Electronic supplementary materials *Mendeleev Commun.*, 2024, **34**, 566–568

Iodocyclization of olefinic amides with acetoxybenziodoxolone and NaI toward 4-iodomethylated benzoxazines

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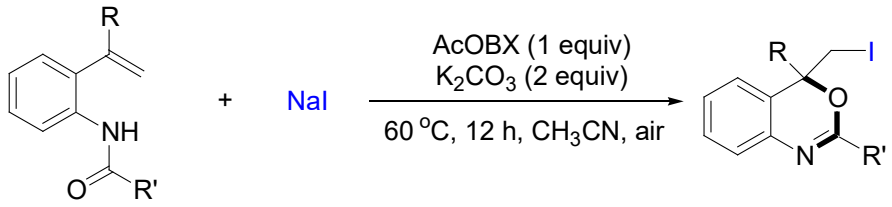
Contents

1. General experimental details

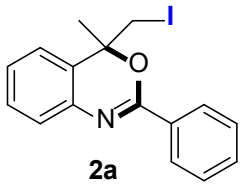
All of the manipulations were performed under air atmosphere. Chemicals were used as received without special purification unless stated otherwise. ^{1}H and ^{13}C NMR were recorded at ambient temperature on a 400 or 300 MHz NMR spectrometer (100 or 75 MHz for ^{13}C NMR). NMR experiments are reported in δ units, parts per million (ppm), and were referenced to $CDCl_3$ (δ 7.26 or 77.0 ppm) as the internal standard. NMR analysis was carried out at 298 K unless noted otherwise. Olefinic amides were prepared according to the published procedure,[S1] acetoxyl-benziodoxolone was prepared according to the published procedure.[S2]

2. General Procedure for the synthesis of 2



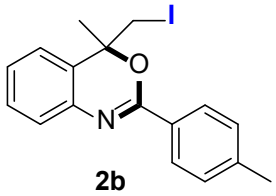
Olefinic amide **1** (0.2 mmol), NaI (0.3 mmol, 45.0 mg), acetoxybenziodoxolone AcOBX (0.3 mmol, 91.5 mg), K_2CO_3 (0.3 mmol, 41.4 mg) and CH_3CN (2.0 mL) was added into a 20 mL Schlenk tube equipped with a Teflon cap. The solution was stirred at room temperature under air for about 12 h. Then, the reaction mixture was concentrated under reduced pressure and the residue was purified by flash column chromatography on silica gel with petroleum ether-ethyl acetate as eluent to give the desired product **2**.

3. Characterization data for the products



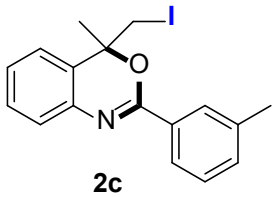
2a

4-Iodomethyl-4-methyl-2-phenyl-4*H*-3,1-benzooxazine **(2a)**[S3] was prepared from *N*-[2-(prop-1-en-2-yl)phenyl]benzamide **1a** (47.4 mg, 0.2 mmol) according to the General Procedure (eluent: ethyl acetate-petroleum ether, 1:10) give the product (66.8 mg, 92% yield) as a white solid. ^{1}H NMR ($CDCl_3$ 400 MHz): δ 8.25-8.28 (m, 2H), 7.45-7.54 (m, 3H), 7.36-7.37 (m, 2H), 7.21-7.26 (m, 1H), 7.16-7.18 (m, 1H), 3.67 (dd, J = 11 Hz, 1H), 3.49 (dd, J = 11 Hz, 1H), 1.93 (s, 3H). ^{13}C NMR ($CDCl_3$ 100 MHz): δ 156.0, 138.7, 132.1, 131.5, 129.4, 128.3, 128.2, 126.9, 126.7, 125.5, 123.0, 77.5, 26.6, 15.7. MS (EI): 363.0 (M^+).



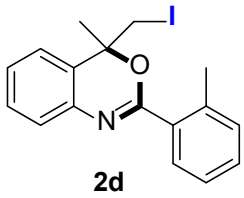
2b

4-Iodomethyl-4-methyl-2-(*p*-tolyl)-4*H*-3,1-benzoxazine **(2b)**[S3] was prepared from 4-methyl-*N*-(2-(prop-1-en-2-yl)phenyl)benzamide **1b** (50.2 mg, 0.2 mmol) according to the General Procedure (eluent: ethyl acetate-petroleum ether, 1:10) give the product (70.1 mg, 93% yield) as a white solid. ^{1}H NMR ($CDCl_3$ 400 MHz): δ 8.17-8.19 (m, 2H), 7.35-7.40 (m, 2H), 7.28-7.31 (m, 2H), 7.22-7.26 (m, 1H), 7.18-7.20 (m, 1H), 3.68 (dd, J = 11 Hz, 1H), 3.49 (dd, J = 11 Hz, 1H), 2.45 (s, 3H), 1.95 (s, 3H). ^{13}C NMR ($CDCl_3$ 100 MHz): δ 156.2, 142.0, 138.9, 129.4, 129.3, 129.0, 128.3, 127.0, 126.5, 125.4, 123.0, 77.2, 26.4, 21.6, 15.8. MS (EI): 377.1 (M^+)



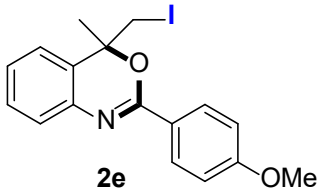
2c

4-Iodomethyl-4-methyl-2-(*m*-tolyl)-4*H*-3,1-benzoxazine **(2c)**[S3] was prepared from 3-methyl-*N*-(2-(prop-1-en-2-yl)phenyl)benzamide **1c** (50.2 mg, 0.2 mmol) according to the General Procedure (eluent: ethyl acetate-petroleum ether, 1:10) give the product (67.2 mg, 89% yield) as a white solid. ^{1}H NMR ($CDCl_3$ 400 MHz): δ 8.05-8.08 (m, 2H), 7.32-7.39 (m, 4H), 7.21-7.25 (m, 1H), 7.16-7.18 (m, 1H), 3.66 (dd, J = 11 Hz, 1H), 3.48 (dd, J = 11 Hz, 1H), 2.45 (s, 3H), 1.94 (s, 3H). ^{13}C NMR ($CDCl_3$ 100 MHz): δ 156.2, 138.8, 137.9, 132.4, 132.1, 129.4, 128.8, 128.1, 127.0, 126.7, 125.5, 125.4, 123.0, 77.4, 26.5, 21.4, 15.7. MS (EI): 377.1 (M^+).



2d

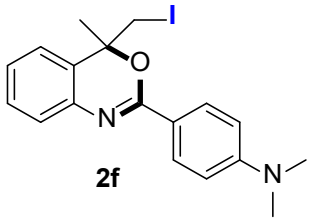
4-Iodomethyl-4-methyl-2-(*o*-tolyl)-4*H*-3,1-benzoxazine **(2d)** was prepared from 2-methyl-*N*-(2-(prop-1-en-2-yl)phenyl)benzamide **1d** (50.2 mg, 0.2 mmol) according to the General Procedure (eluent: ethyl acetate-petroleum ether, 1:10) give the product (62.5 mg, 80% yield) as a white solid. ^{1}H NMR ($CDCl_3$ 400 MHz): δ 8.00-8.03 (m, 2H), 7.25-7.41 (m, 6H), 7.16-7.18 (m, 1H), 3.73 (dd, J = 11 Hz, 1H), 3.59 (dd, J = 11 Hz, 1H), 2.71 (s, 3H), 1.95 (s, 3H). ^{13}C NMR ($CDCl_3$ 100 MHz): δ 157.4, 138.5, 138.4, 132.1, 131.4, 130.4, 129.9, 129.3, 126.9, 126.6, 125.6, 125.4, 123.0, 78.2, 27.0, 21.8, 15.7. MS (EI): 377.1 (M^+).



2e

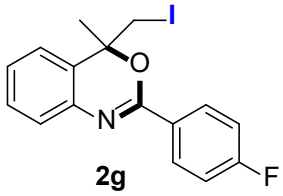
4-Iodomethyl-2-(4-methoxyphenyl)-4-methyl-4*H*-3,1-benzoxazine **(2e)**[S3] was prepared from 4-methoxy-*N*-(2-(prop-1-en-2-yl)phenyl)benzamide **1e** (53.4 mg, 0.2 mmol) according to the General Procedure (eluent: ethyl acetate-petroleum ether, 1:10) give the product (73.1 mg, 93% yield) as a white solid. ^{1}H NMR ($CDCl_3$ 400 MHz): δ 8.19-8.23 (m, 2H), 7.31-7.35 (m, 2H),

7.15-7.22 (m, 2H), 6.95-6.99 (m, 2H), 3.87 (s, 3H), 3.66 (dd, J = 11 Hz, 1H), 3.45 (dd, J = 11 Hz, 1H), 1.92 (s, 3H). ^{13}C NMR ($CDCl_3$ 100 MHz): δ 162.4, 156.0, 139.0, 130.2, 129.4, 126.8, 126.3, 125.2, 124.5, 122.9, 113.6, 77.1, 55.3, 26.4, 15.8. MS (EI): 393.0 (M^+).



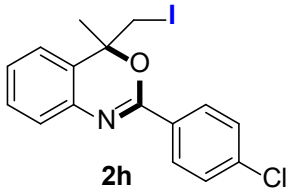
2f

4-(4-Iodomethyl-4-methyl-4*H*-3,1-benzoxazin-2-yl)-*N,N*-dimethylaniline (**2f**) was prepared from 4-dimethylamino-*N*-(2-(prop-1-en-2-yl)phenyl)benzamide **1f** (56.0 mg, 0.2 mmol) according to the General Procedure (eluent: ethyl acetate-petroleum ether, 1:10) give the product (60.9 mg, 75% yield) as a white solid. ^{1}H NMR ($CDCl_3$ 400 MHz): δ 8.13-8.15 (m, 2H), 7.31-7.32 (m, 2H), 7.15-7.16 (m, 2H), 6.71-6.73 (m, 2H), 3.87 (s, 3H), 3.66 (dd, *J* = 11 Hz, 1H), 3.43 (dd, *J* = 11 Hz, 1H), 3.05 (s, 3H), 1.91 (s, 3H). ^{13}C NMR ($CDCl_3$ 100 MHz): δ 156.8, 152.6, 139.6, 130.0, 129.3, 126.8, 125.6, 124.8, 122.9, 119.0, 111.0, 40.1, 26.2, 15.9. MS (EI): 406.1 (M^+).



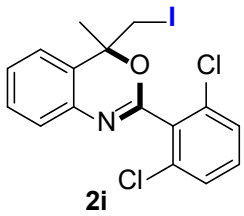
2g

2-(4-Fluorophenyl)-4-iodomethyl-4-methyl-4*H*-3,1-benzoxazine (**2g**)[S3] was prepared from 4-fluoro-*N*-[2-(prop-1-en-2-yl)phenyl]benzamide **1g** (51.0 mg, 0.2 mmol) according to the General Procedure (eluent: ethyl acetate-petroleum ether, 1:10) give the product (55.6 mg, 73% yield) as a white solid. ^{1}H NMR ($CDCl_3$ 400 MHz): δ 8.24-8.28 (m, 2H), 7.31-7.38 (m, 2H), 7.21-7.25 (m, 1H), 7.11-7.18 (m, 3H), 3.66 (dd, J = 11 Hz, 1H), 3.46 (dd, J = 11 Hz, 1H), 1.93 (s, 3H). ^{13}C NMR ($CDCl_3$ 100 MHz): δ 165.0 (d, *J* = 250.4 Hz), 155.1, 138.7, 130.6 (d, *J* = 8.8 Hz), 129.5, 128.3 (d, *J* = 2.9 Hz), 126.8, 125.5, 123.0, 116.6, 115.3 (d, *J* = 21.7 Hz), 77.6, 26.5, 15.8. MS (EI): 381 (M^+).



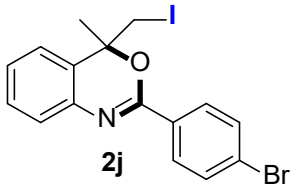
2h

2-(4-Chlorophenyl)-4-iodomethyl-4-methyl-4*H*-3,1-benzoxazine (**2h**)[S3] was prepared from 4-chloro-*N*-(2-(prop-1-en-2-yl)phenyl)benzamide **1h** (54.2 mg, 0.2 mmol) according to the General Procedure (eluent: ethyl acetate-petroleum ether, 1:10) give the product (67.5 mg, 85% yield) as a white solid. ^{1}H NMR ($CDCl_3$ 400 MHz): δ 8.20-8.22 (m, 2H), 7.44-7.47 (m, 2H), 7.33-7.40 (m, 2H), 7.23-7.28 (m, 1H), 7.17-7.19 (m, 1H), 3.67 (dd, J = 11 Hz, 1H), 3.47 (dd, J = 11 Hz, 1H), 1.94 (s, 3H). ^{13}C NMR ($CDCl_3$ 100 MHz): δ 155.1, 138.5, 137.7, 130.6, 129.6, 129.5, 128.5, 127.0, 126.9, 125.5, 123.0, 77.6, 26.5, 15.7. MS (EI): 397.0 (M^+).



2i

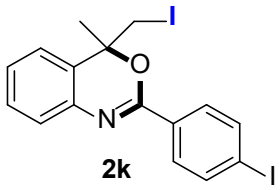
2-(2,6-Dichlorophenyl)-4-iodomethyl-4-methyl-4*H*-3,1-benzoxazine (**2i**) was prepared from 2,6-dichloro-*N*-(2-(prop-1-en-2-yl)phenyl)benzamide **1i** (61.0 mg, 0.2 mmol) according to the General Procedure (eluent: ethyl acetate-petroleum ether, 1:10) give the product (54.3 mg, 63% yield) as a white solid. ^{1}H NMR ($CDCl_3$ 400 MHz): δ 7.36-7.40 (m, 3H), 7.28-7.33 (m, 3H), 7.16-7.18 (m, 1H), 3.82 (dd, J = 11 Hz, 1H), 3.47 (dd, J = 11 Hz, 1H), 1.96 (s, 3H). ^{13}C NMR ($CDCl_3$ 100 MHz): δ 153.8, 137.2, 134.1, 132.8, 130.8, 129.5, 128.0, 127.7, 126.6, 125.7, 123.4, 79.9, 28.0, 15.6. MS (EI): 431 (M^+).



2j

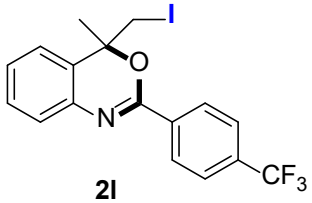
2-(4-Bromophenyl)-4-iodomethyl-4-methyl-4*H*-3,1-benzoxazine **(2j)**[S3] was prepared from 4-bromo-*N*-(2-(prop-1-en-2-yl)phenyl)benzamide **1j** (63.0 mg, 0.2 mmol) according to the General Procedure (eluent: ethyl acetate-petroleum ether, 1:10) give the product (65.6 mg, 75%

yield) as a white solid. ^{1}H NMR ($CDCl_3$ 400 MHz): δ 8.13-8.15 (m, 2H), 7.60-7.63 (m, 2H), 7.33-7.40 (m, 2H), 7.24-7.28 (m, 1H), 7.17-7.19 (m, 1H), 3.67 (dd, J = 11 Hz, 1H), 3.47 (dd, J = 11 Hz, 1H), 1.94 (s, 3H). ^{13}C NMR ($CDCl_3$ 100 MHz): δ 155.2, 138.6, 131.6, 131.2, 130.0, 129.6, 127.1, 127.0, 126.4, 125.7, 123.1, 77.7, 26.6, 15.8. MS (EI): 441 (M^+).



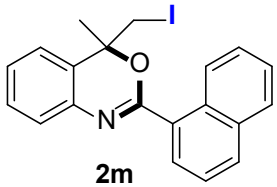
2k

4-Iodomethyl-2-(4-iodophenyl)-4-methyl-4*H*-3,1-benzoxazine (**2k**) was prepared from 4-bromo-*N*-(2-(prop-1-en-2-yl)phenyl)benzamide **1k** (72.6 mg, 0.2 mmol) according to the General Procedure (eluent: ethyl acetate-petroleum ether, 1:10) give the product (81.2 mg, 83% yield) as a white solid. ^{1}H NMR ($CDCl_3$ 400 MHz): δ 7.97-8.00 (m, 2H), 7.81-7.84 (m, 2H), 7.33-7.40 (m, 2H), 7.23-7.28 (m, 1H), 7.17-7.19 (m, 1H), 3.66 (dd, *J* = 11 Hz, 1H), 3.47 (dd, *J* = 11 Hz, 1H), 1.94 (s, 3H). ^{13}C NMR ($CDCl_3$ 100 MHz): δ 155.3, 138.5, 137.4, 131.7, 129.8, 129.5, 127.0, 126.9, 125.6, 123.0, 98.8, 77.6, 26.5, 15.8. MS (EI): 488.9 (M^+).



