Supporting Information

Metal-Free Intermolecular Oxidative C-N Bond Formation via Tandem C-H / N-H Bond Activation

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Experimental Section

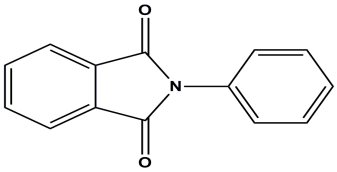
Reagents

Substrates including phthalimide, 4-nitrophthalimide, 3,4,5,6-tetrachlorophthalimide, napthalimide, saccharin, succinimde, potassium phthalimdie, 2-pyrrolidinone, benzene, *p*-xylene, *o*-xylene, *m*-xylene, toluene, 1,4-difluorobenzene, 1,2-dichlorobenzene, 4-methylanisole, pentamethylbenzene, pentalfluorobenzene were purchased from Sigma Aldrich and Fisher Scientific. Iodobenzene diacetate was purchased from Acros Chemicals. Flash chromatography was performed on Silicycle silica gel (60Å, 40-63 μm). All reagents were stored under an inert atmosphere before use.

Instrumentation

Reactions were carried out in a CEM Discover microwave. GC/MS analysis was carried out on an Agilent Technologies 6890 GC system fixed with a 5973 mass selective detector. NMR spectra were acquired using a Bruker Avance 300MHz spectrometer.

Synthesis of 2-phenylisoindoline-1, 3-Dione (2)



2

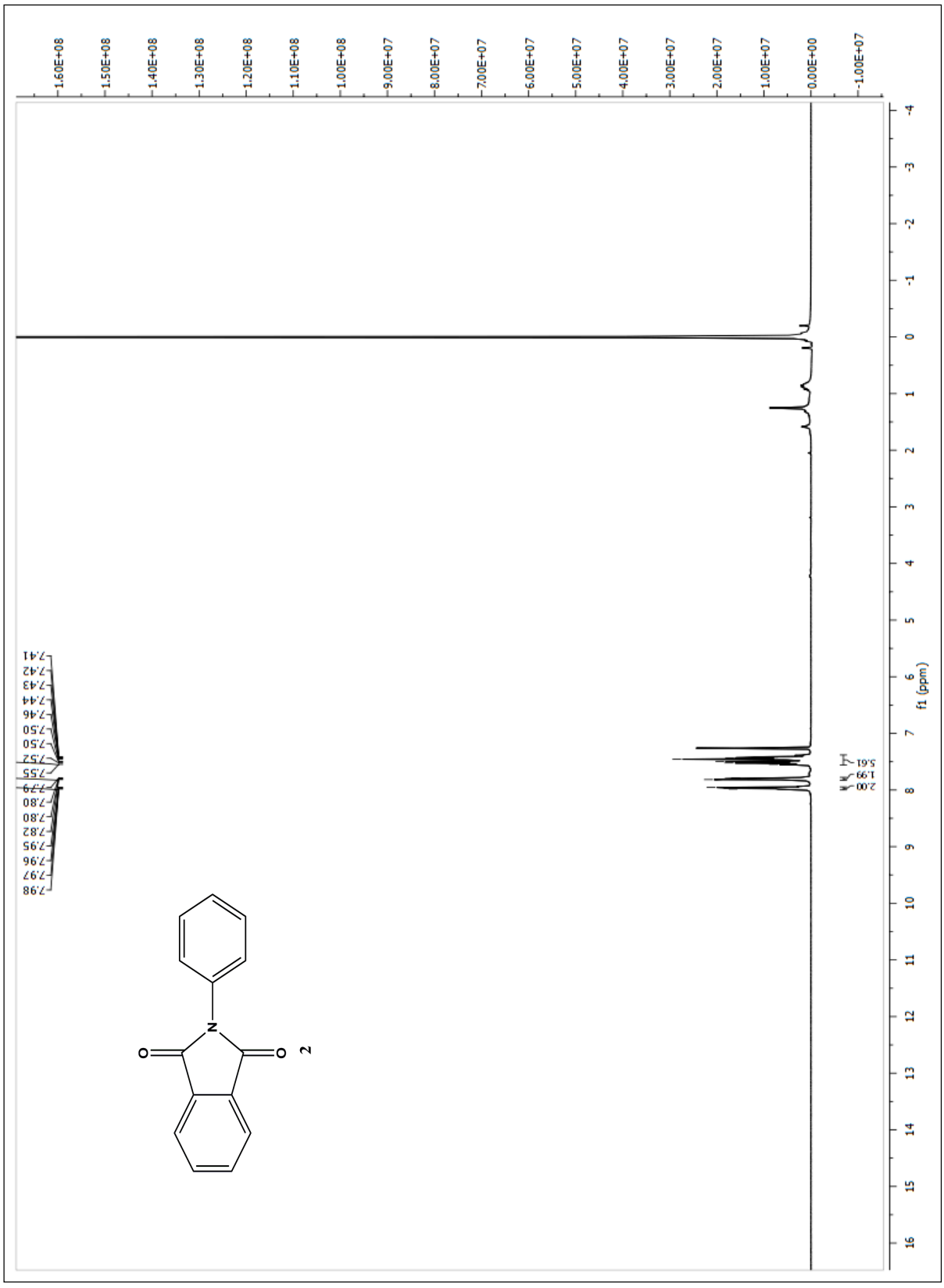
A magnetically stirred solution of phthalimide (0.10 g, 0.68mmol), iodobenzene diacetate (0.55 g, 1.7mmol) in 4 mL of benzene was microwave heated at 145 ^{0}C for 3 h. The excess solvent from the mixture is removed at reduced pressure and the crude product was purified by column chromatography to give pure **2** (0.133 g, 88 %). The NMR spectra matched with that of previously published.[1]

***R_f* -Value:** Hexane/Ethyl acetate (8:2 *v/v*) = 0.31.

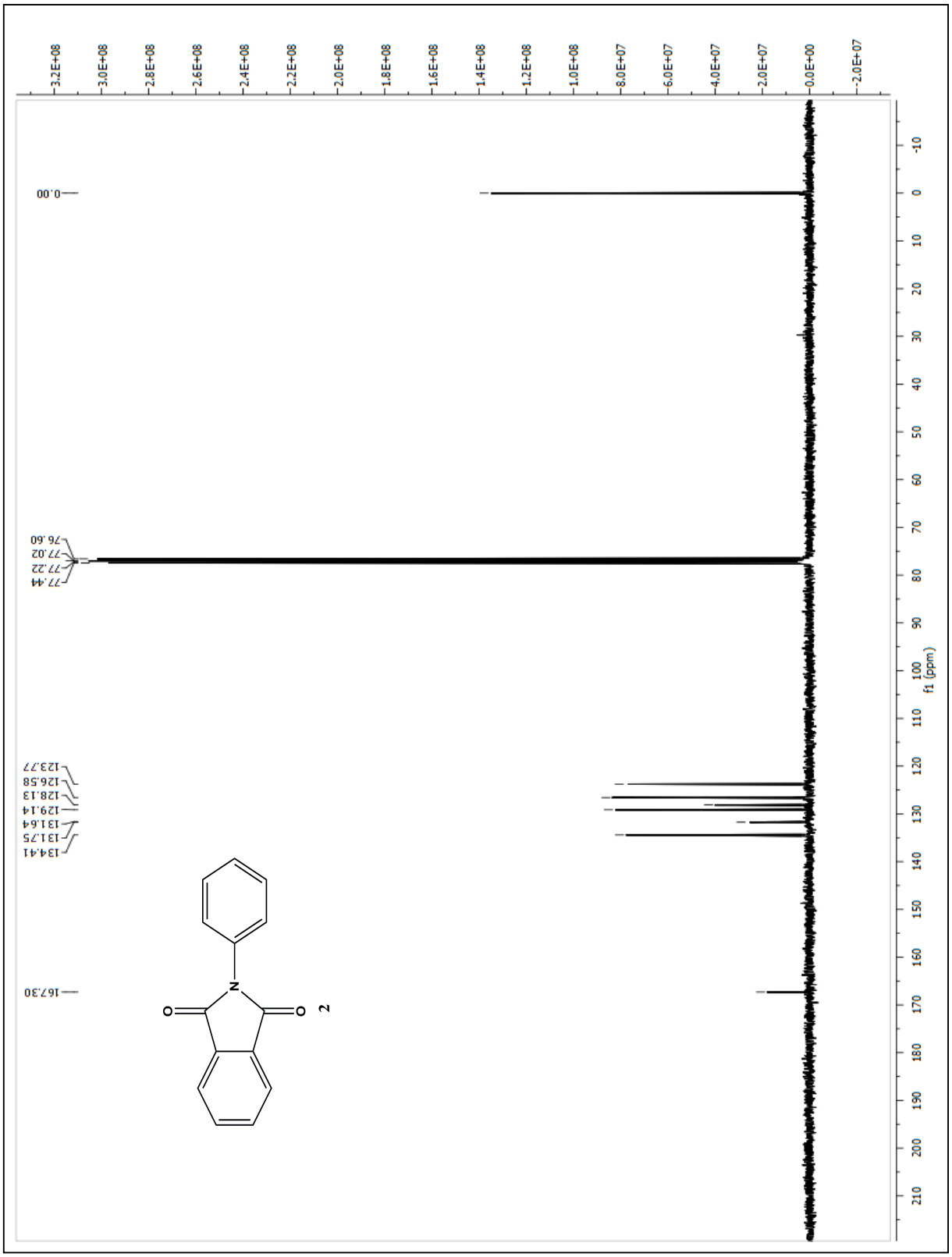
^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.39-7.53 (m, 5H), 7.80 (dd, *J* = 5.4 Hz, 2.8 Hz, 2H), 7.96 (dd, *J* = 5.6 Hz, 3.2 Hz, 2H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 123.7, 126.5, 128.1, 129.1, 131.7, 134.4, 167.30.

LRMS EI (m/z): [M+] calc'd for $C_{14}H_9NO_2$ 223.06, observed 223.10 m/z.

