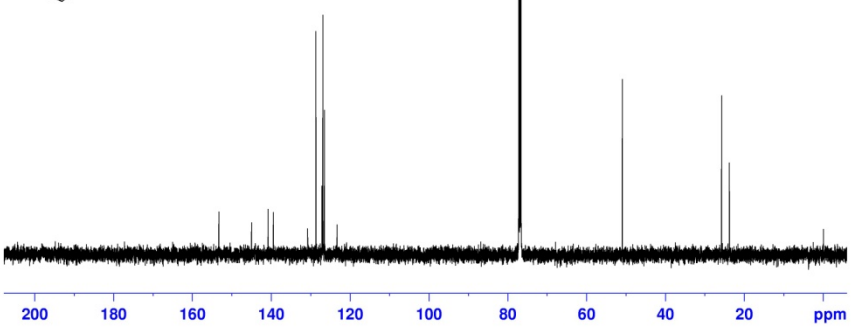

8.080
8.059
7.683
7.669
7.666
7.662
7.648
7.475
7.456
7.437
7.369
7.350
7.334
7.332
7.260
3.248
3.235
3.222
1.807
1.793
1.779
1.766
1.752
1.688
1.673
1.660
1.647
1.633
1.602
5


10
9
8
7
6
5
4
3
2
1
ppm
3.95
8.16
4.17
2.00
8.00
7.97
4.27


NAME H
EXPNO 11
PROCNO 1
Date_ 20140521
Time 10.34
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 362
DW 60.400 usec
DE 6.50 usec
TE 299.1 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.58 usec
PL1 0.00 dB
PL1W 10.87646866 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300093 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

0004-3


153.28
145.06
140.83
139.52
130.81
128.73
127.88
127.46
126.92
126.55
123.34
77.22
77.06
76.71
50.98
25.79
23.82
-0.06
5


200
180
160
140
120
100
80
60
40
20
ppm


NAME 0004-3
EXPNO 1
PROCNO 1
Date_ 20140522
Time 18.56
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 203
DW 16.800 usec
DE 6.50 usec
TE 298.6 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 13.84 usec
PL1 2.50 dB
PL1W 46.89626786 W
SFO1 125.7703643 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.50 dB
PL12 17.40 dB
PL13 17.40 dB
PL2W 13.02359581 W
PL12W 0.42143536 W
PL13W 0.42143536 W
SFO2 500.1320005 MHz
SI 32768
SF 125.7577966 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40