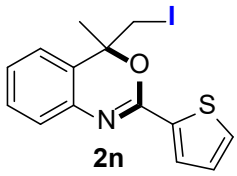
2l

4-Iodomethyl-4-methyl-2-(4-trifluoromethylphenyl)-4*H*-3,1-benzoxazine **(2l)**[S3] was prepared from *N*-(2-(prop-1-en-2-yl)phenyl)-4-(trifluoromethyl)benzamide **1l** (61.2 mg, 0.2 mmol) according to the General Procedure (eluent: ethyl acetate-petroleum ether, 1:10) give the product (78.4 mg, 91% yield) as a white solid. ^{1}H NMR ($CDCl_3$ 400 MHz): δ 8.35-8.37 (m, 2H), 7.70-7.72 (m, 2H), 7.34-7.37 (m, 2H), 7.23-7.27 (m, 1H), 7.16-7.18 (m, 1H), 3.66 (dd, *J* = 11 Hz, 1H), 3.47 (dd, *J* = 11 Hz, 1H), 1.94 (s, 3H). ^{13}C NMR ($CDCl_3$ 100 MHz): δ 154.6, 138.3, 135.6, 132.9 (q, *J* = 32 Hz), 129.6, 128.6, 127.4, 127.0, 125.8, 125.1 (q, *J* = 4 Hz), 123.0, 121.2 (q, *J* = 271 Hz), 77.9, 26.6, 15.7. MS (EI): 431.0 (M^+).



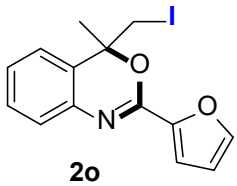
2m

4-Iodomethyl-4-methyl-2-(naphthalen-1-yl)-4*H*-3,1-benzoxazine **(2m)** was prepared from *N*-(2-(prop-1-en-2-yl)phenyl)-1-naphthamide **1m** (57.4 mg, 0.2 mmol) according to the General Procedure (eluent: ethyl acetate-petroleum ether, 1:10) give the product (61.9 mg, 75% yield) as a white solid. ^{1}H NMR ($CDCl_3$ 400 MHz): δ 9.16-9.18 (m, 1H), 8.32-8.34 (m, 1H), 8.00-8.02 (m, 1H), 7.92-7.94 (m, 1H), 7.55-7.67 (m, 3H), 7.41-7.50 (m, 2H), 7.28-7.33 (m, 1H), 7.21-7.23 (m, 1H), 3.80 (dd, J = 11 Hz, 1H), 3.63 (dd, J = 11 Hz, 1H), 2.01 (s, 3H). ^{13}C NMR ($CDCl_3$ 100 MHz): δ 156.6, 138.6, 133.9, 131.8, 131.1, 129.5, 129.3, 129.2, 128.4, 127.2, 127.1, 126.8, 126.2, 126.0, 125.6, 124.7, 123.0, 78.2, 27.0, 15.7. MS (EI): 413.0 (M^+).



2n

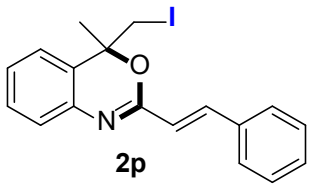
4-Iodomethyl-4-methyl-2-(thiophen-2-yl)-4*H*-3,1-benzoxazine **(2n)** was prepared from *N*-(2-(prop-1-en-2-yl)phenyl)thiophene-2-carboxamide **2n** (48.6 mg, 0.2 mmol) according to the General Procedure (eluent: ethyl acetate-petroleum ether, 1:10) give the product (60.5 mg, 82% yield) as a white solid. ^{1}H NMR ($CDCl_3$ 400 MHz): δ 7.86-7.87 (m, 1H), 7.50-7.52 (m, 1H), 7.29-7.36 (m, 2H), 7.11-7.22 (m, 3H), 3.66 (dd, J = 11 Hz, 1H), 3.44 (dd, J = 11 Hz, 1H), 1.91 (s, 3H). ^{13}C NMR ($CDCl_3$ 100 MHz): δ 152.6, 138.7, 136.4, 130.8, 130.6, 129.4, 127.7, 126.8, 126.6, 125.2, 123.0, 77.7, 26.3, 15.5. MS (EI): 369 (M^+).



2o

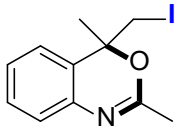
2-(Furan-2-yl)-4-iodomethyl-4-methyl-4*H*-3,1-benzoxazine **(2o)** was prepared from *N*-(2-(prop-1-en-2-yl)phenyl)furan-2-carboxamide **2o** (45.4 mg, 0.2 mmol) according to the General Procedure (eluent: ethyl acetate-petroleum ether, 1:10) give the product (46.5 mg, 66%

yield) as a white solid. ^{1}H NMR ($CDCl_3$ 400 MHz): δ 7.62 – 7.61 (m, 1H), 7.39 – 7.31 (m, 2H), 7.22 – 7.18 (m, 2H), 7.13 (dd, J = 7.8, 1.4 Hz, 1H), 6.53 (dd, J = 3.4, 1.7 Hz, 1H), 3.63 (d, J = 11.1 Hz, 1H), 3.41 (d, J = 11.2 Hz, 1H), 1.89 (s, 3H). ^{13}C NMR ($CDCl_3$ 100 MHz): δ 148.9, 146.2, 145.8, 138.1, 129.5, 126.9, 126.8, 125.6, 123.0, 115.8, 111.9, 77.5, 26.2, 15.2. MS (EI): 353.0 (M^+).



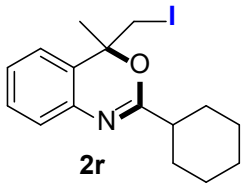
2p

4-Iodomethyl-4-methyl-2-styryl-4*H*-3,1-benzoxazine **(2p)** was prepared from *N*-(2-(prop-1-en-2-yl)phenyl)cinnamamide **1p** (52.6 mg, 0.2 mmol) according to the General Procedure (eluent: ethyl acetate-petroleum ether, 1:10) give the product (65.3 mg, 84% yield) as a white solid. ^{1}H NMR ($CDCl_3$ 400 MHz): δ 7.68-7.72 (m, 1H), 7.58-7.60 (m, 2H), 7.34-7.43 (m, 3H), 7.28-7.30 (m, 1H), 7.21-7.25 (m, 1H), 7.16-7.18 (m, 1H), 3.64 (dd, J = 11 Hz, 1H), 3.45 (dd, J = 11 Hz, 1H), 1.92 (s, 3H). ^{13}C NMR ($CDCl_3$ 100 MHz): δ 156.6, 140.3, 139.0, 135.4, 129.6, 128.9, 127.8, 127.3, 126.9, 125.4, 123.2, 121.5, 77.1, 26.4, 15.9. MS (EI): 389 (M^+).



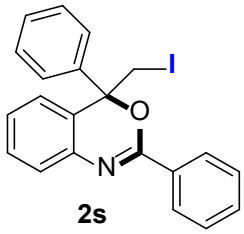
2q

4-Iodomethyl-2,4-dimethyl-4*H*-3,1-benzoxazine **(2q)**[S4] was prepared from *N*-(2-(prop-1-en-2-yl)phenyl)acetamide **1q** (35.0 mg, 0.2 mmol) according to the General Procedure (eluent: ethyl acetate-petroleum ether, 1:10) give the product (54.2 mg, 90% yield) as a white solid. The compound is unknown. ^{1}H NMR ($CDCl_3$ 400 MHz): δ 7.28-7.32 (m, 1H), 7.14-7.20 (m, 2H), 7.07-7.09 (m, 1H), 3.57 (dd, J = 11 Hz, 1H), 3.38 (dd, J = 11 Hz, 1H), 2.17 (s, 3H), 1.81 (s, 3H). ^{13}C NMR ($CDCl_3$ 100 MHz): δ 159.5, 138.2, 129.4, 126.6, 126.4, 124.6, 123.0, 77.4, 26.6, 21.9, 16.3. MS (EI): 301.0 (M^+).



2r

2-Cyclohexyl-4-iodomethyl-4-methyl-4*H*-3,1-benzoxazine (**2r**) was prepared from *N*-(2-(prop-1-en-2-yl)phenyl)cyclohexanecarboxamide **1r** (48.6 mg, 0.2 mmol) according to the General Procedure (eluent: ethyl acetate-petroleum ether, 1:10) give the product (68.6 mg, 83% yield) as a white solid. The compound is unknown. ^{1}H NMR ($CDCl_3$ 400 MHz): δ 7.28-7.32 (m, 1H), 7.15-7.19 (m, 2H), 7.06-7.08 (m, 1H), 3.56 (dd, J = 11 Hz, 1H), 3.44 (dd, J = 11 Hz, 1H), 2.31-2.39 (m, 1H), 1.99-2.05 (m, 2H), 1.80-1.85 (m, 5H), 1.50-1.73 (m, 3H), 1.20-1.41 (m, 3H). ^{13}C NMR ($CDCl_3$ 100 MHz): δ 164.5, 138.3, 129.2, 126.6, 126.3, 124.8, 123.0, 76.9, 43.9, 29.6, 29.5, 26.8, 25.8, 25.7, 15.8. MS (EI): 369.1 (M^+).



2s

4-Iodomethyl-2,4-diphenyl-4*H*-3,1-benzoxazine (**2s**)[s5] was prepared from *N*-(2-(1-phenylvinyl)phenyl)benzamide **1s** (59.8 mg, 0.2 mmol) according to the General Procedure (eluent: ethyl acetate-petroleum ether, 1:10) give the product (46.7 mg, 55% yield) as a white solid. The compound is unknown. ^{1}H NMR ($CDCl_3$ 400 MHz): δ 8.31-8.34 (m, 2H), 7.47-7.54 (m, 3H), 7.42-7.45 (m, 2H), 7.31-7.39 (m, 5H), 7.22-7.25 (m, 1H), 7.10-7.12 (m, 1H), 4.03 (s, 2H). ^{13}C NMR ($CDCl_3$ 100 MHz): δ 155.9, 140.3, 139.2, 132.0, 131.6, 129.6, 128.5, 128.4, 128.3, 128.2, 126.6, 126.5, 126.1, 125.6, 124.5, 81.6, 13.2. MS (EI): 425.0 (M^+).

References:

S1. X.-Q. Chu, X.-P. Xu, H. Meng and S.-J. Ji, *RSC Adv.,* 2015, **5**, 67829.

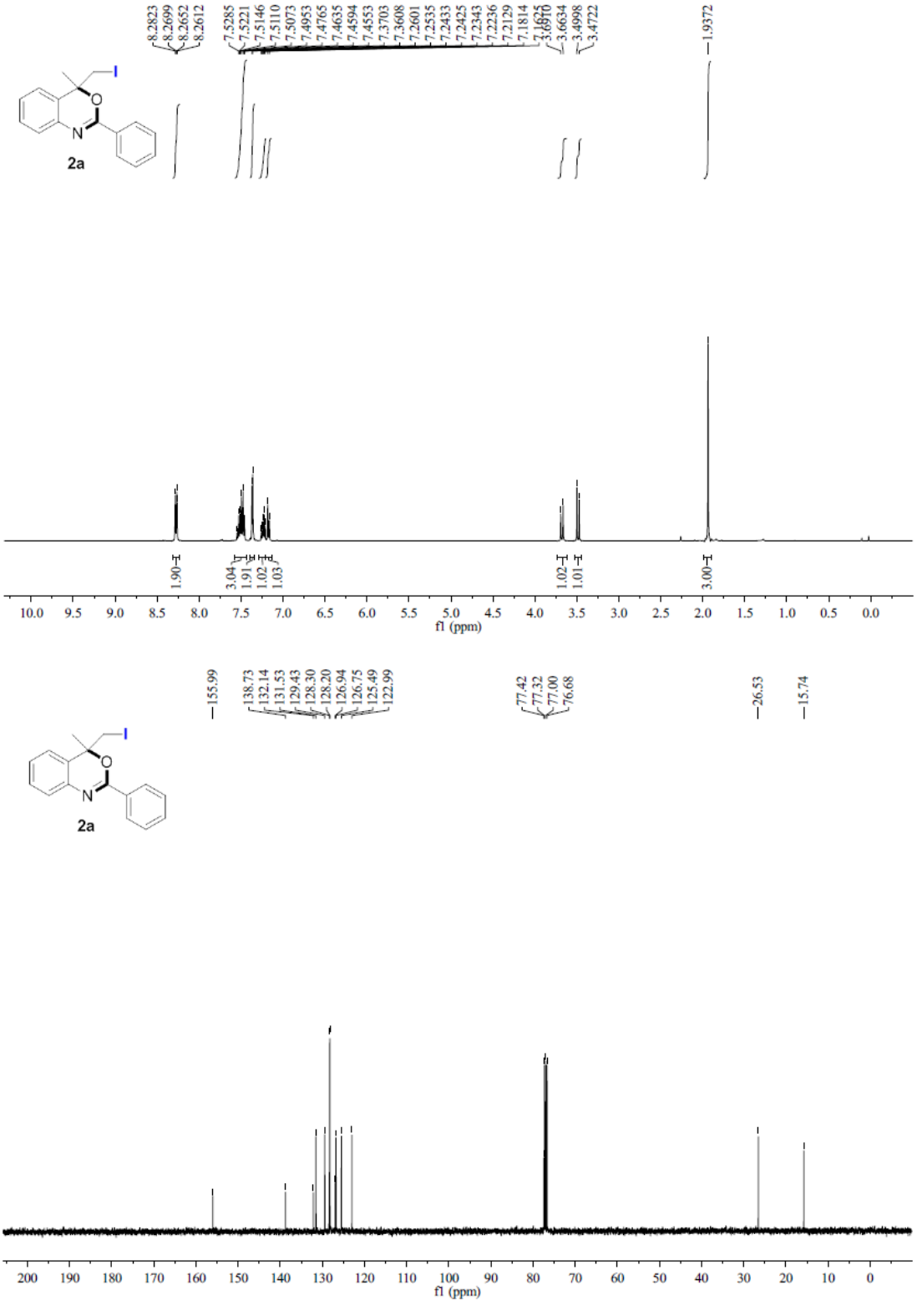
S2. S. Alazet, J. Preindl, R. Simonet-Davin, S. Nicolai, A. Nanchen, T. Meyer and J. Waser, *J. Org. Chem*., 2018, **83**, 12334

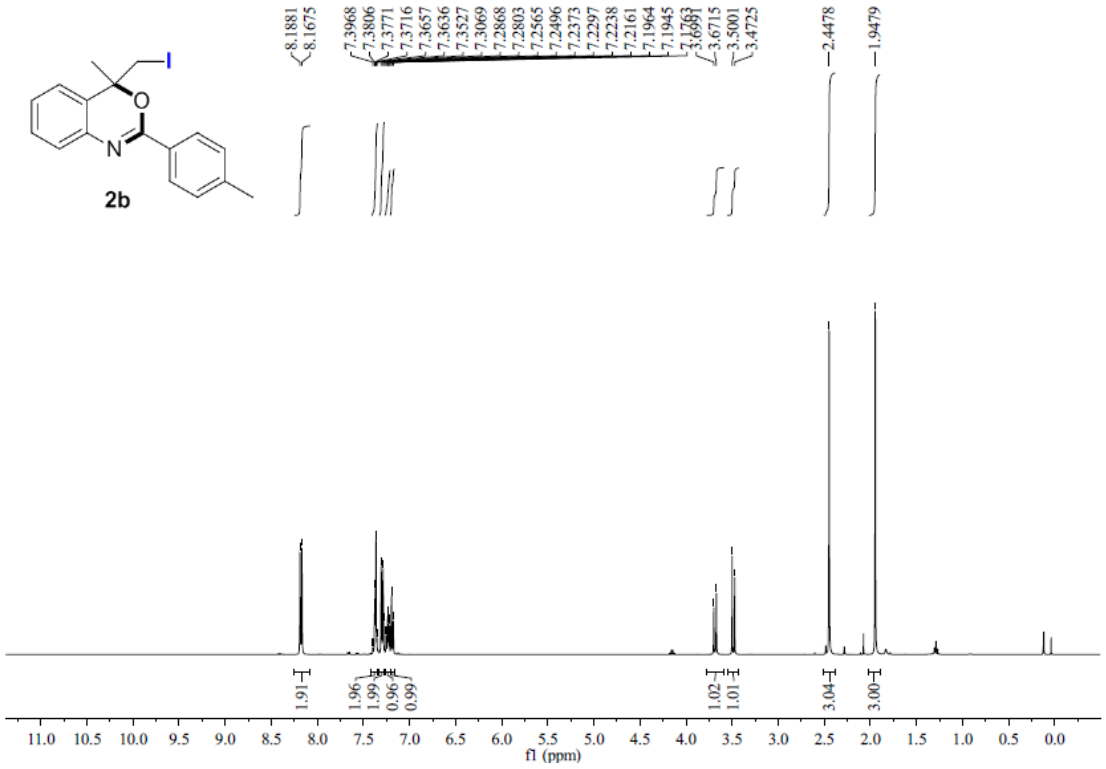
S3. T.-T. Cao, W.-K. Zhang, F.-H. Qin, Q.-Q. Kang, Y. Dong, Q. Li, C. Kang and W.-T. Wei, *ACS Sustainable Chem. Eng.*, 2020, **8**, 16946.