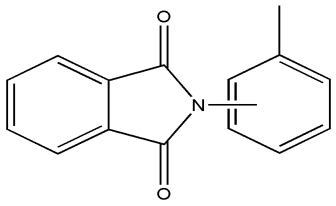


Spectra 1. ^{1}H NMR of Compound **2**



Spectra 2. ^{13}C NMR of Compound **2**

Synthesis of 3



3
o:m:p= 10 : 6 : 5

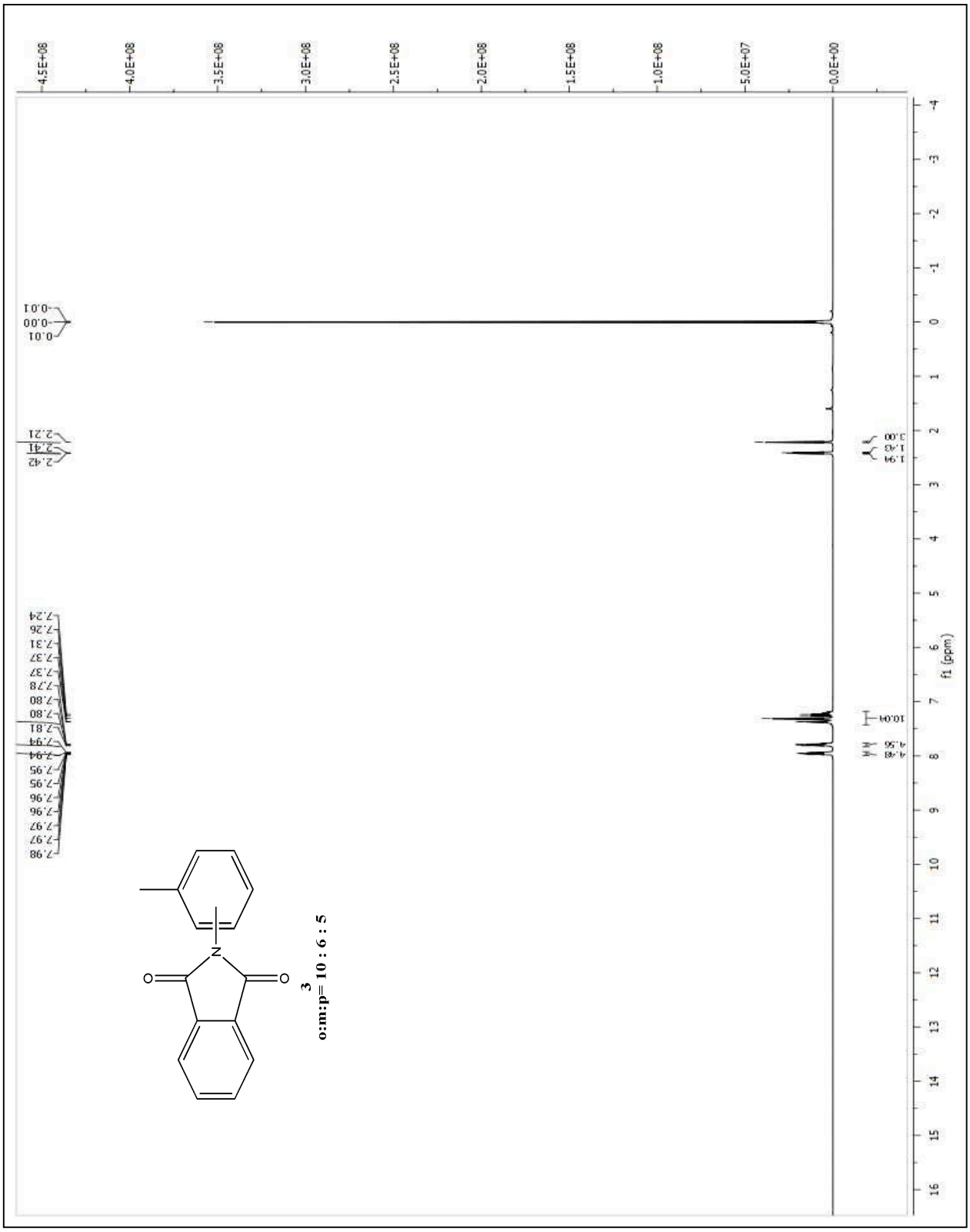
A magnetically stirred solution of phthalimide (0.10 g, 0.68mmol), iodobenzene diacetate (0.55 g, 1.7mmol) in 4 mL of toluene was microwave heated at 145 ^{0}C for 3 h. The excess solvent from the mixture is removed at reduced pressure and crude product was purified by column chromatography to give pure **3** (0.1071 g, 70 %, o: m: p = **10**:**6**:**5**). The isomers were identified by comparing with known NMR spectra.[2]

***R_f* -Value:** Hexane/Ethyl acetate (9:1 *v/v*) = 0.2.

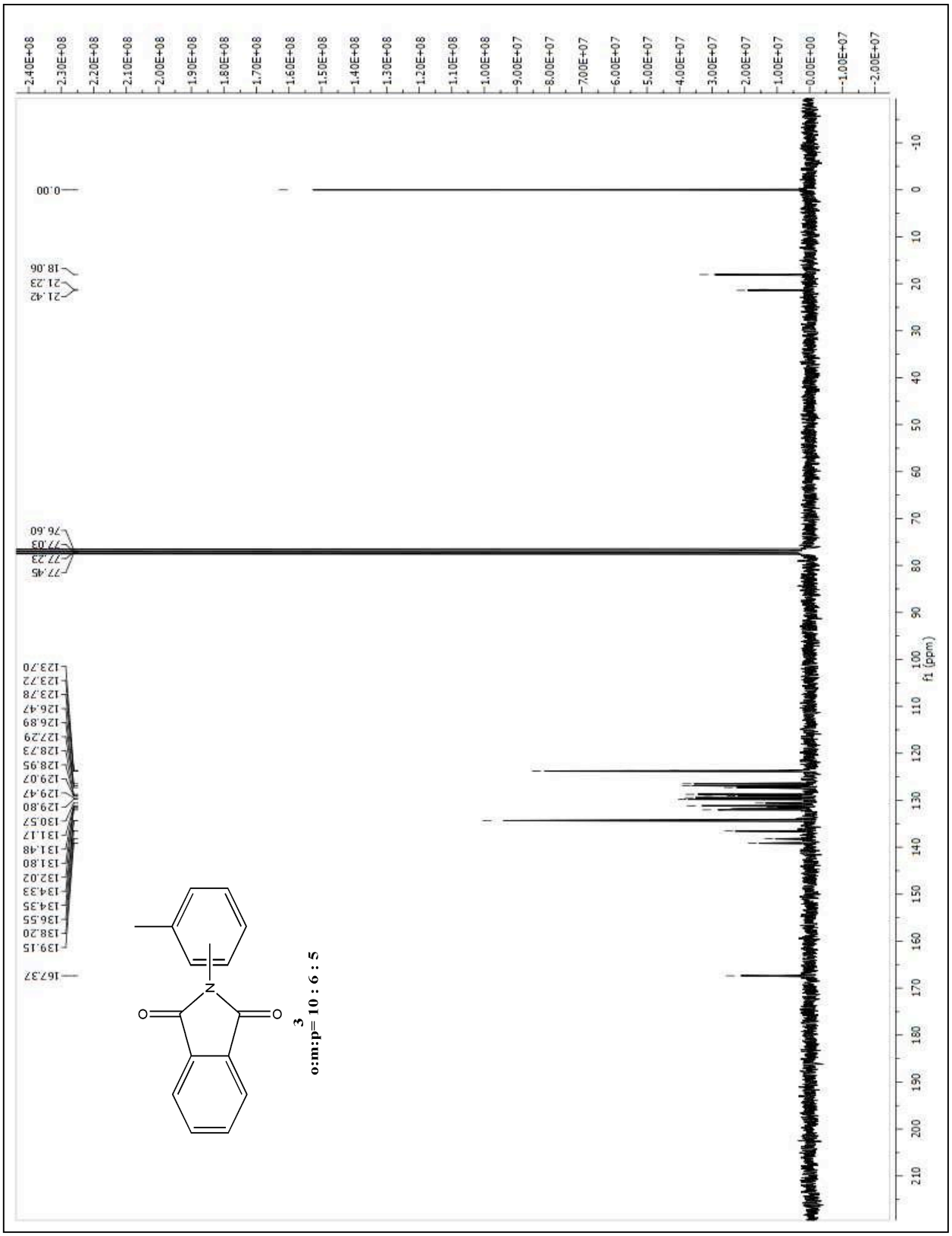
^{1}H NMR (300 MHz, $CDCl_3$): δ = 2.21 (s, 3H), 2.41 (s, 1H), 2.42 (s, 2H), 7.18 – 7.43 (m, 10H), 7.80 (dd, *J* = 5.4, 3.1 Hz, 5H), 7.92 – 7.99 (m, 4H).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 18.06, 21.23, 21.42, 123.70, 123.72, 123.78, 126.47, 126.89, 127.29, 128.73, 128.95, 129.07, 129.47, 129.80, 130.57, 131.17, 131.48, 131.80, 132.02, 134.33, 134.35, 136.55, 138.20, 139.15, 167.37.

LRMS EI (m/z): [M+] calc'd for $C_{14}H_9NO_2$ 237.08, observed 237.10 m/z.

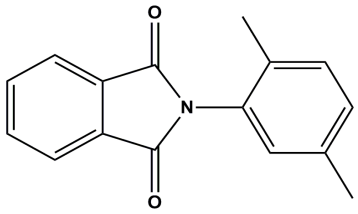


Spectra 3. ^{1}H NMR of Compound **3**



Spectra 4. ^{13}C NMR of Compound **3**

Synthesis of 2-(2, 5-dimethylphenyl) isoindoline-1, 3-dione (5)