S4. Kentaro Okuma, Takumi Yasuda, Kosei Shioji, Yoshinobu Yokomori, *Bull. Chem. Soc. Japan*, 2007, **80**, 1824.

S5. Q. Xie, H.-J. Long, Q.-Y. Zhang, P. Tang, and J. Deng, *J. Org. Chem.* 2020, **85**, 1882.

4. Copies of ^{1}H NMR and ^{13}C NMR spectra of 2a-s

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O
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2b
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1.99
0.96
0.99
1.02
1.01
3.04
3.00
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
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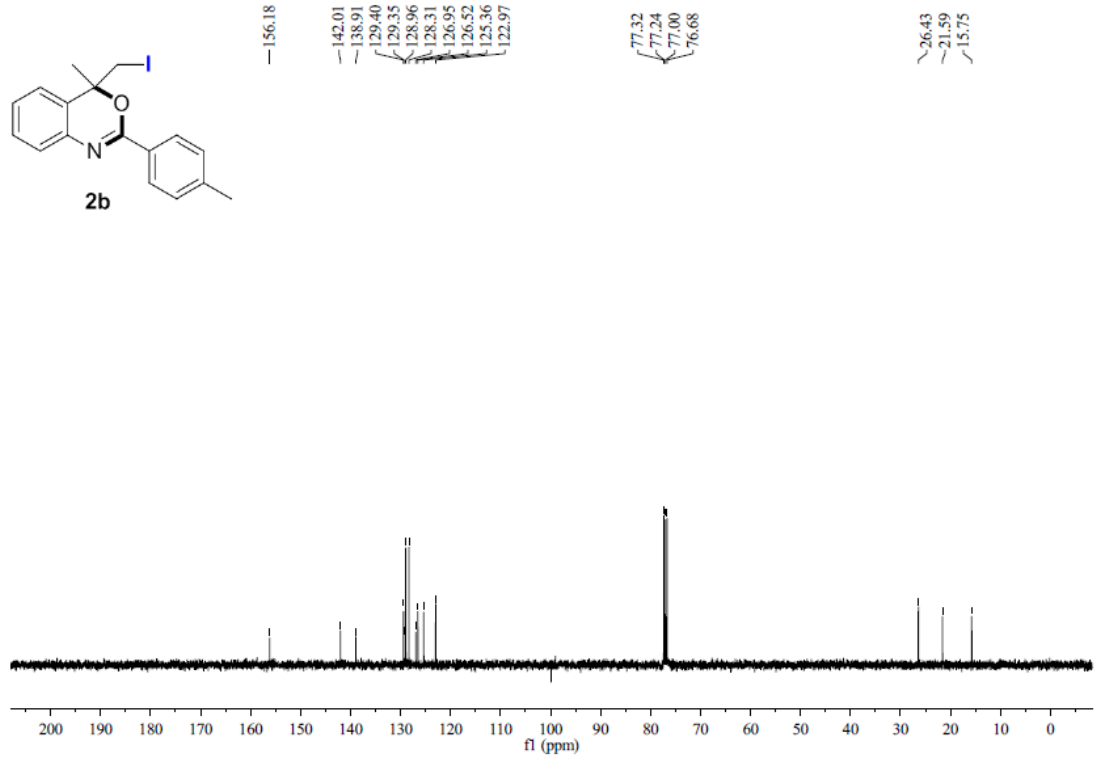
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77.00
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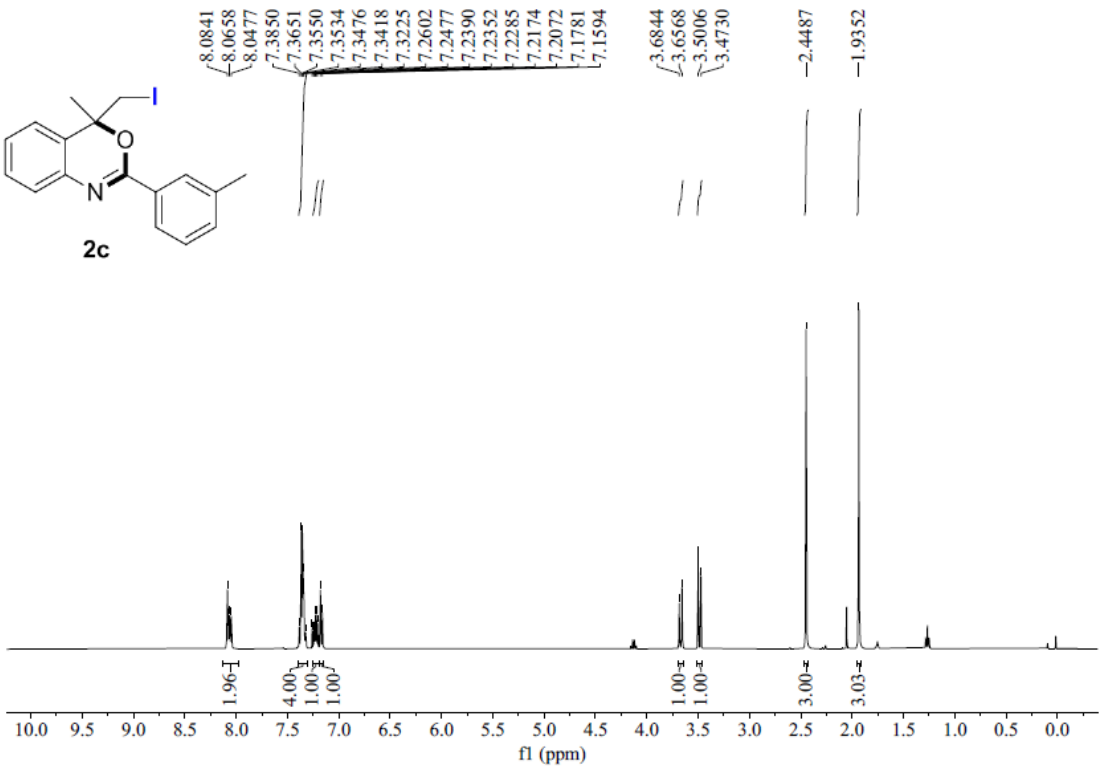
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1.00
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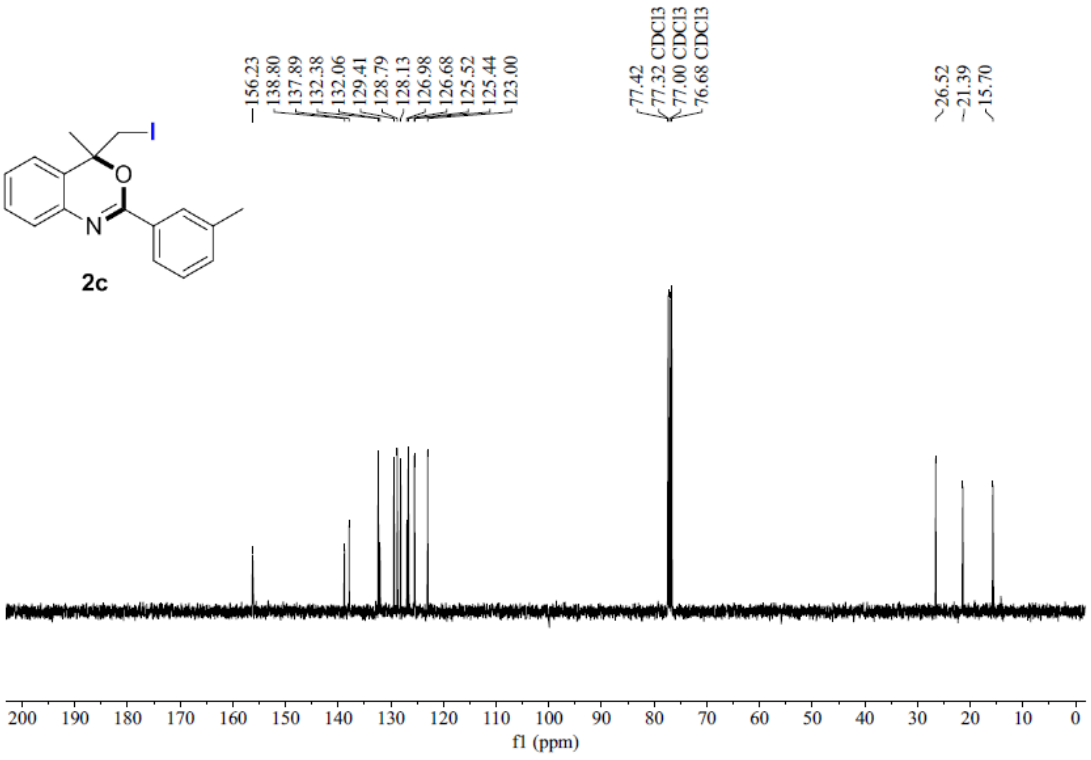
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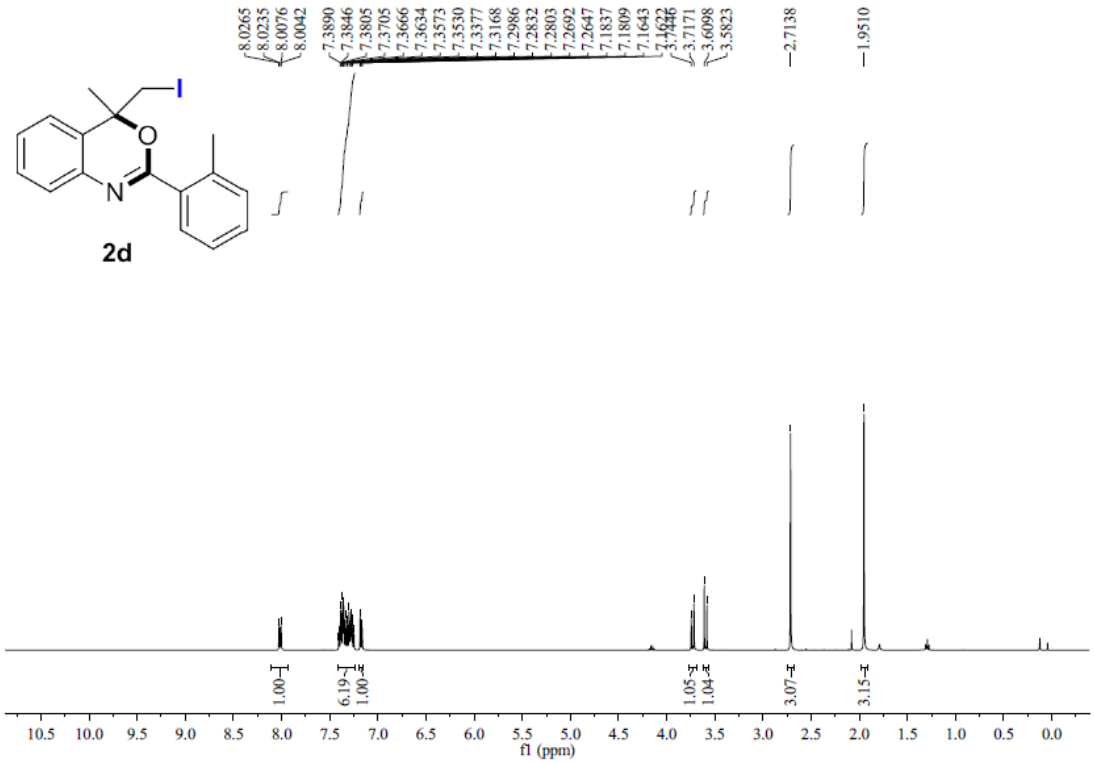
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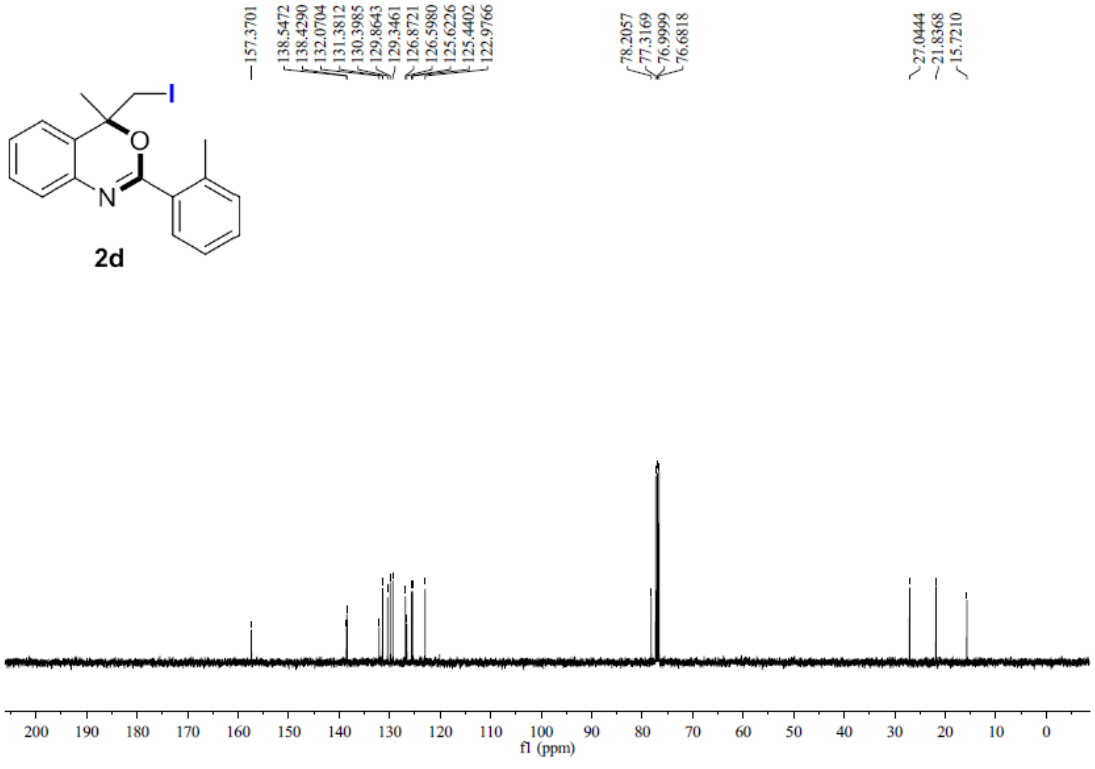
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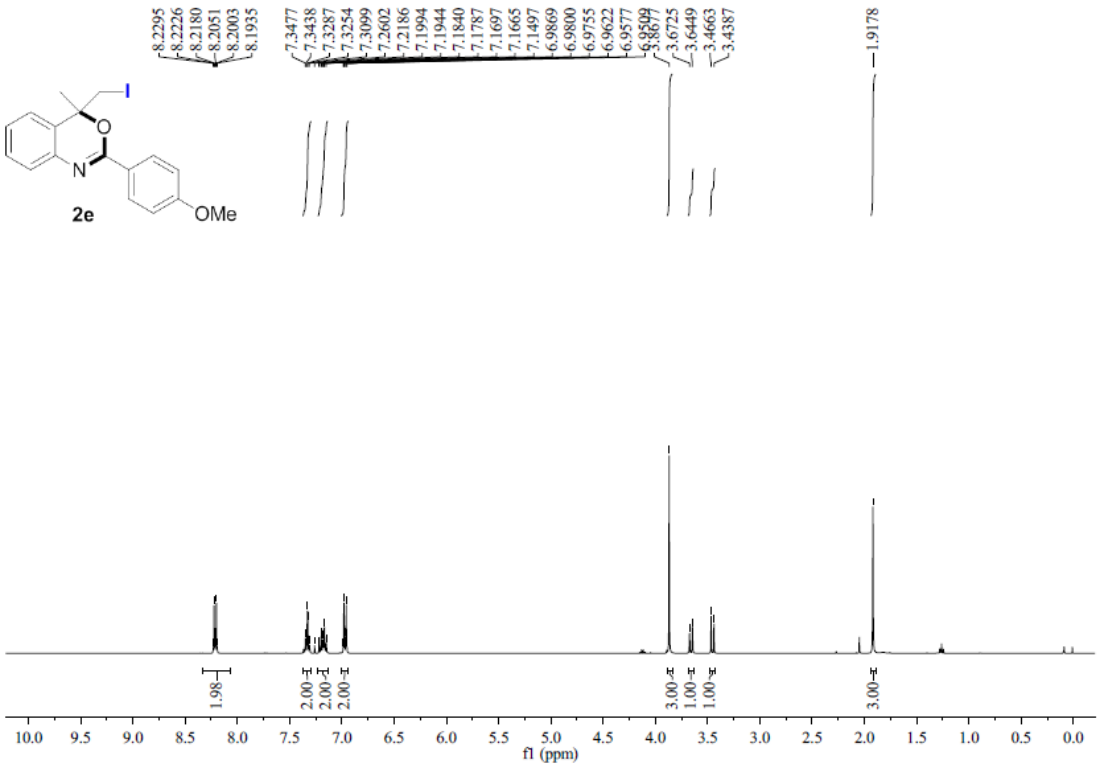
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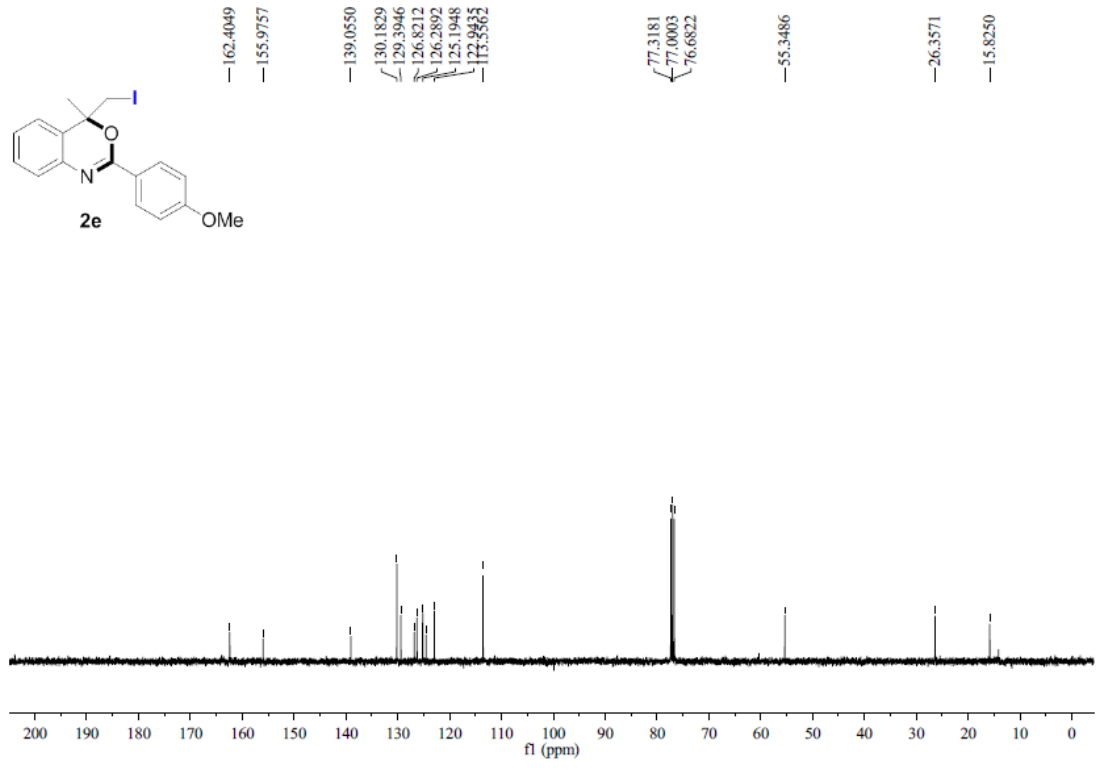
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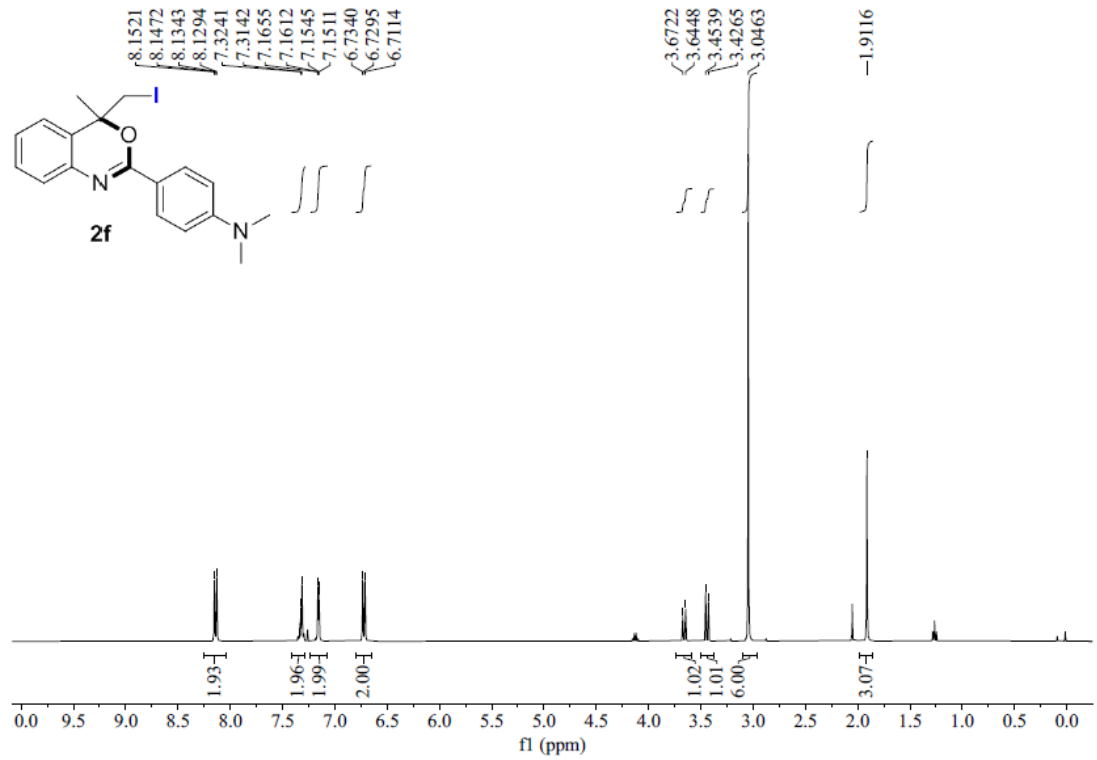
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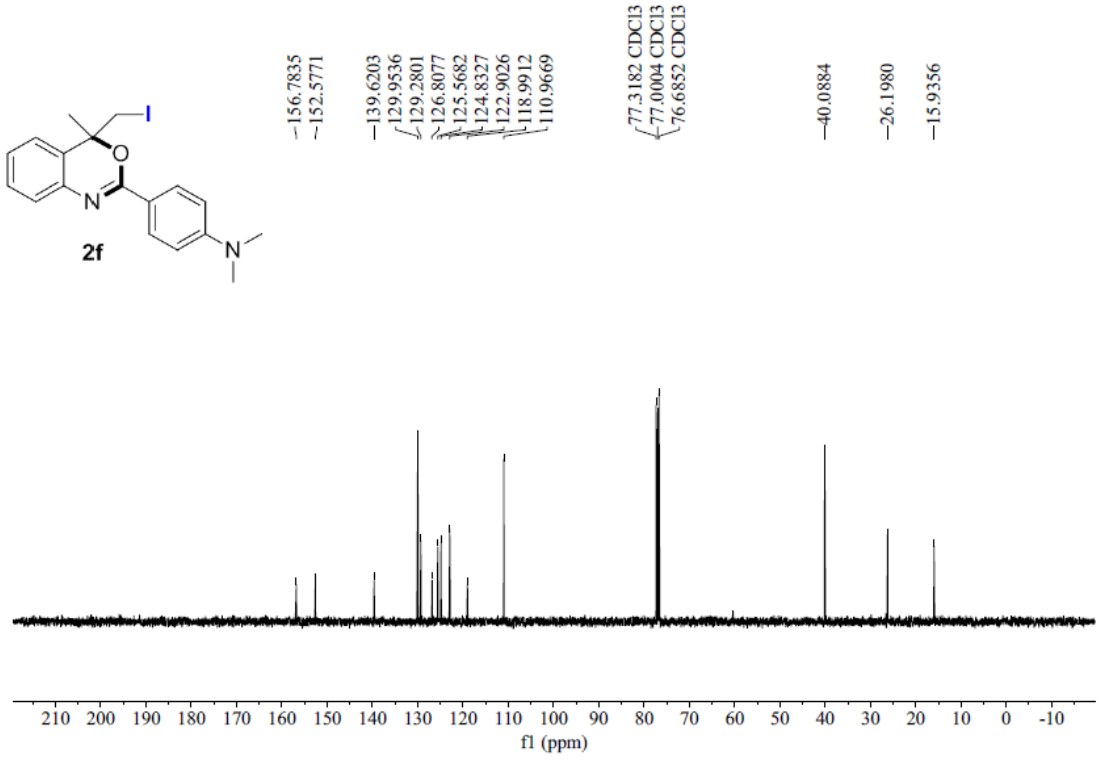
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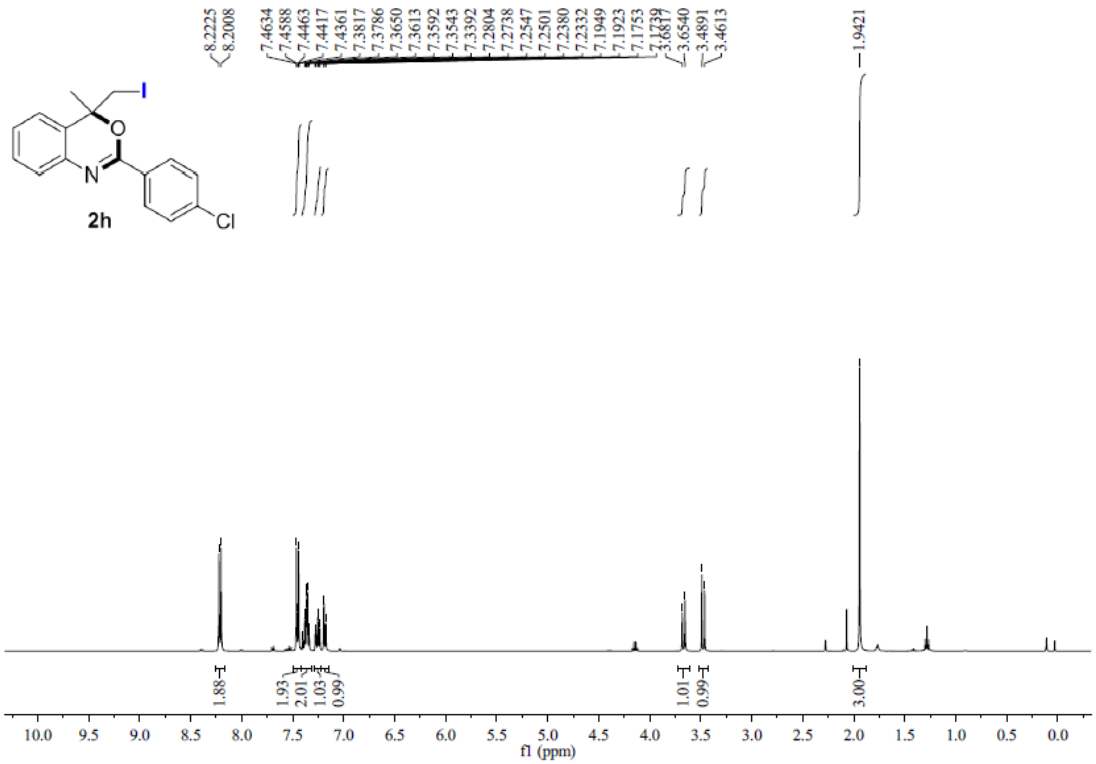
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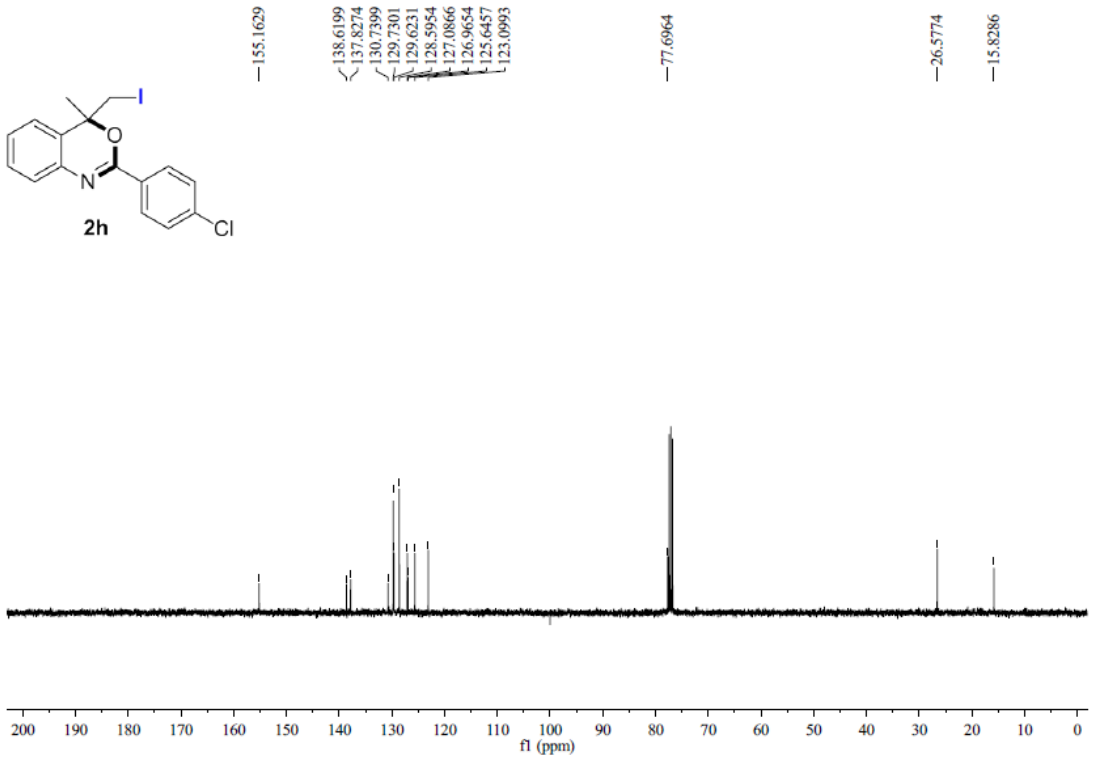
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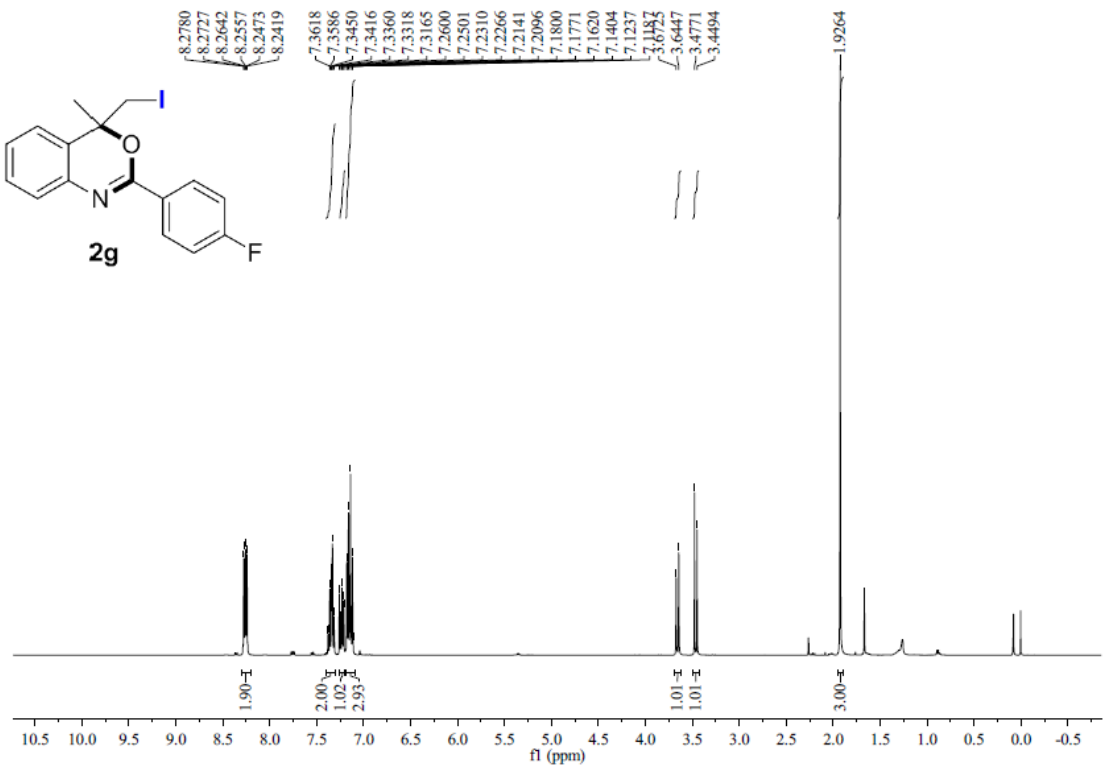
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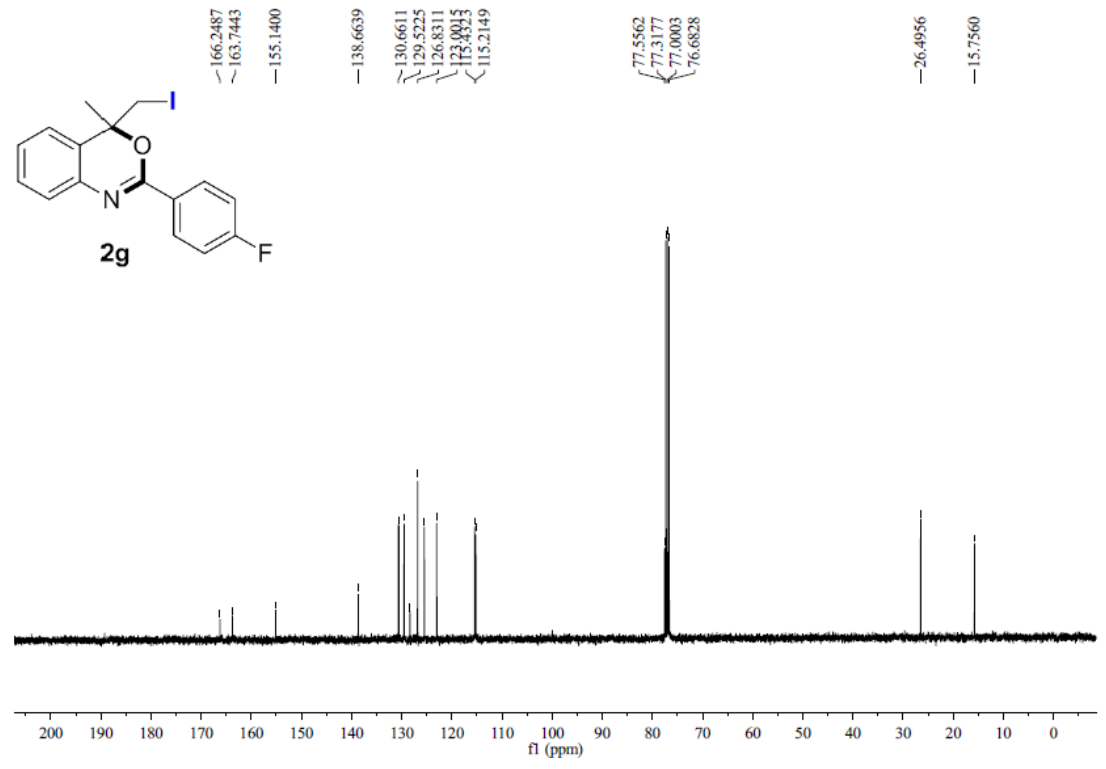
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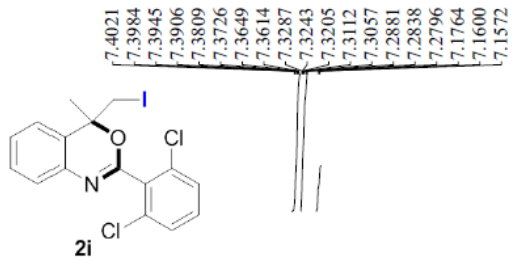
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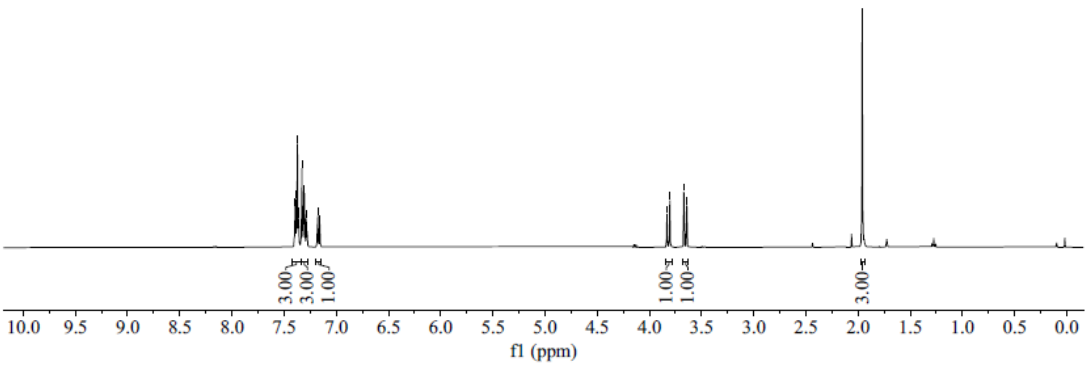
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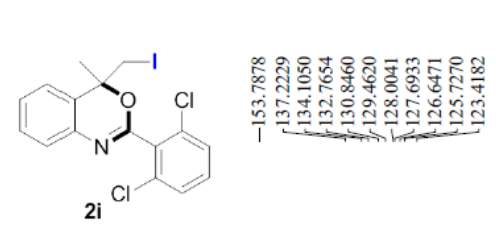
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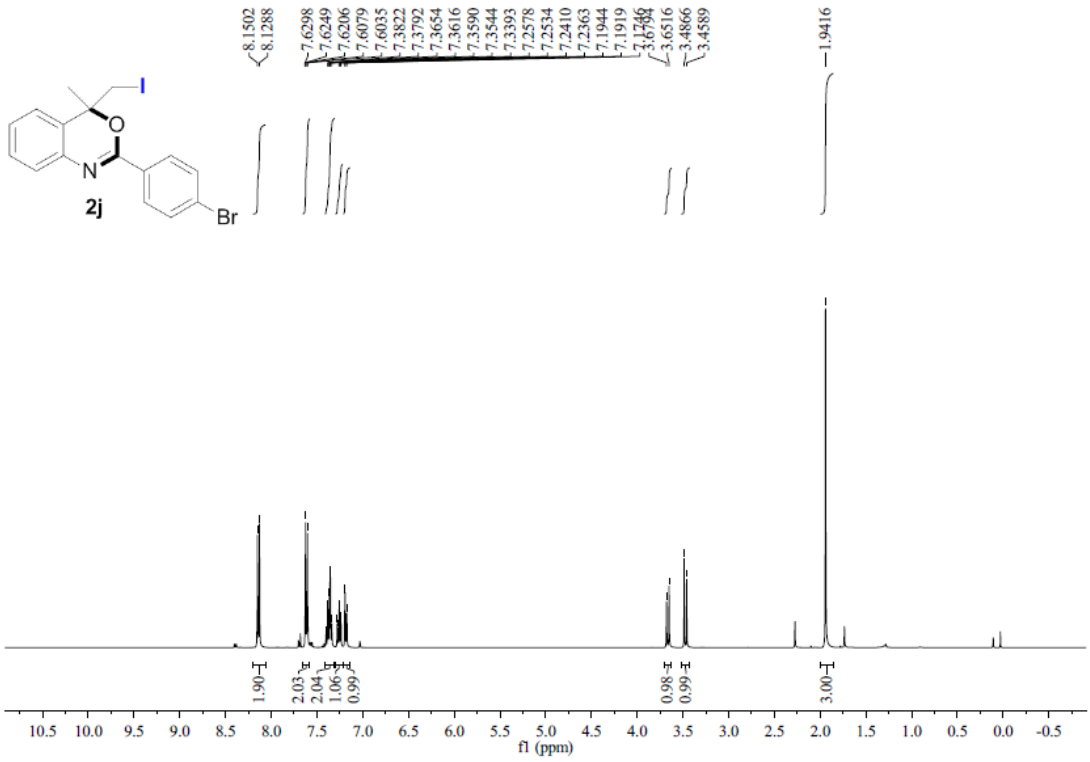
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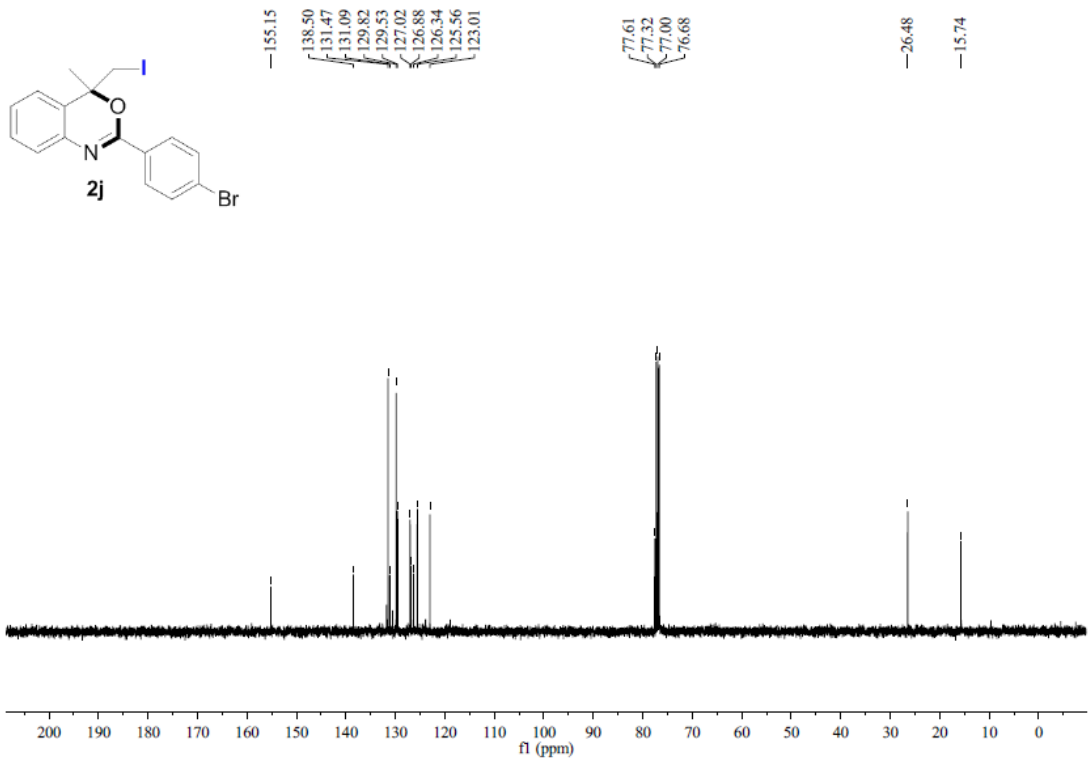
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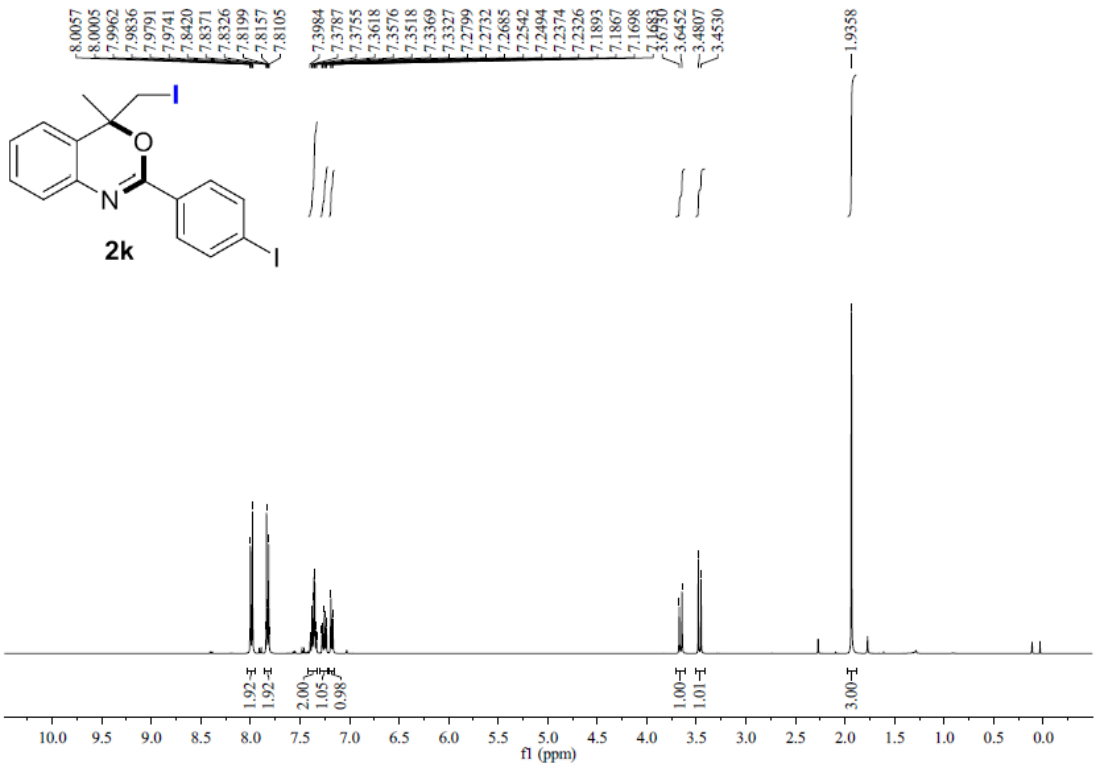
f1 (ppm)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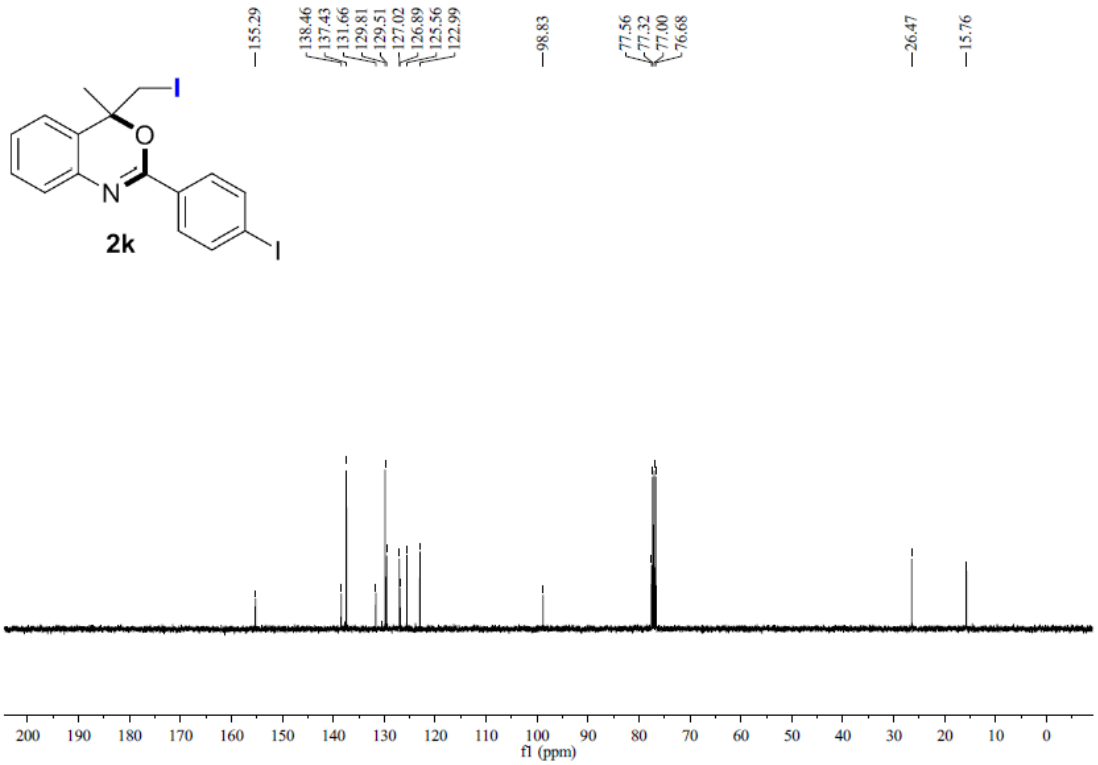
7.8105
7.3984
7.3787
7.3755
7.3618
7.3576
7.3518
7.3369
7.3327
7.2799
7.2732
7.2685
7.2542
7.2494
7.2374
7.2326
7.1893
7.1867
7.1698
7.1683
3.6530
3.6452
3.4807
3.4530
1.9358
I
O
N
2k
I
1.92
1.92
2.00
1.05
0.98
1.00
1.01
3.00