5

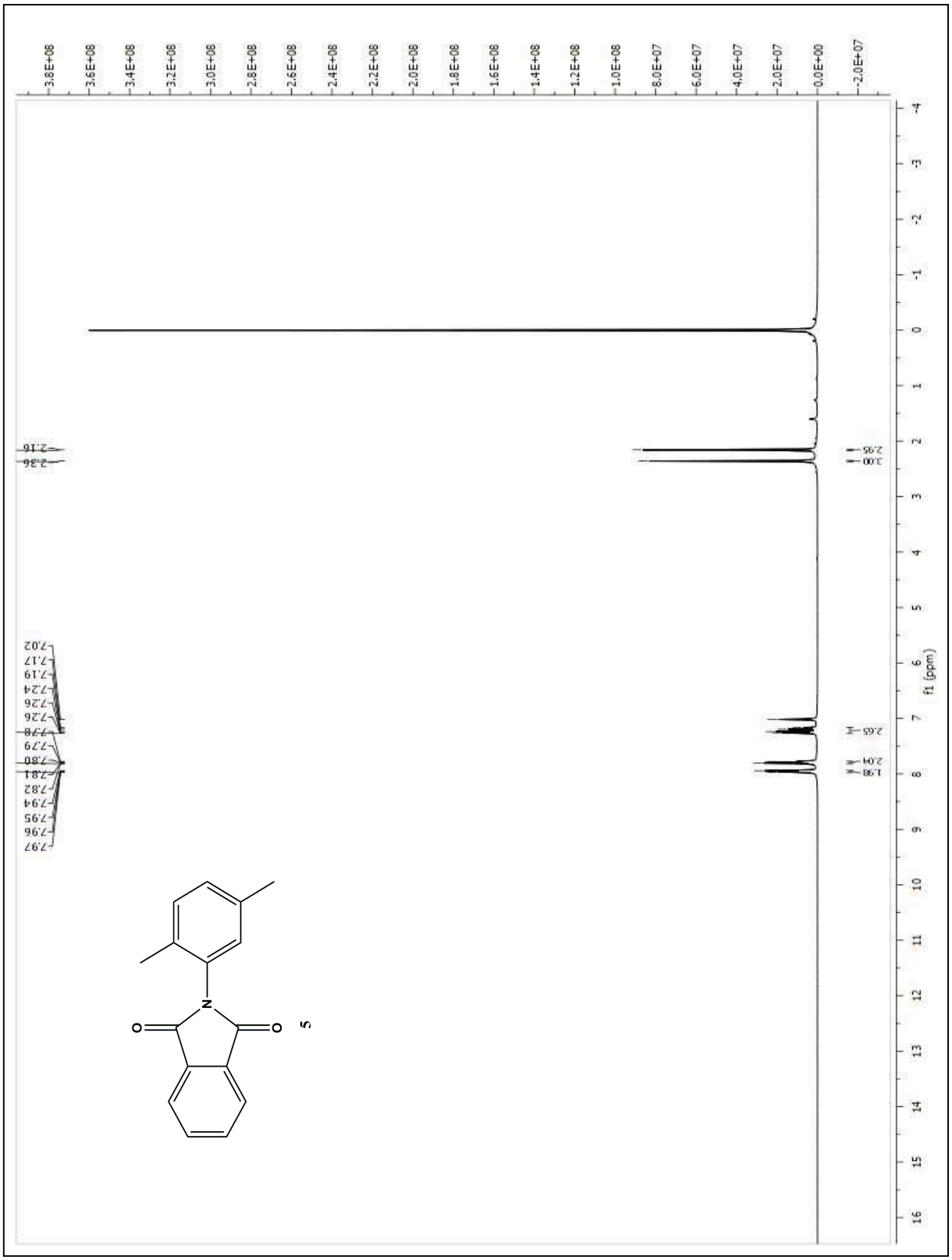
A magnetically stirred solution of Phthalimide (0.10 g, 0.68mmol), iodobenzene diacetate (0.55 g, 1.7mmol) in 4 mL of *p*-xylene was microwave heated at 145 ^{0}C for 3 h. The excess solvent from the mixture is removed at reduced pressure and the crude product was purified by column chromatography to give pure **5** 0.1535g (90 %)

***R*$_f$ -Value:** Hexane/Ethyl acetate (9:1 *v/v*) = 0.19.

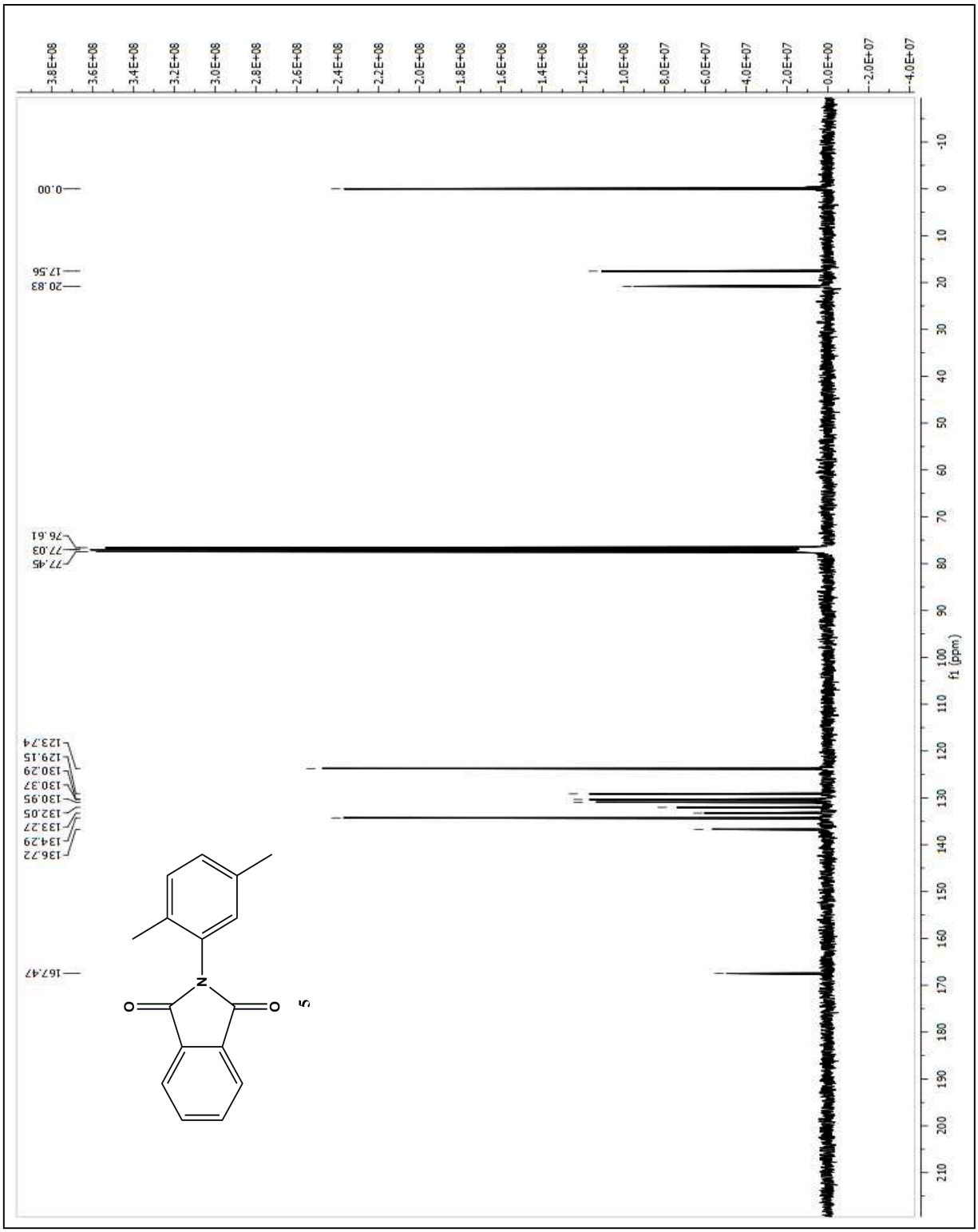
^{1}H NMR (300 MHz, CDCl$_3$): δ = 2.16(s, 3H), 2.36 (s, 3H), 7.17-7.28 (m, 3H), 7.80 (dd, *J* = 6 Hz, *J* = 3 Hz, 2H), 7.97 (dd, *J* = 3 Hz, *J* = 3 Hz, 2H).

^{13}C NMR (75 MHz, CDCl$_3$): δ = 17.56, 20.83, 123.74, 129.15, 130.29, 130.37, 130.95, 132.05, 133.27, 134.29, 136.72, 167.47.

LRMS EI (m/z): [M+] calc'd for $C_{16}H_{13}NO_2$ 251.09, observed 251.10 m/z.

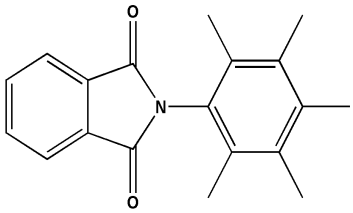


Spectra 5. ^{1}H NMR of Compound **5**



Spectra 6. ^{13}C NMR of Compound **5**

Synthesis of 2-(2, 3, 4, 5, 6-pentamethylphenyl) isoindoline-1, 3-dione (6)



6

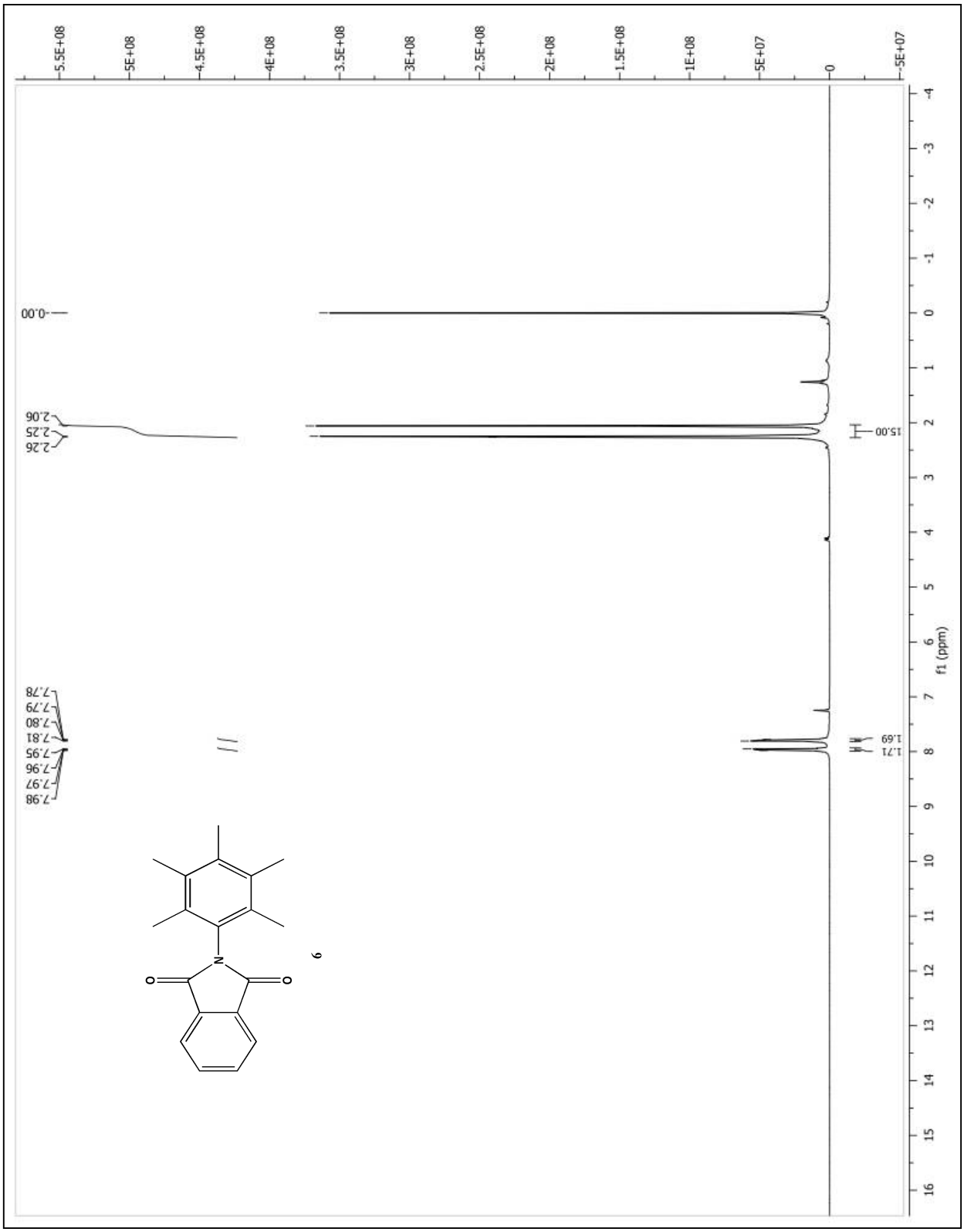
A magnetically stirred solution of phthalimide (0.10 g, 0.68mmol), iodobenzene diacetate (0.55 g, 1.7mmol), pentamethyl benzene (0.3 g, 2.048 mmol) in 4 mL of acetonitrile was microwave heated at 145 ^{0}C for 3h. The excess solvent from the mixture is removed at reduced pressure and the crude product was purified by column chromatography to give pure **6** (0.0846 g, 43 %).