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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137.43
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129.51
127.02
126.89
125.56
122.99
98.83
77.56
77.32
77.00
76.68
26.47
15.76
I
O
N
2k
I
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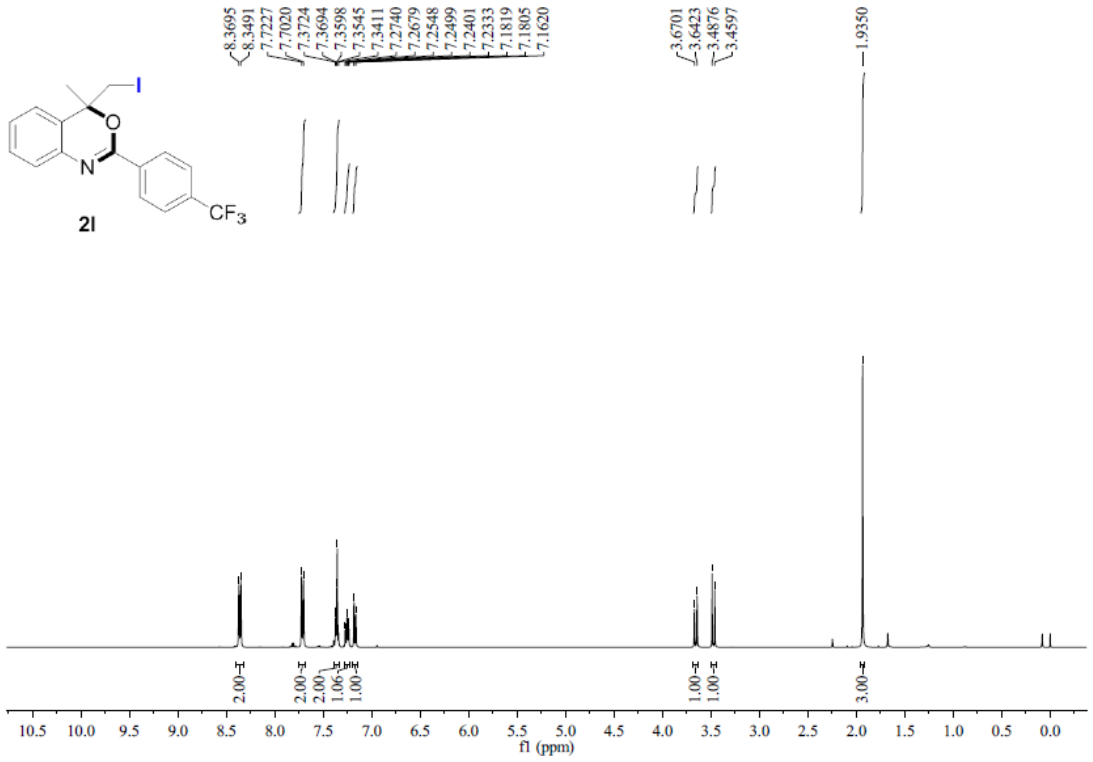
f1 (ppm)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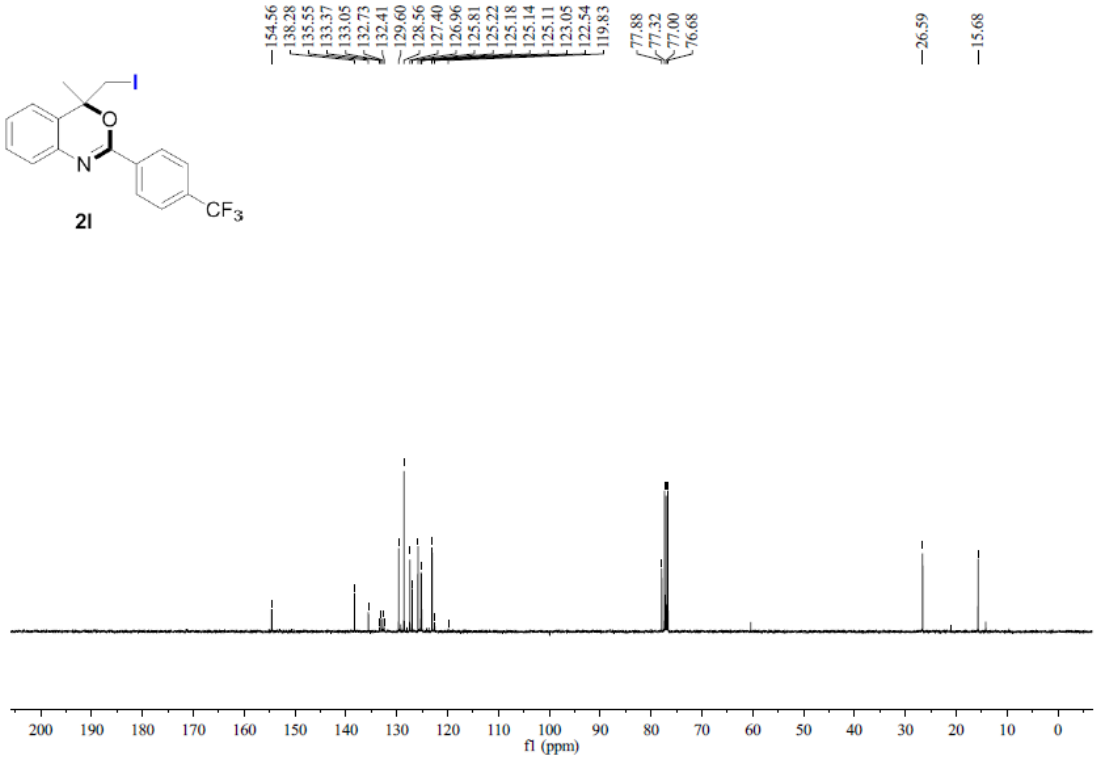
7.2679
7.2548
7.2499
7.2401
7.2333
7.1819
7.1805
7.1620
3.6701
3.6423
3.4876
3.4597
1.9350
I
O
N
CF3
2l
2.00
2.00
2.06
1.00
1.00
1.00
3.00
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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133.05
132.73
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129.60
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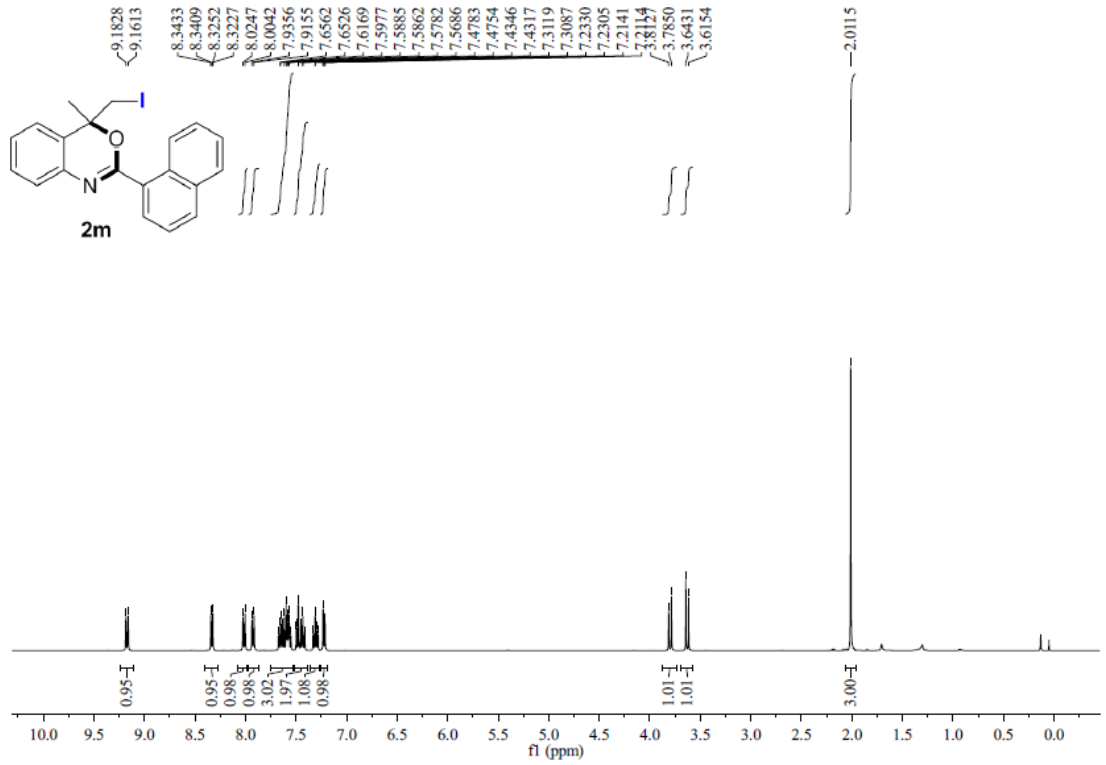
127.40
126.96
125.81
125.22
125.18
125.14
125.11
123.05
122.54
119.83
77.88
77.32
77.00
76.68
26.59
15.68
I
O
N
CF3
2l
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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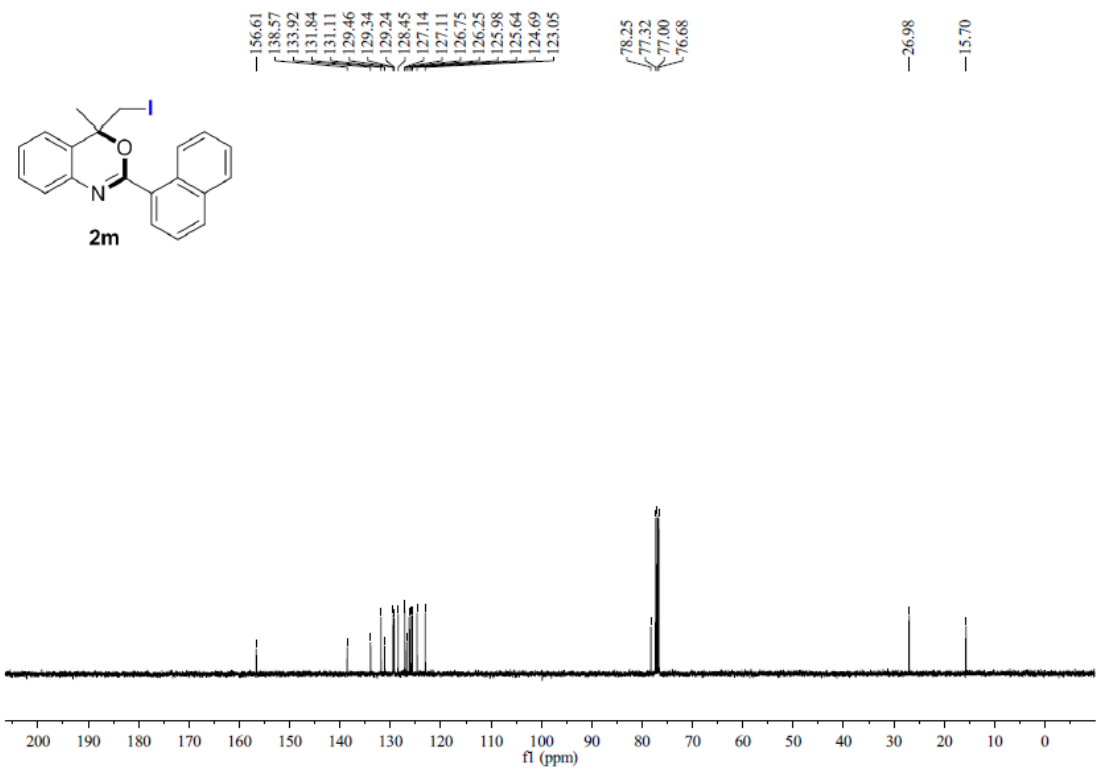
7.6526
7.6169
7.5977
7.5885
7.5862
7.5782
7.5686
7.4783
7.4754
7.4346
7.4317
7.3119
7.3087
7.2330
7.2305
7.2141
7.2114
3.7850
3.6431
3.6154
2.0115
I
O
N
2m
0.95
0.95
0.98
0.98
3.02
1.97
1.08
0.98
1.01
1.01
3.00
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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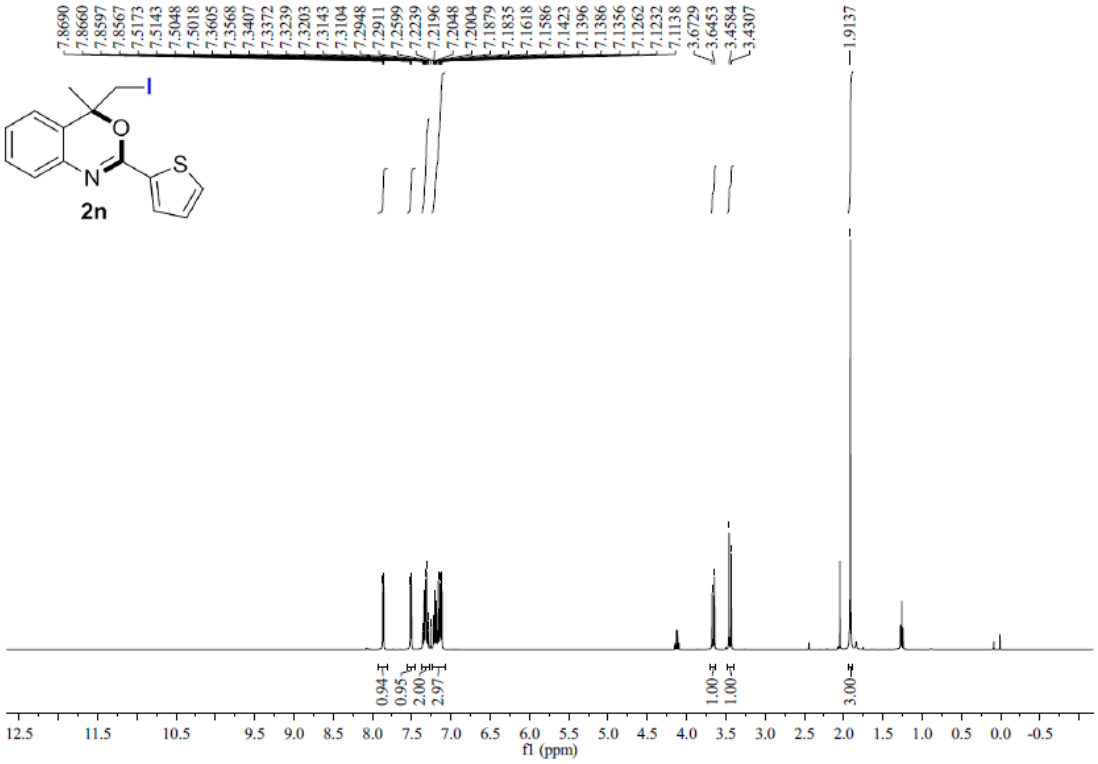