***R_f*-Value:** Hexane/Ethyl acetate (9:1 *v/v*) = 0.23.

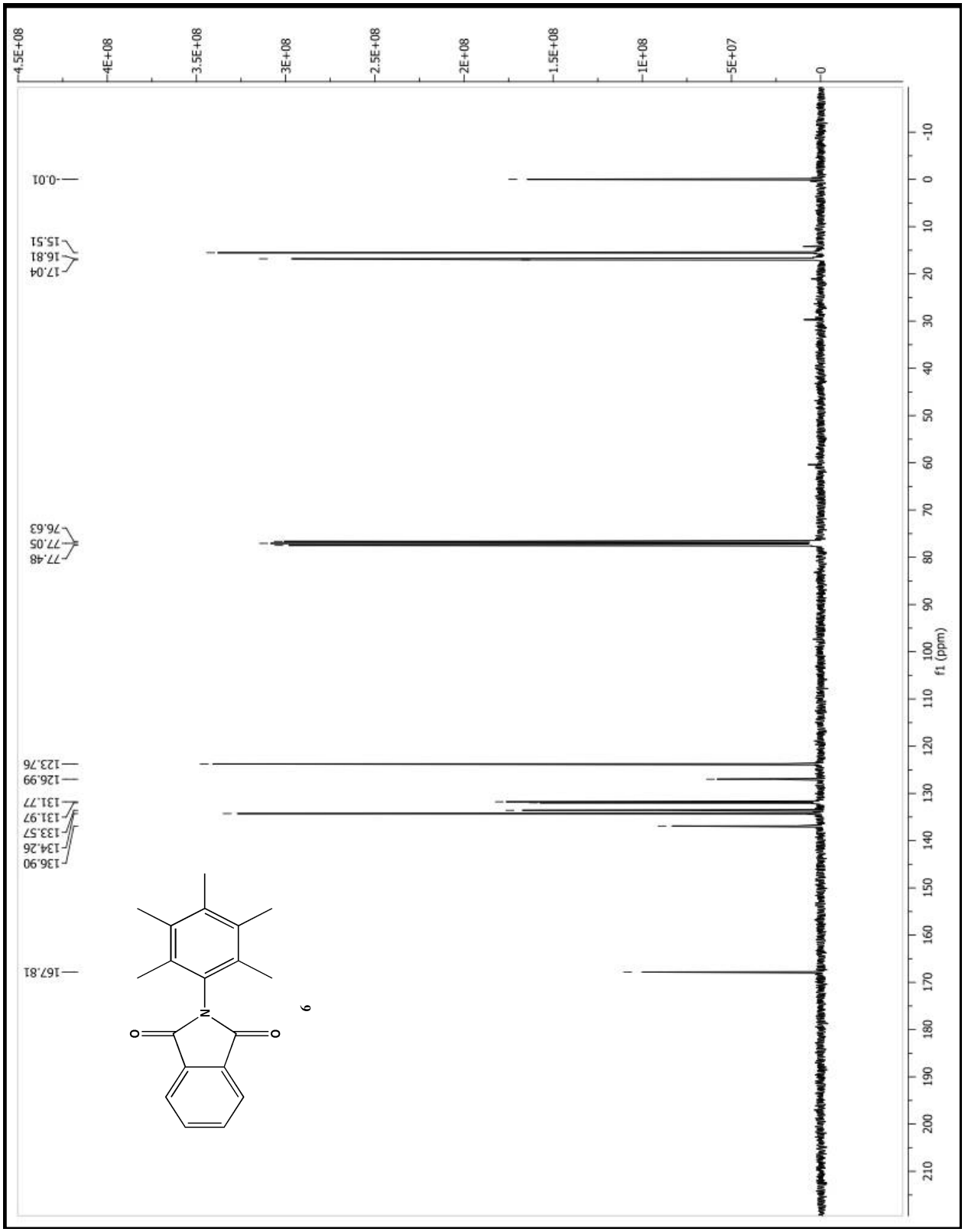
^{1}H NMR (300 MHz, $CDCl_3$): δ = 2.06-2.26 (m, 15H), 7.79 (dd, *J* = 3 Hz, 3 Hz, 2H), 7.97 (dd, *J* = 3 Hz, 3 Hz, 2H).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 15.51, 16.81, 17.04, 123.76, 126.99, 131.77, 131.97, 133.57, 134.26, 136.9, 167.81.

LRMS EI (m/z): [M+] calc'd for $C_{19}H_{19}NO_2$ 293.14, observed 293.15 m/z.

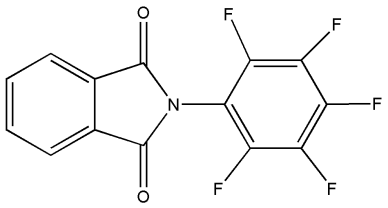


Spectra 7. ^{1}H NMR of Compound **6**



Spectra 8. ^{13}C NMR of Compound **6**

Synthesis of 2-(perfluorophenyl)isoindoline-1,3-dione (7)



7

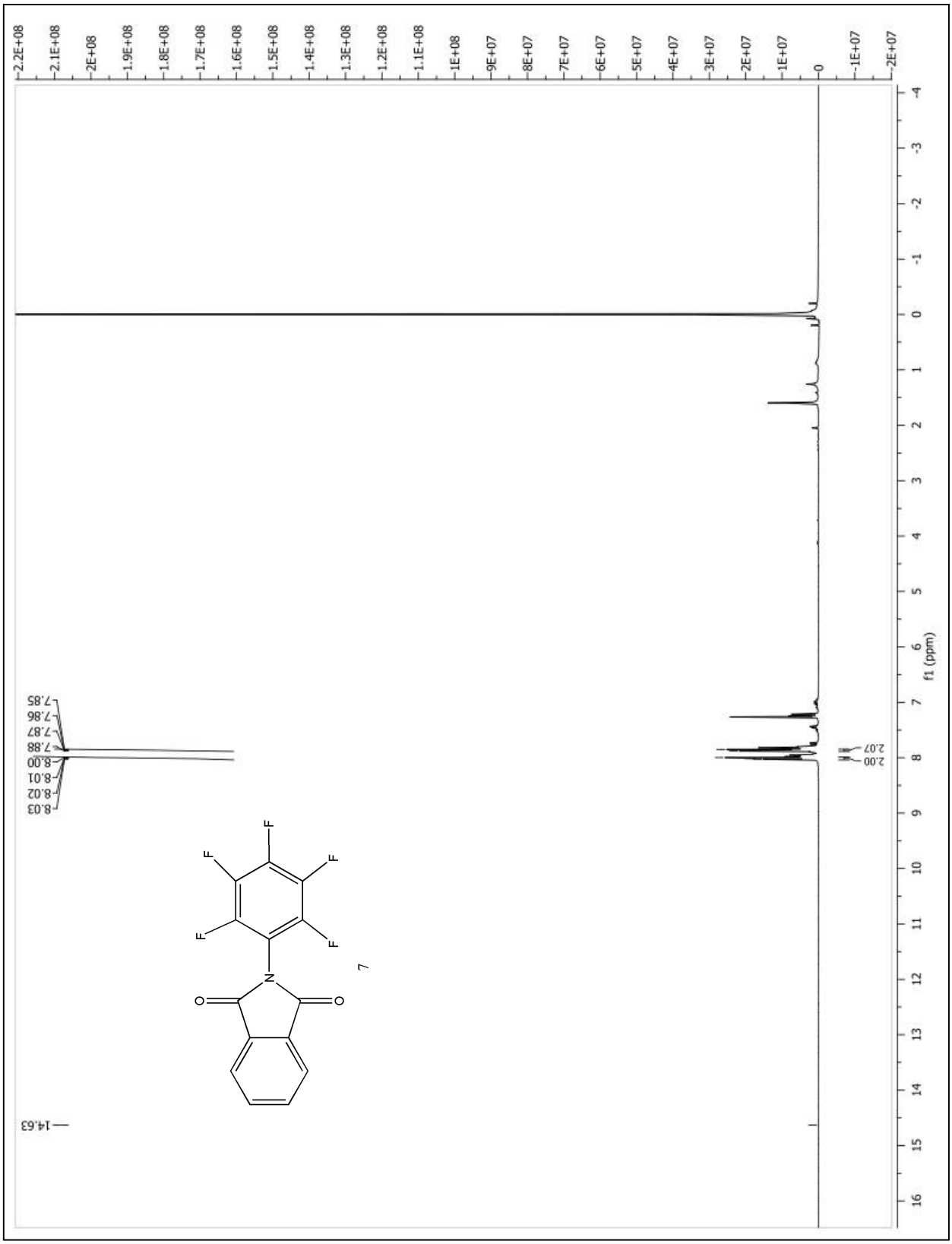
A magnetically stirred solution of Phthalimide (0.10 g, 0.68mmol), iodobenzene diacetate (0.55 g, 1.7mmol), in 4 mL of pentafluorobenzene was microwave heated at 145 ^{0}C for 3h. The excess solvent from the mixture is removed at reduced pressure and the crude product was purified by column chromatography to give pure **7** (0.04 g, 20 %).