131.84
131.11
129.46
129.34
129.24
128.45
127.14
127.11
126.75
126.25
125.98
125.64
124.69
123.05
78.25
77.32
77.00
76.68
26.98
15.70
I
O
N
2m
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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7.5143
7.5048
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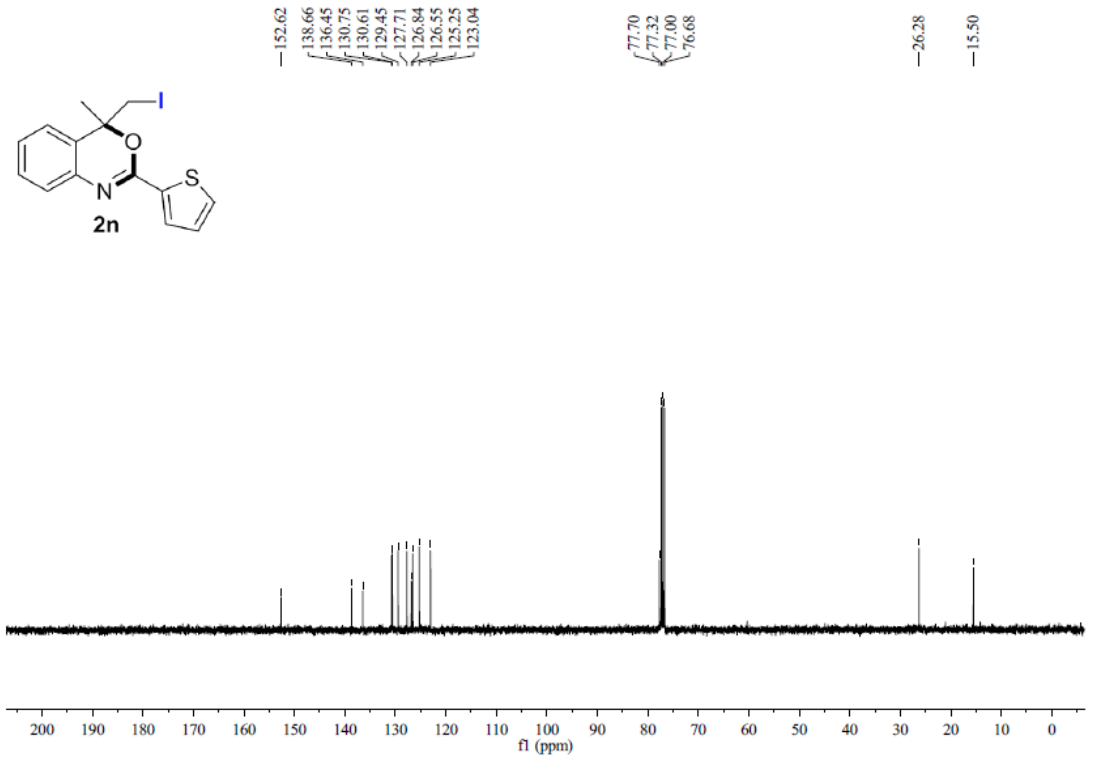
7.3605
7.3568
7.3407
7.3372
7.3239
7.3203
7.3143
7.3104
7.2948
7.2911
7.2599
7.2239
7.2196
7.2048
7.2004
7.1879
7.1835
7.1618
7.1586
7.1423
7.1396
7.1386
7.1356
7.1262
7.1232
7.1138
3.6729
3.6453
3.4584
3.4307
1.9137
I
O
N
S
2n
0.94
0.95
2.00
2.97
1.00
1.00
3.00
12.5 11.5 10.5 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)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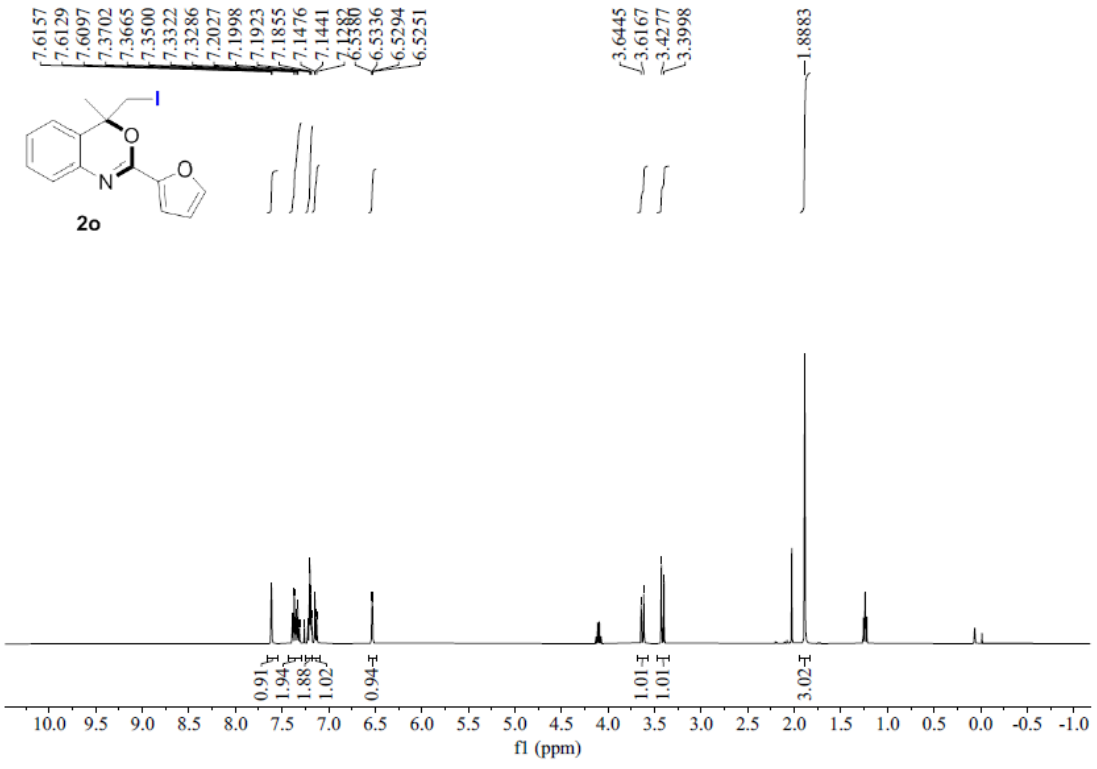
136.45
130.75
130.61
129.45
127.71
126.84
126.55
125.25
123.04
77.70
77.32
77.00
76.68
26.28
15.50
I
O
N
S
2n
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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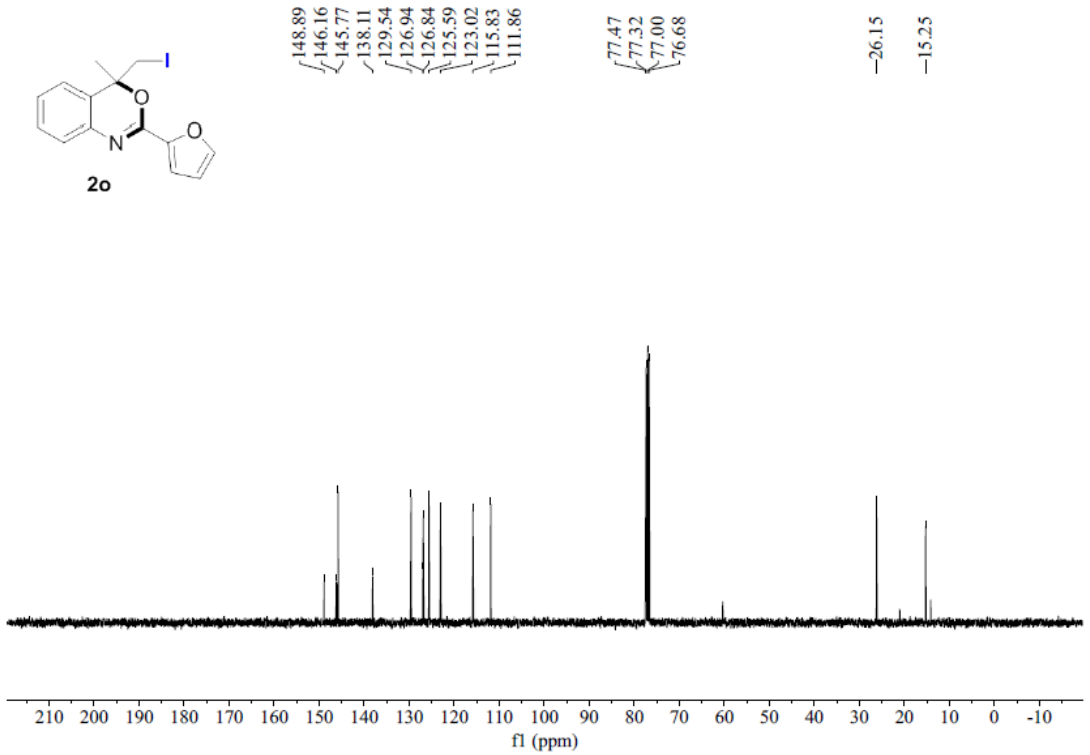
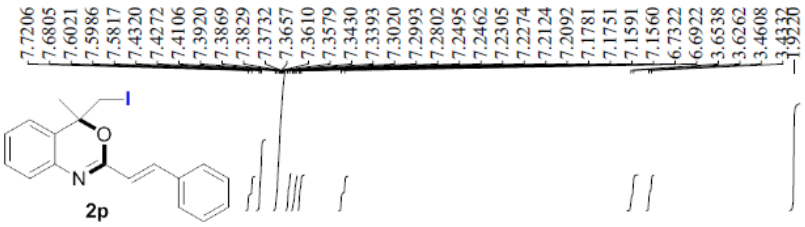
7.6129
7.6097
7.3702
7.3665
7.3500
7.3322
7.3286
7.2027
7.1998
7.1923
7.1855
7.1476
7.1441
7.1282
6.5336
6.5294
6.5251
3.6445
3.6167
3.4277
3.3998
1.8883
I
O
N
O
2o
0.91
1.94
1.88
1.02
0.94
1.01
1.01
3.02
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)