***R_f* -Value:** Hexane/Ethyl acetate (9:1 *v/v*) = 0.18.

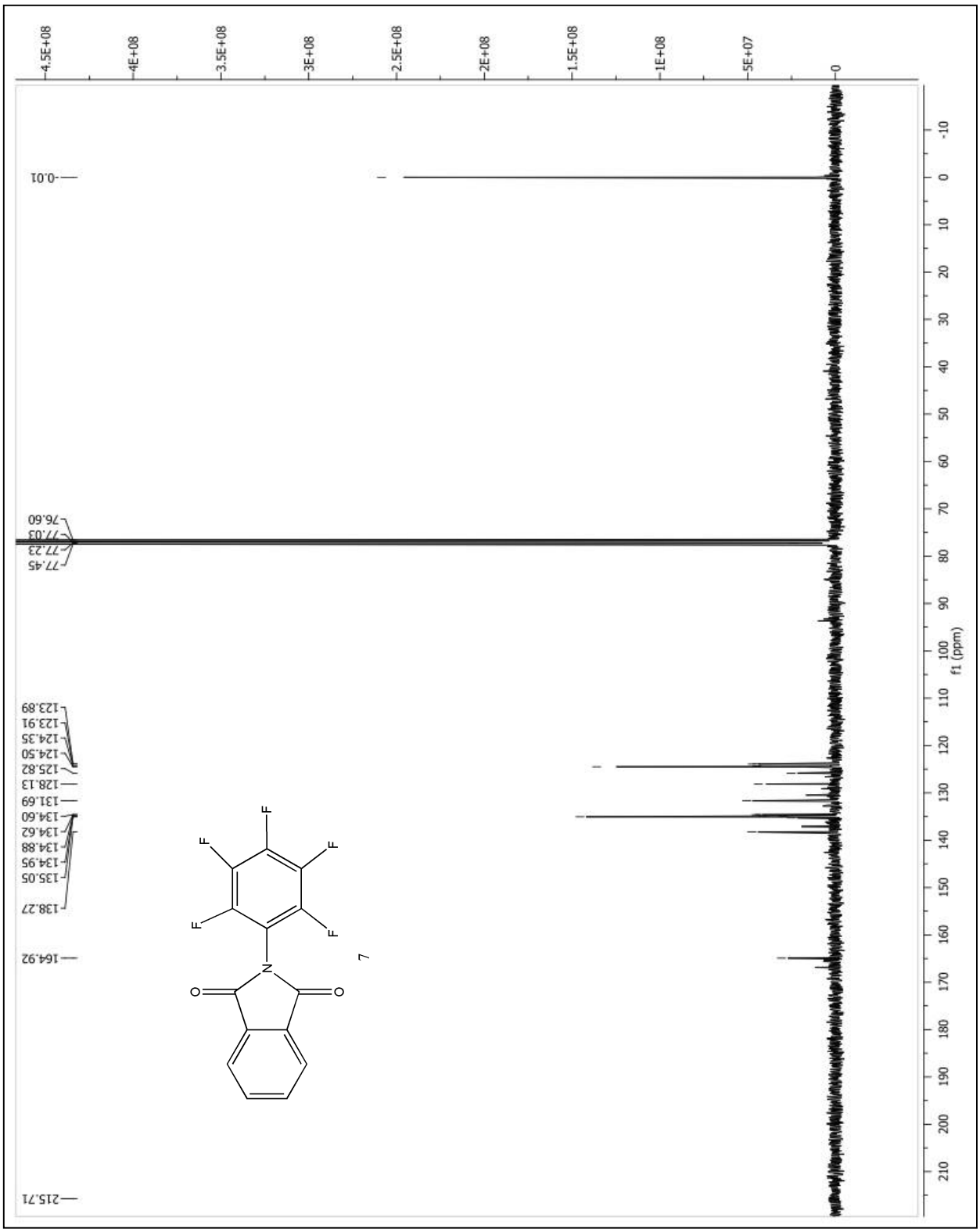
^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.87 (dd, *J* = 3 Hz, *J* = 3 Hz, 2H), 8.01 (dd, *J* = 3 Hz, *J* = 3 Hz, 2H).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 123.89, 123.91, 124.35, 124.5, 125.82, 128.13, 131.69, 134.62, 134.88, 134.95, 135.05, 138.27.

LRMS EI (m/z): [M+] calc'd for $C_{14}H_4F_5NO_2$ 313.02, observed 313.0 m/z.

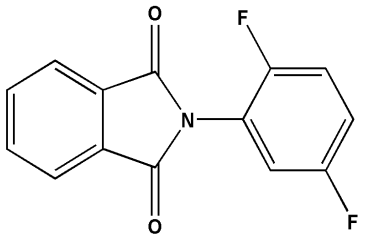


Spectra 9. ^{1}H NMR of Compound **7**



Spectra 10. ^{13}C NMR of Compound **7**

Synthesis of 2-(2,5-difluorophenyl)isoindoline-1,3-dione (8)



8

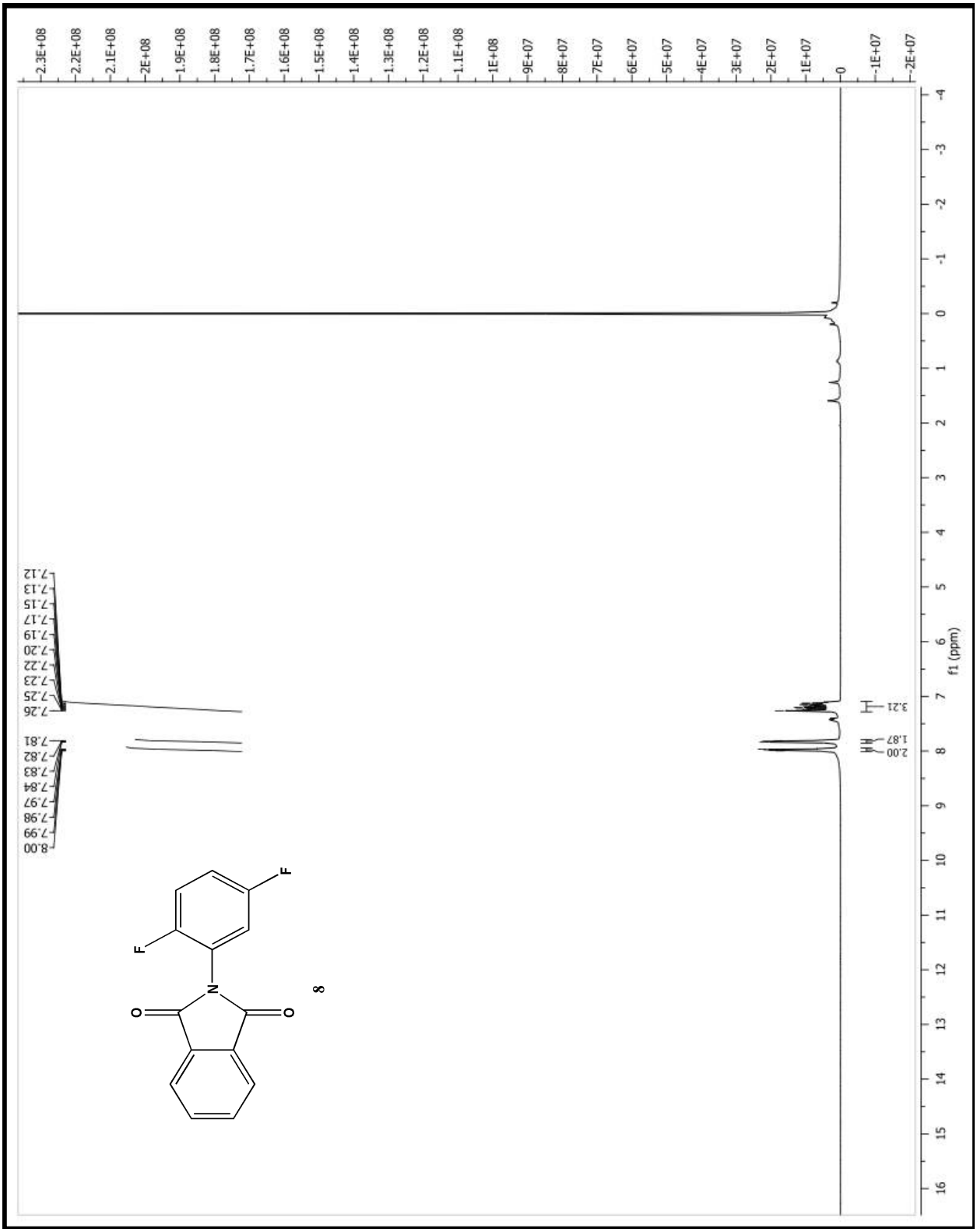
A magnetically stirred solution of phthalimide (0.10 g, 0.68mmol), iodobenzene diacetate (0.55 g, 1.7mmol) in 4 mL of 1, 4-difluorobenzene was microwave heated at 145 ^{0}C for 3h. The excess solvent from the mixture is removed at reduced pressure and crude product was purified by column chromatography to give pure **8** (0.0917 g, 53 %).

***R*$_f$ -Value:** Hexane/Ethyl acetate (9:1 *v/v*) = 0.19.

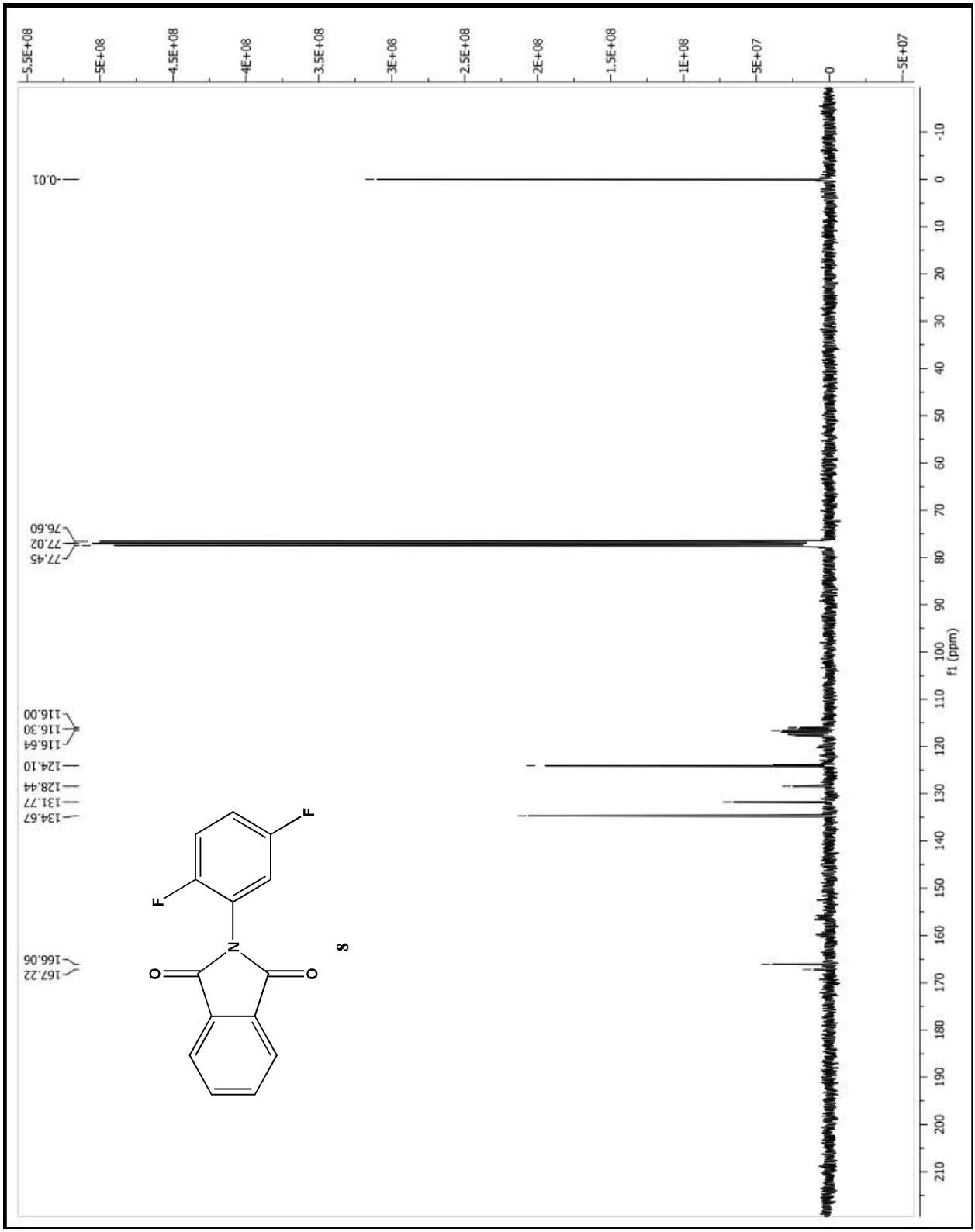
^{1}H NMR (300 MHz, CDCl$_3$): δ = 7.12-7.26 (m, 3H), 7.82 (dd, *J* = 3 Hz, *J* = 3 Hz, 2H), 7.98 (dd, *J* = 3 Hz, *J* = 3 Hz, 2H).

^{13}C NMR (75 MHz, CDCl$_3$): δ = 116.00, 116.30, 116.64, 124.10, 128.44, 131.77, 134.67, 166.06, 167.22.

LRMS EI (m/z): [M+] calc'd for $C_{14}H_7F_2NO_2$ 259.04, observed 259.1 m/z.

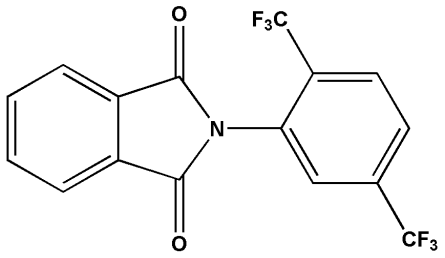


Spectra 11. ^{1}H NMR of Compound **8**



Spectra 12. ^{13}C NMR of Compound **8**

Synthesis of 2-(2, 5-bis (trifluoromethyl) phenyl) isoindoline-1, 3-dione (9)



9

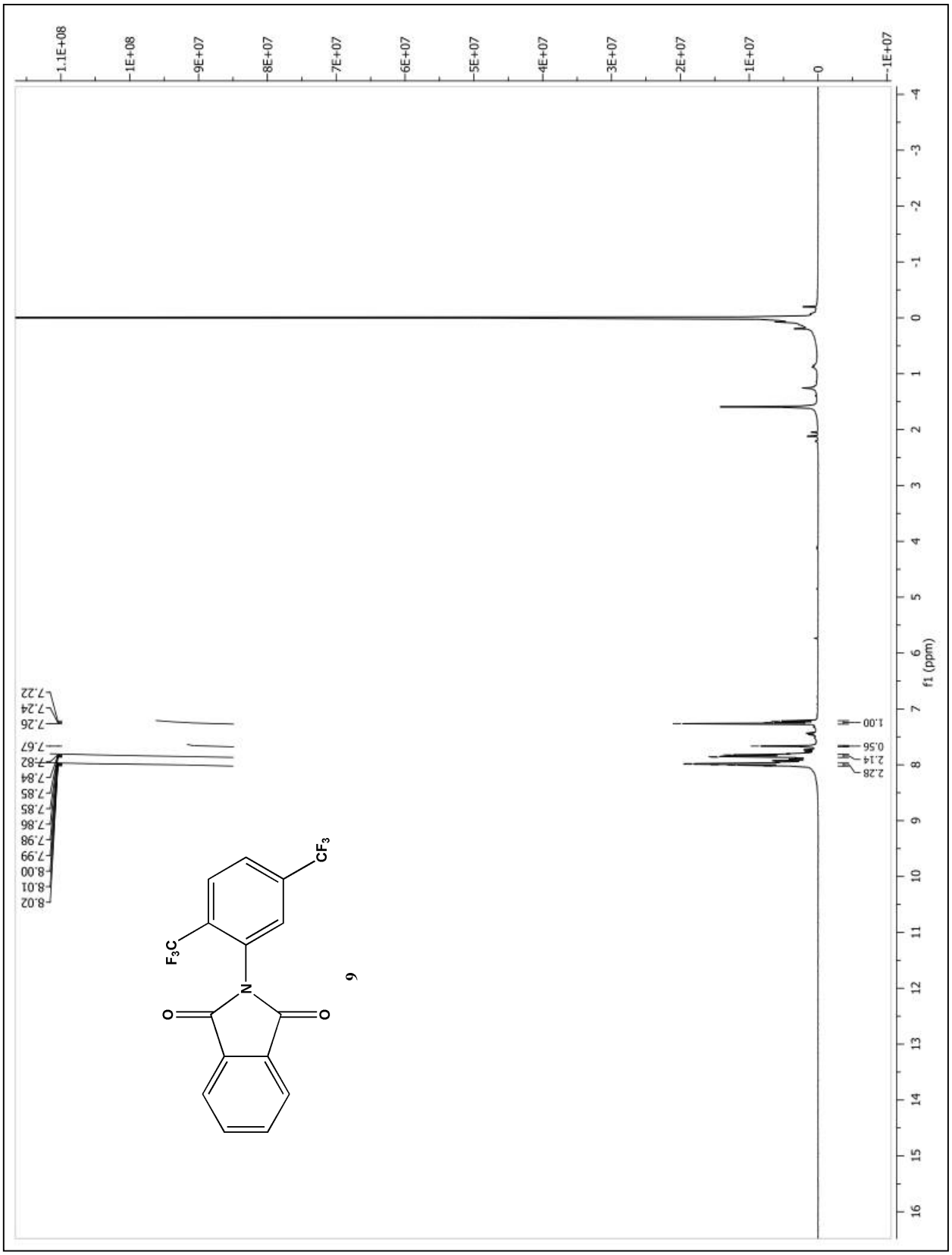
A magnetically stirred solution of Phthalimide (0.1g, 0.68mmol), (Diacetoxyiodo) benzene (0.55 g, 1.7mmol), in 4 mL of 1,4-bistrifluoromethylbenzene was microwave heated at 145 ^{0}C for 3h. The excess solvent from the mixture is removed at reduced pressure and crude product was purified by column chromatography to give pure **9** (0.2441 g, 24 %).

***R_f* -Value:** Hexane/Ethyl acetate (9:1 *v/v*) = 0.2187.

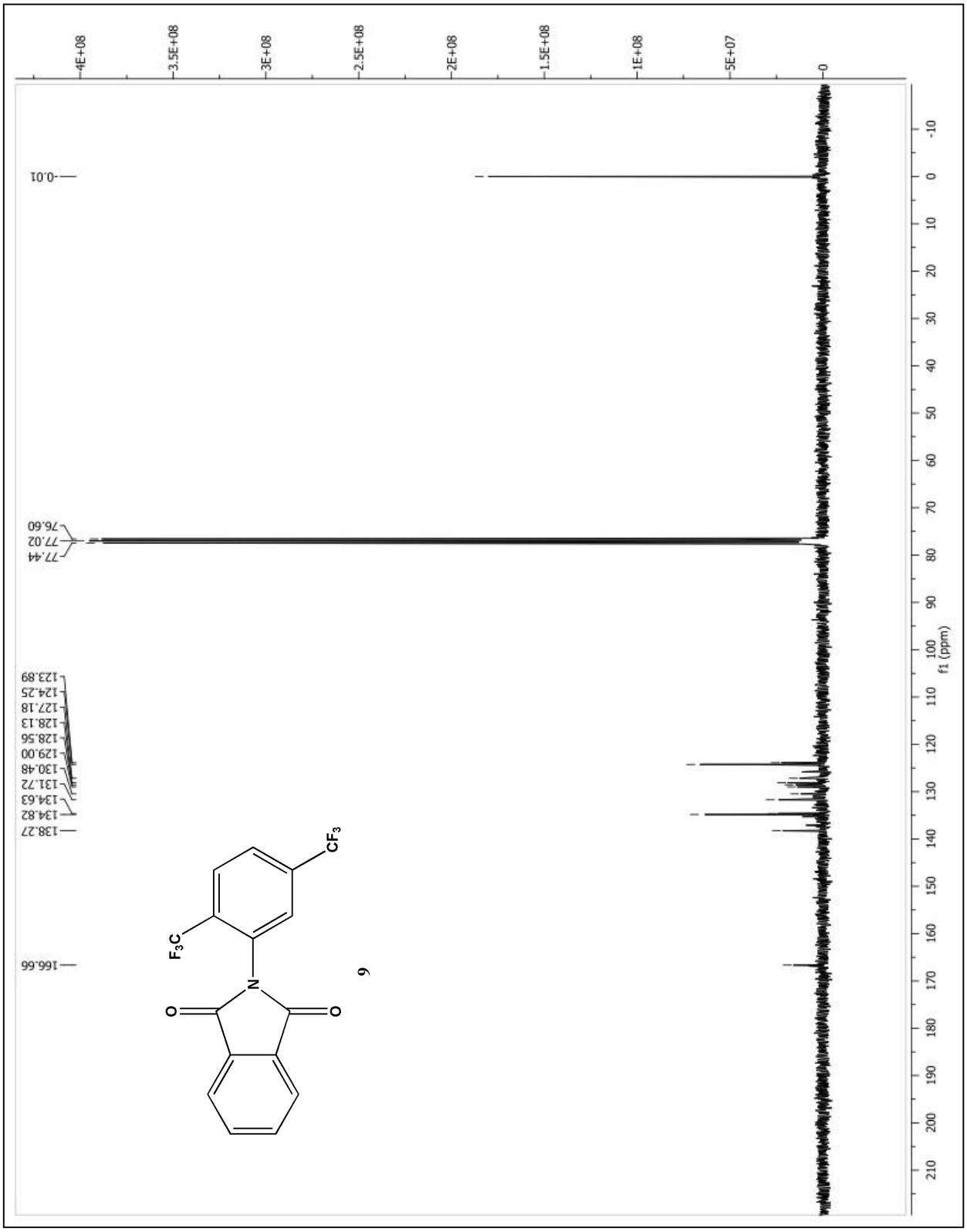
^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.22-7.26 (m, 1H), 7.67 (s, 1H), 7.82-8.02 (m, 5H).