148.89
146.16
145.77
138.11
129.54
126.94
126.84
125.59
123.02
115.83
111.86
77.47
77.32
77.00
76.68
26.15
15.25
I
O
N
O
2o
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)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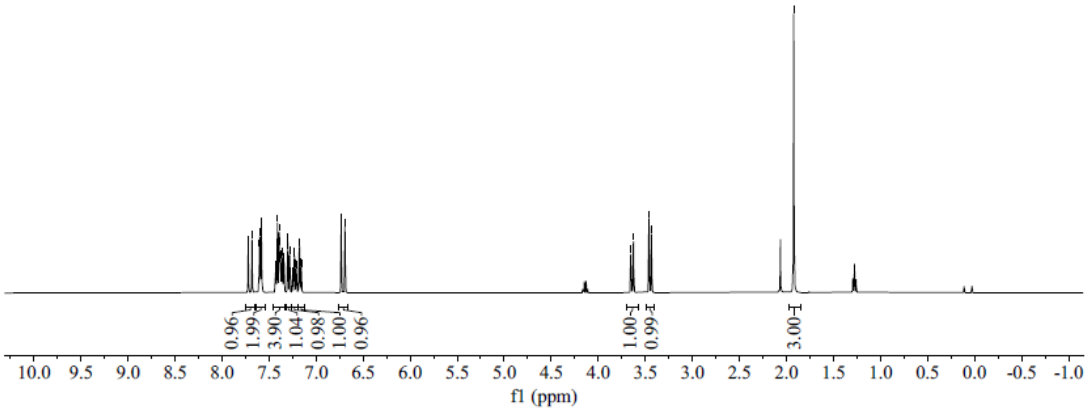
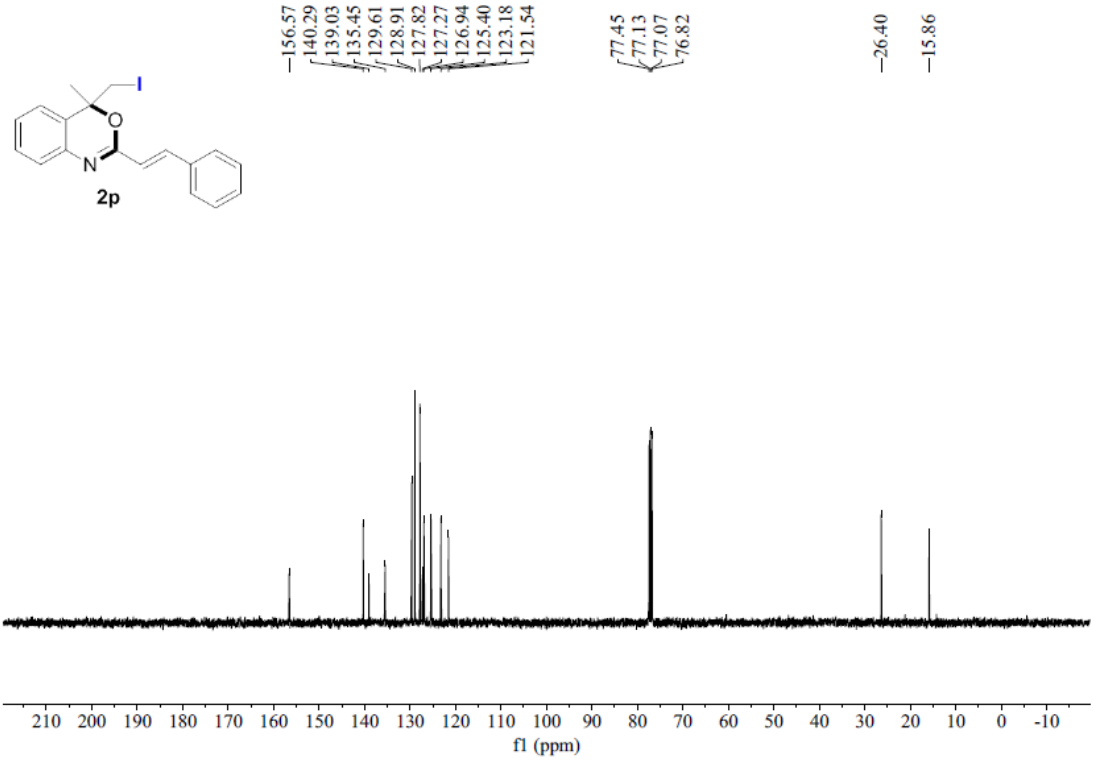
7.6021
7.5986
7.5817
7.4320
7.4272
7.4106
7.3920
7.3869
7.3829
7.3732
7.3657
7.3610
7.3579
7.3430
7.3393
7.3020
7.2993
7.2802
7.2495
7.2462
7.2305
7.2274
7.2124
7.2092
7.1781
7.1751
7.1591
7.1560
6.7322
6.6922
3.6538
3.6262
3.4608
3.4332
1.9220
I
O
N
2p