^{13}C NMR (75 MHz, $CDCl_3$): δ= 123.89, 124.25, 127.18, 128.13, 129.00, 130.48, 131.72, 134.63, 134.82, 138.27, 166.66.

LRMS EI (m/z): [M+] calc'd for $C_{16}H_7F_6NO_2$ 359.0, observed 359.0 m/z.

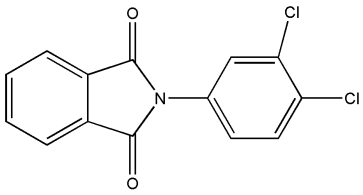


Spectra 13. ^{1}H NMR of Compound **9**



Spectra 14. ^{13}C NMR of Compound **9**

Synthesis of 2-(3, 4-dichlorophenyl) isoindoline-1,3-dione (10)



10

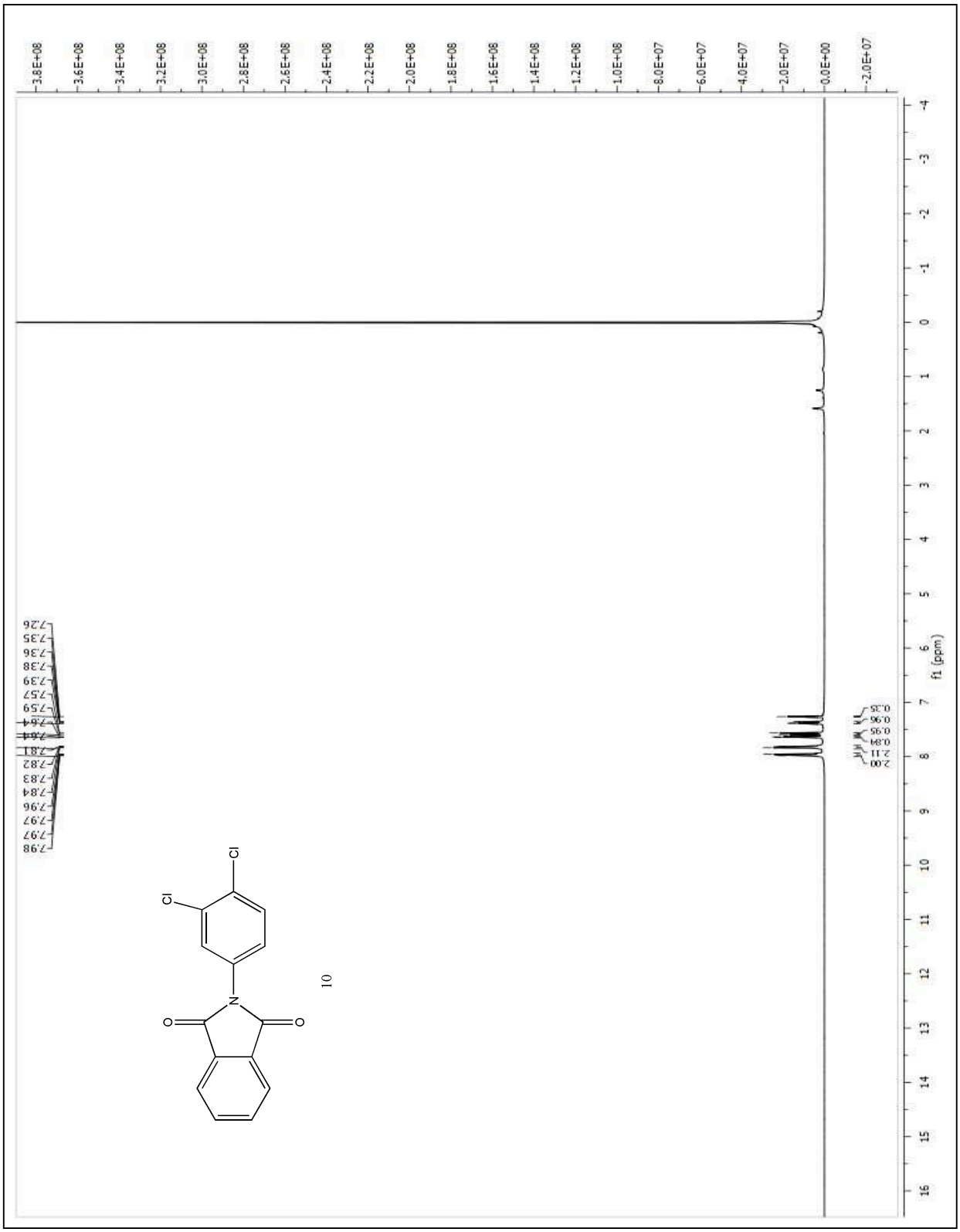
A magnetically stirred solution of phthalimide (0.10 g, 0.68mmol), iodobenzene diacetate (0.55 g, 1.7mmol) in 4 mL of 1,2-dichlorobenzene was microwave heated at 145 ^{0}C for 3 h. The excess solvent from the mixture is removed at reduced pressure and crude product was purified by column chromatography to give pure **10** (0.1118 g, 56 %).

***R_f*-Value:** Hexane/Ethyl acetate (9:1 *v/v*) = 0.19.

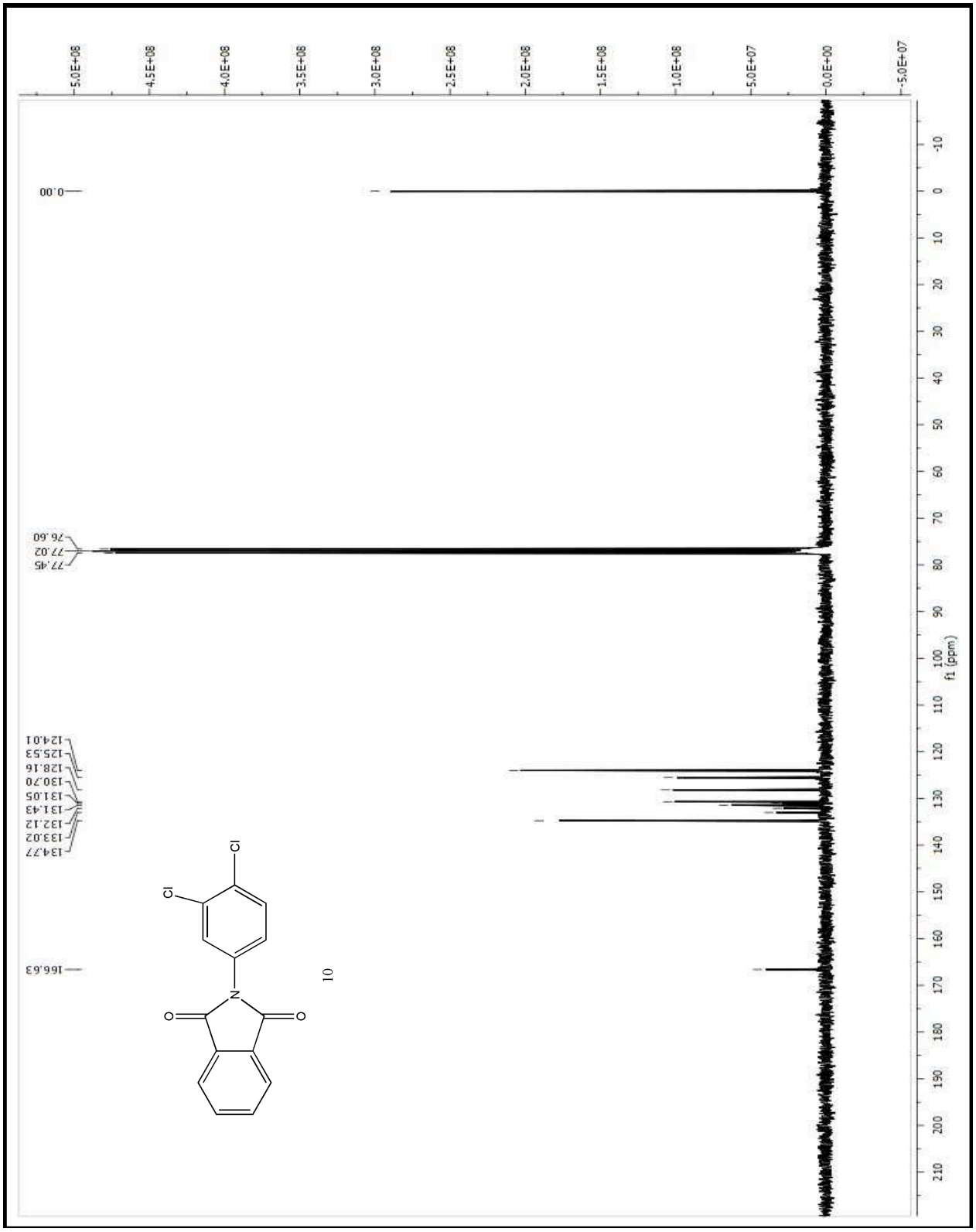
^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.37 (dd, *J* = 8.6, 2.4 Hz, 1H), 7.58 (d, *J* = 8.6 Hz, 1H), 7.64 (d, *J* = 2.4 Hz, 1H), 7.86 – 7.79 (m, 2H), 7.98 – 7.94 (m, 2H).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 124.01, 125.53, 128.16, 130.70, 131.05, 131.43, 132.12, 133.02, 134.77, 166.63.