0.96
1.99
3.90
1.04
0.98
1.00
0.96
1.00
0.99
3.00
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)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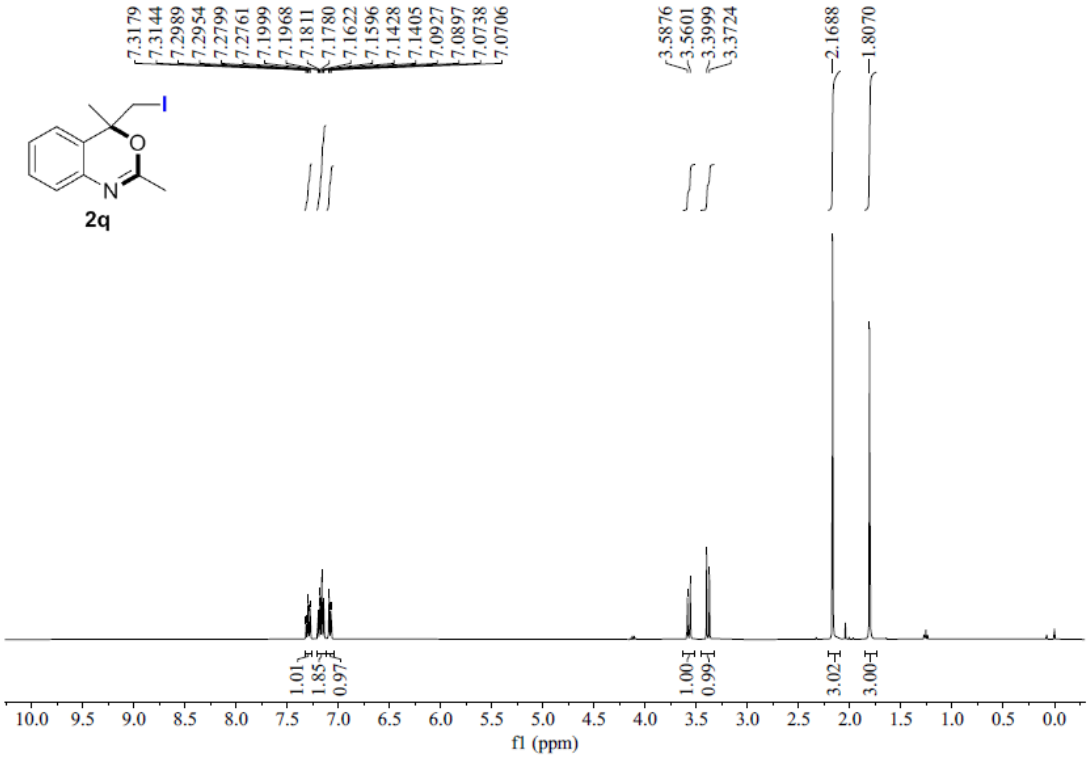
135.45
129.61
128.91
127.82
127.27
126.94
125.40
123.18
121.54
77.45
77.13
77.07
76.82
26.40
15.86
I
O
N
2p
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)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7.1999
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7.1811
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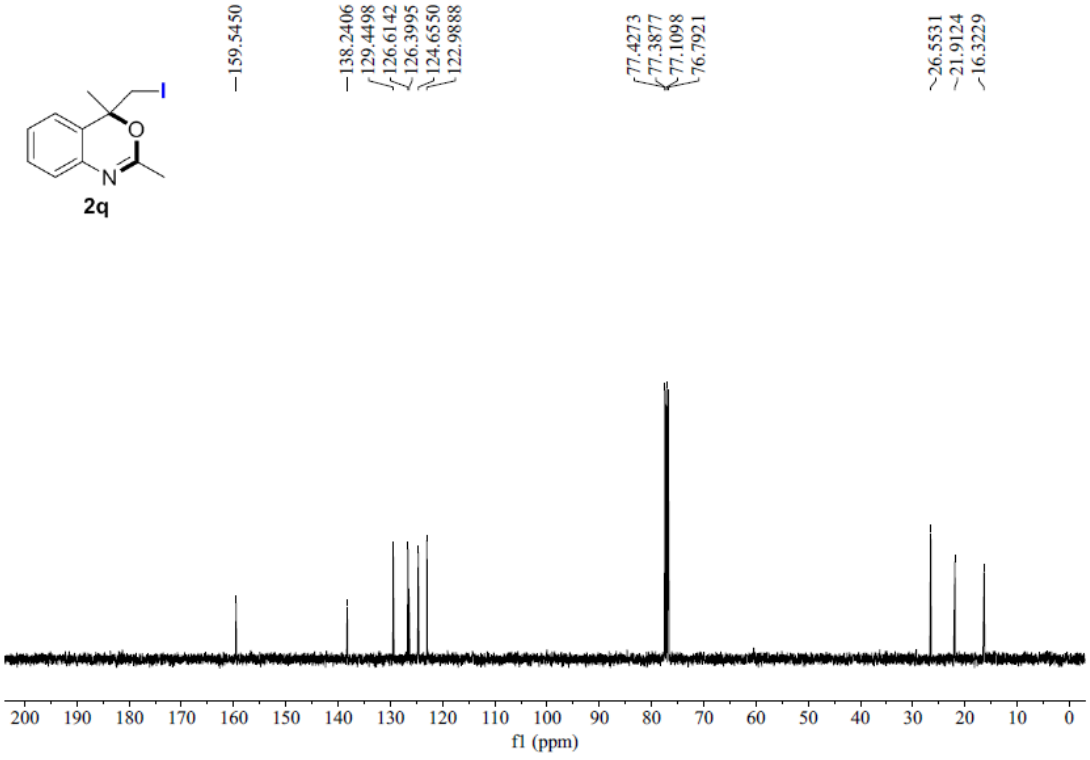
7.1622
7.1596
7.1428
7.1405
7.0927
7.0897
7.0738
7.0706
3.5876
3.5601
3.3999
3.3724
2.1688
1.8070
I
O
N
2q
1.01
1.85
0.97
1.00
0.99
3.02
3.00
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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126.3995
124.6550
122.9888
77.4273
77.3877
77.1098
76.7921
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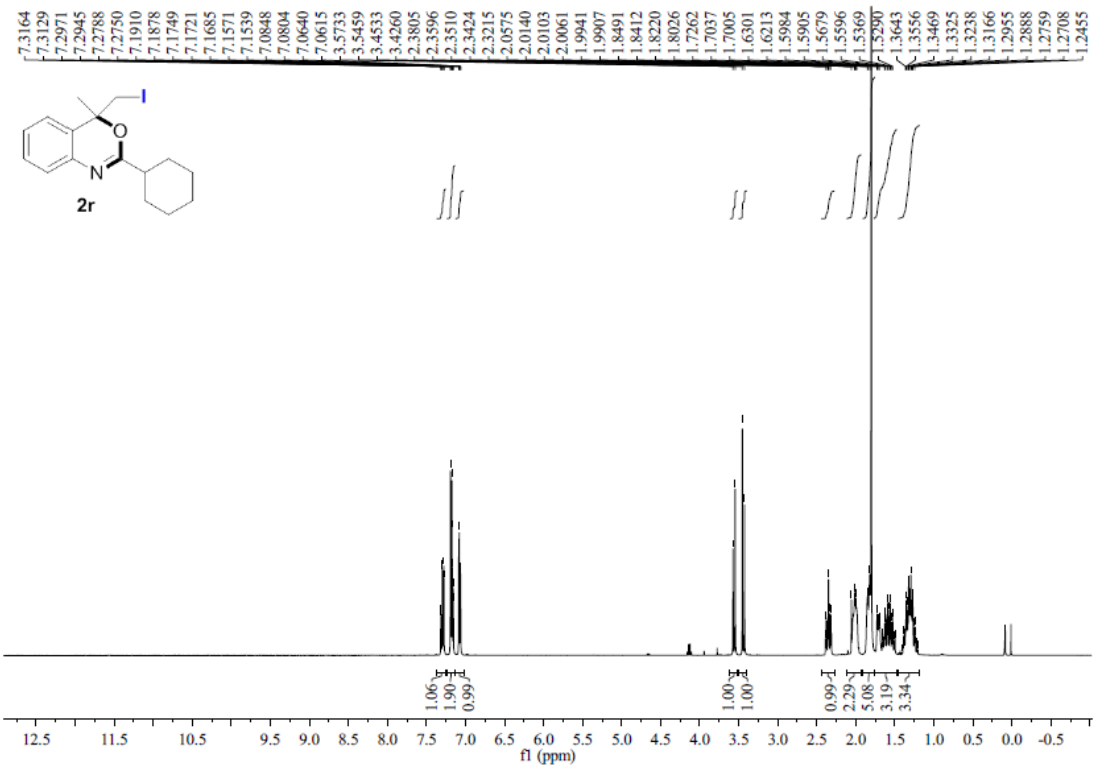
21.9124
16.3229
I
O
N
2q
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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7.1685
7.1571
7.1539
7.0848
7.0804
7.0640
7.0615
3.5733
3.5459
3.4533
3.4260
2.3805
2.3596
2.3510
2.3424
2.3215
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2.0140
2.0103
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1.9941
1.9907
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1.8412
1.8220
1.8026
1.7262
1.7037
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1.6301
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1.5905
1.5679
1.5596
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1.5290
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1.3166
1.2955
1.2888
1.2759
1.2708
1.2455
I
O
N
2r
1.06
1.90
0.99
1.00
1.00
0.99
2.29
5.08
3.19
3.34
12.5
11.5
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
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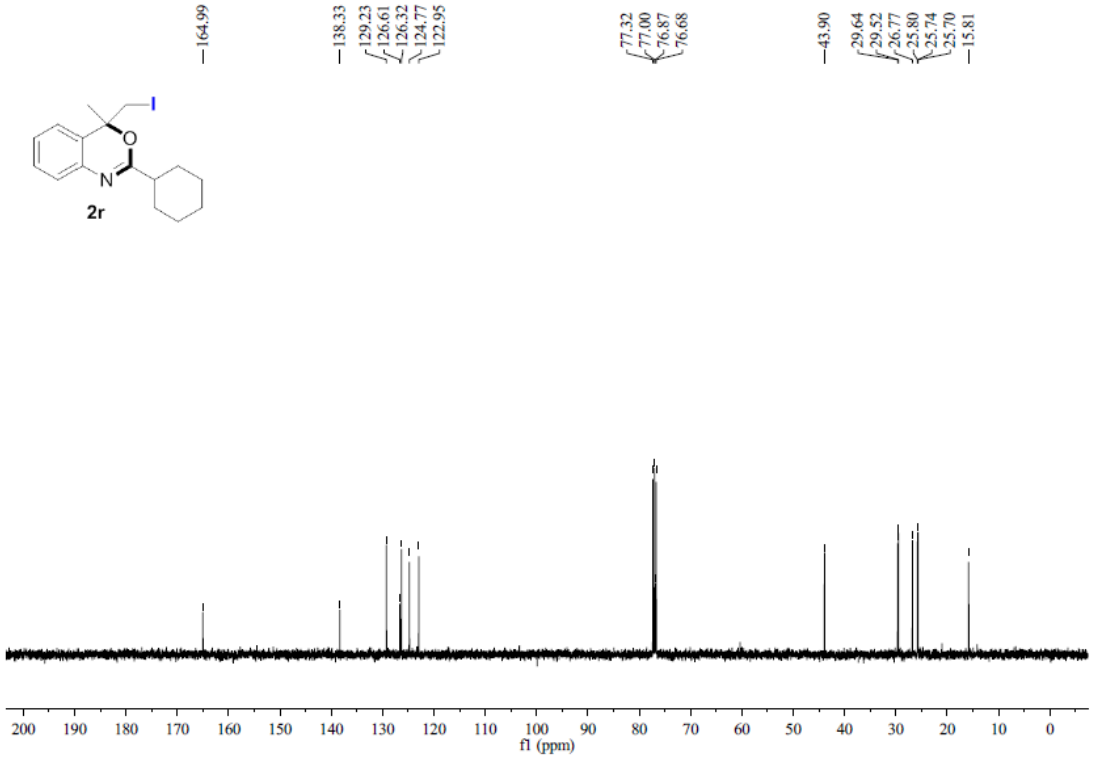
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)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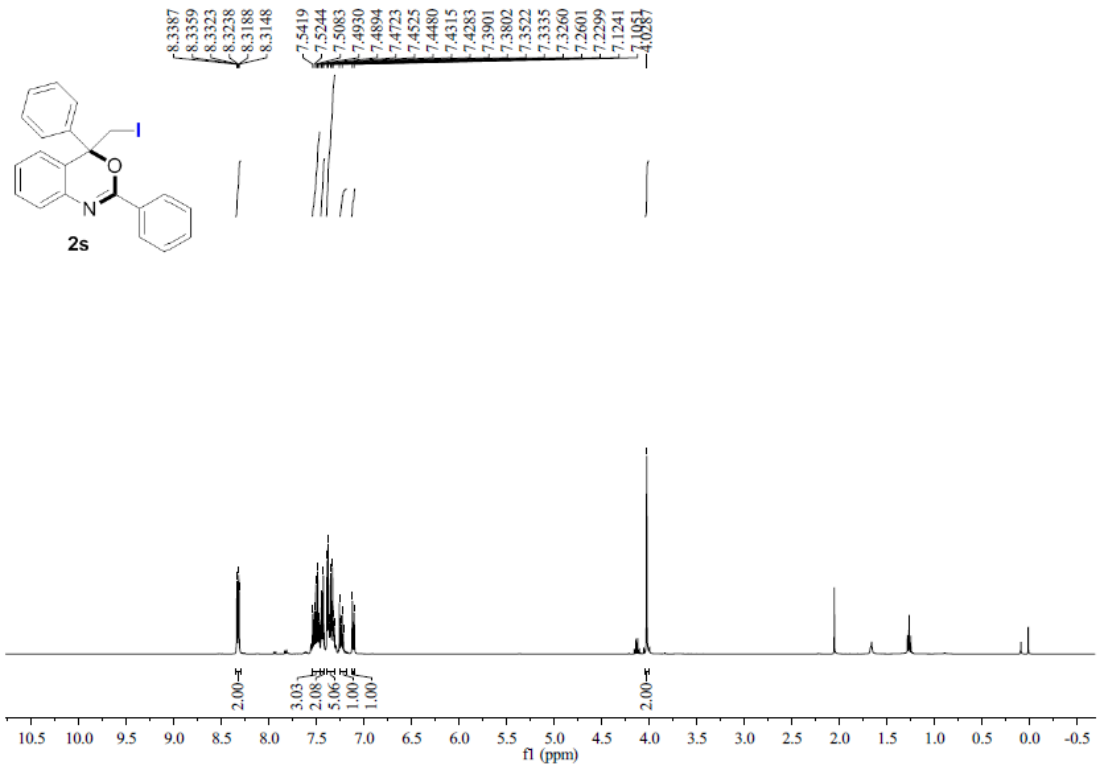
124.77
122.95
77.32
77.00
76.87
76.68
43.90
29.64
29.52
26.77
25.80
25.74
25.70
15.81
I
O
N
2r
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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7.4723
7.4525
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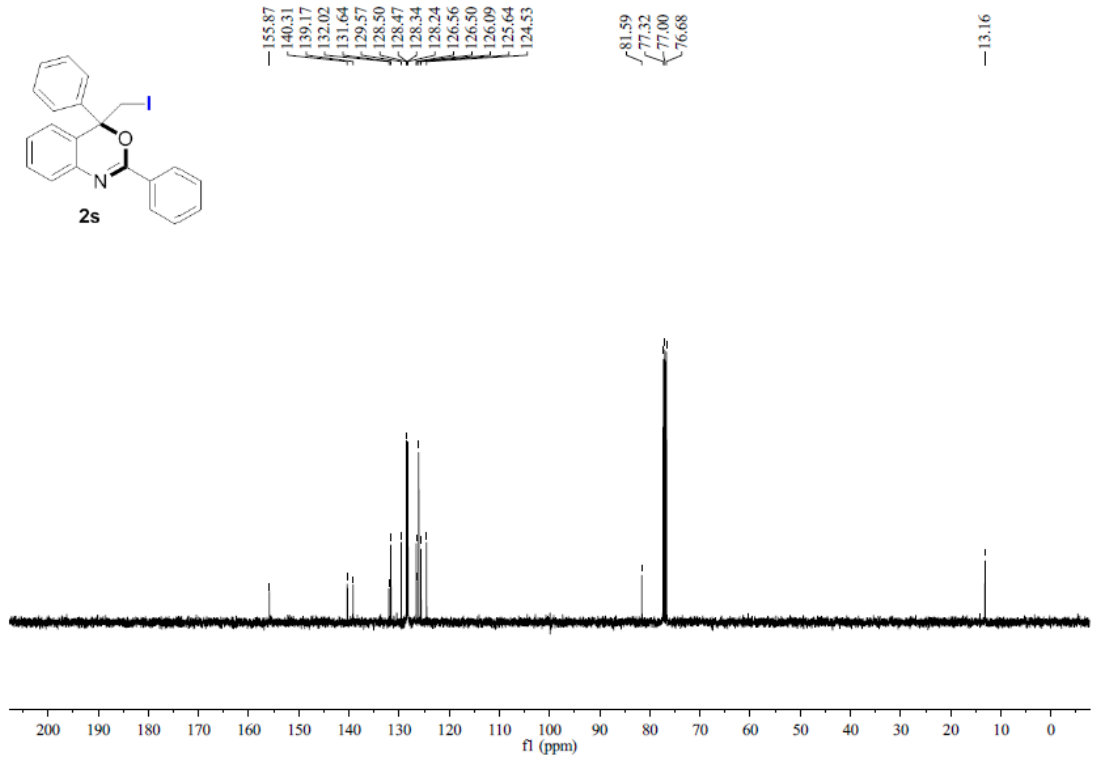
7.4315
7.4283
7.3901
7.3802
7.3522
7.3335
7.3260
7.2601
7.2299
7.1241
7.1051
4.0287
I
O
N
2s
2.00
3.03
2.08
5.06
1.00
1.00
2.00
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)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128.47
128.34
128.24
126.56
126.50
126.09
125.64
124.53
81.59
77.32
77.00
76.68
13.16
I
O
N
2s
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)