LRMS EI (m/z): [M+] calc'd for $C_{14}H_7F_2NO_2$ 291.0, observed 291.0 m/z.

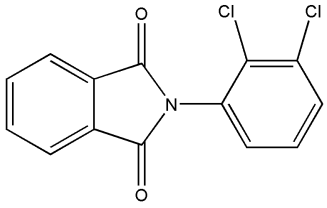


Spectra 15. ^{1}H NMR of Compound **10**



Spectra 16. ^{13}C NMR of Compound **10**

Synthesis of 2-(2,3-dichlorophenyl)isoindoline-1,3-dione (11)



11

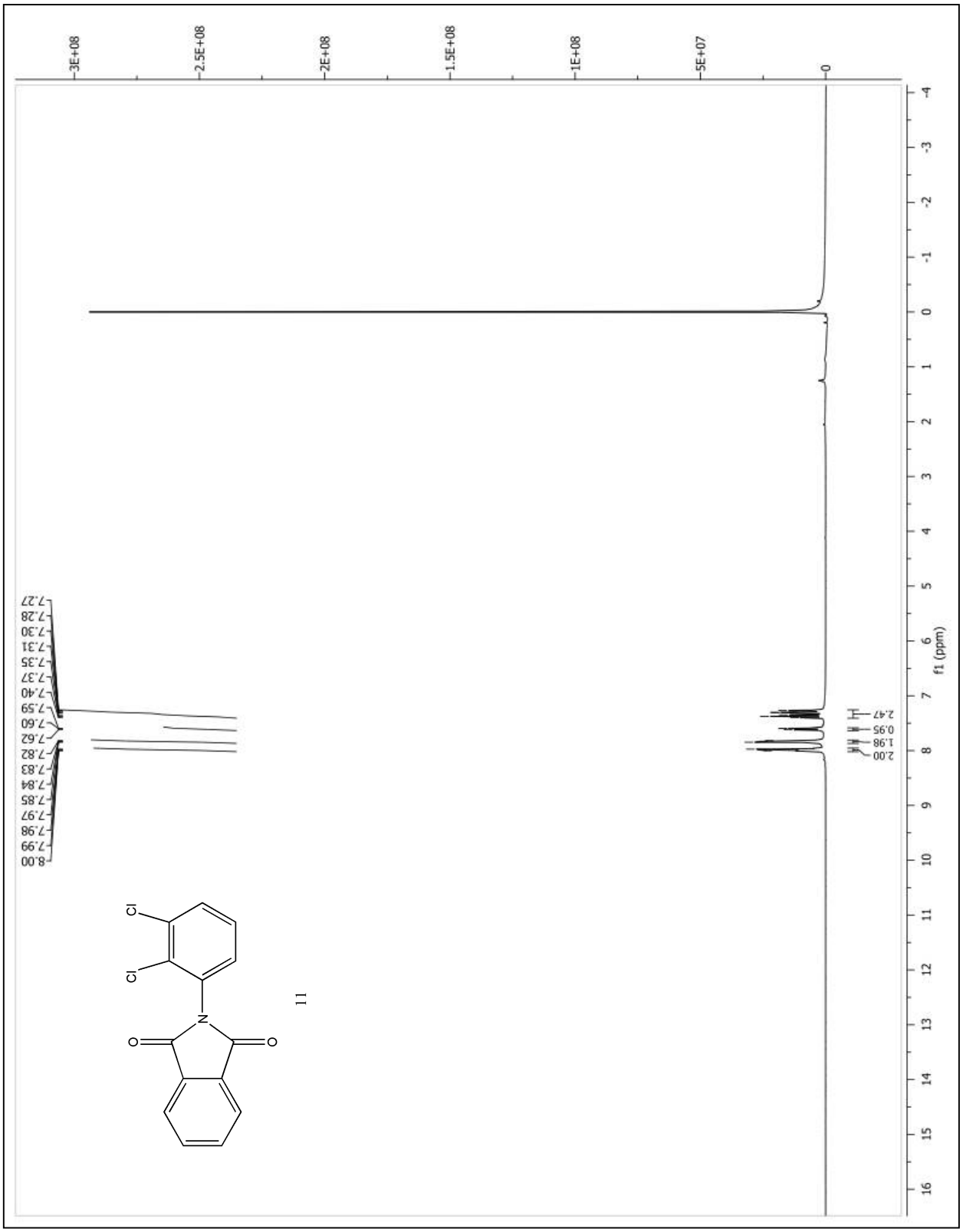
A magnetically stirred solution of Phthalimide (0.10 g, 0.68mmol), iodobenzene diacetate (0.55 g, 1.7mmol) in 4 mL of 1,2-dichlorobenzene was microwave heated at 145 ^{0}C for 3 h. The excess solvent from the mixture is removed at reduced pressure and crude product was purified by column chromatography to give pure **11** (0.034 g, 17 %).

***R_f* -Value:** Hexane/Ethyl acetate (9:1 *v/v*) = 0.09.

^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.27-7.4 (m, 2H), 7.59-7.62 (m, 1H), 7.83 (dd, *J* = 3, 3 Hz, 2H), 7.99 (dd, *J* = 3 Hz, 3 Hz, 2H).

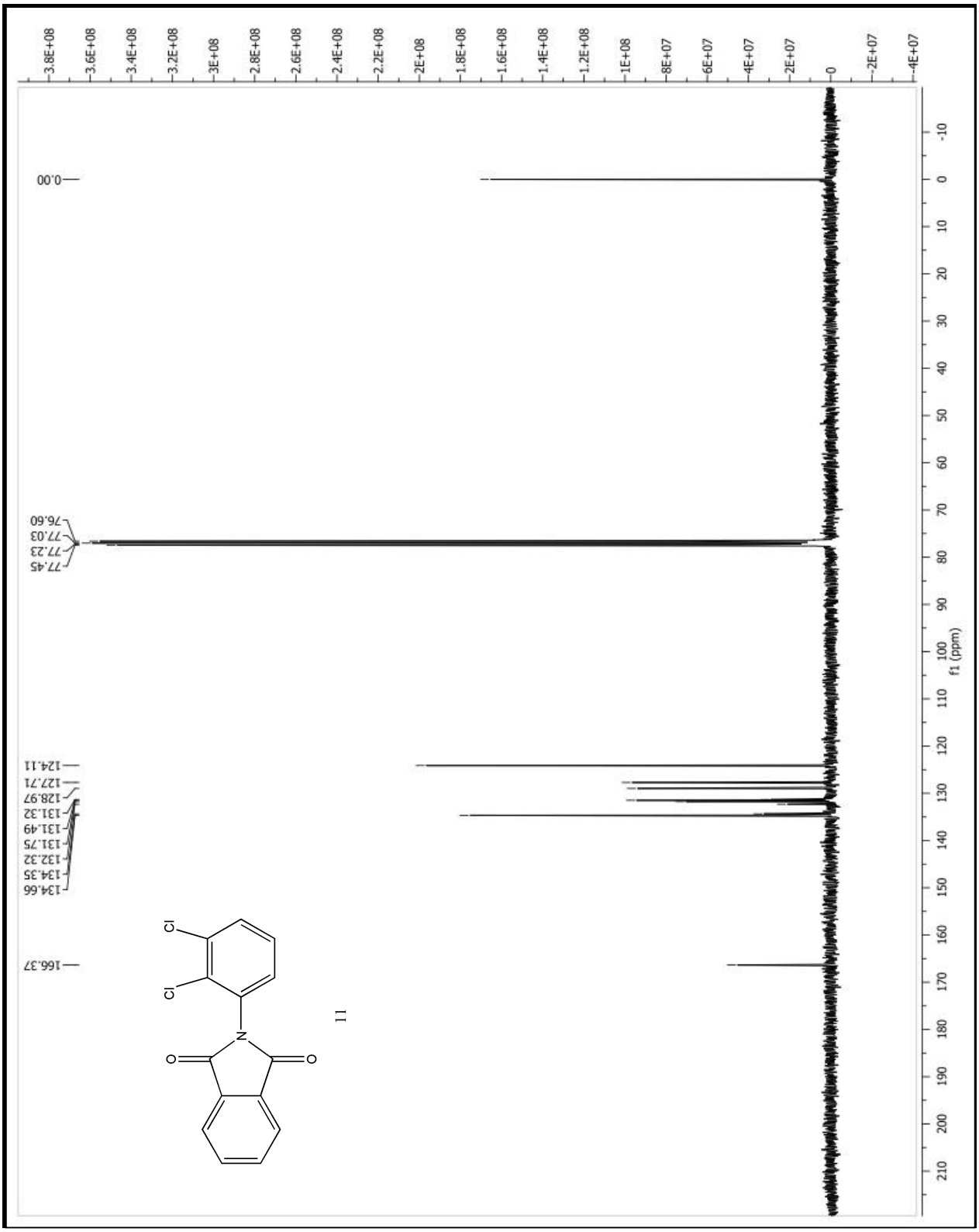
^{13}C NMR (75 MHz, $CDCl_3$): δ = 124.11, 127.71, 128.97, 131.32, 131.49, 131.75, 132.32, 134.35, 134.66, 166.37.

LRMS EI (m/z): [M+] calc'd for $C_{14}H_7F_2NO_2$ 291.0, observed 291.0 m/z.



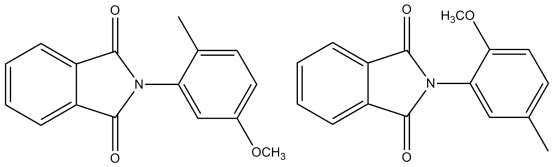
11

Spectra 17. ^{1}H NMR of Compound **11**



Spectra 18. ^{13}C NMR of Compound **11**

Synthesis of 14 and 15



14 15

14:15 = 2:3

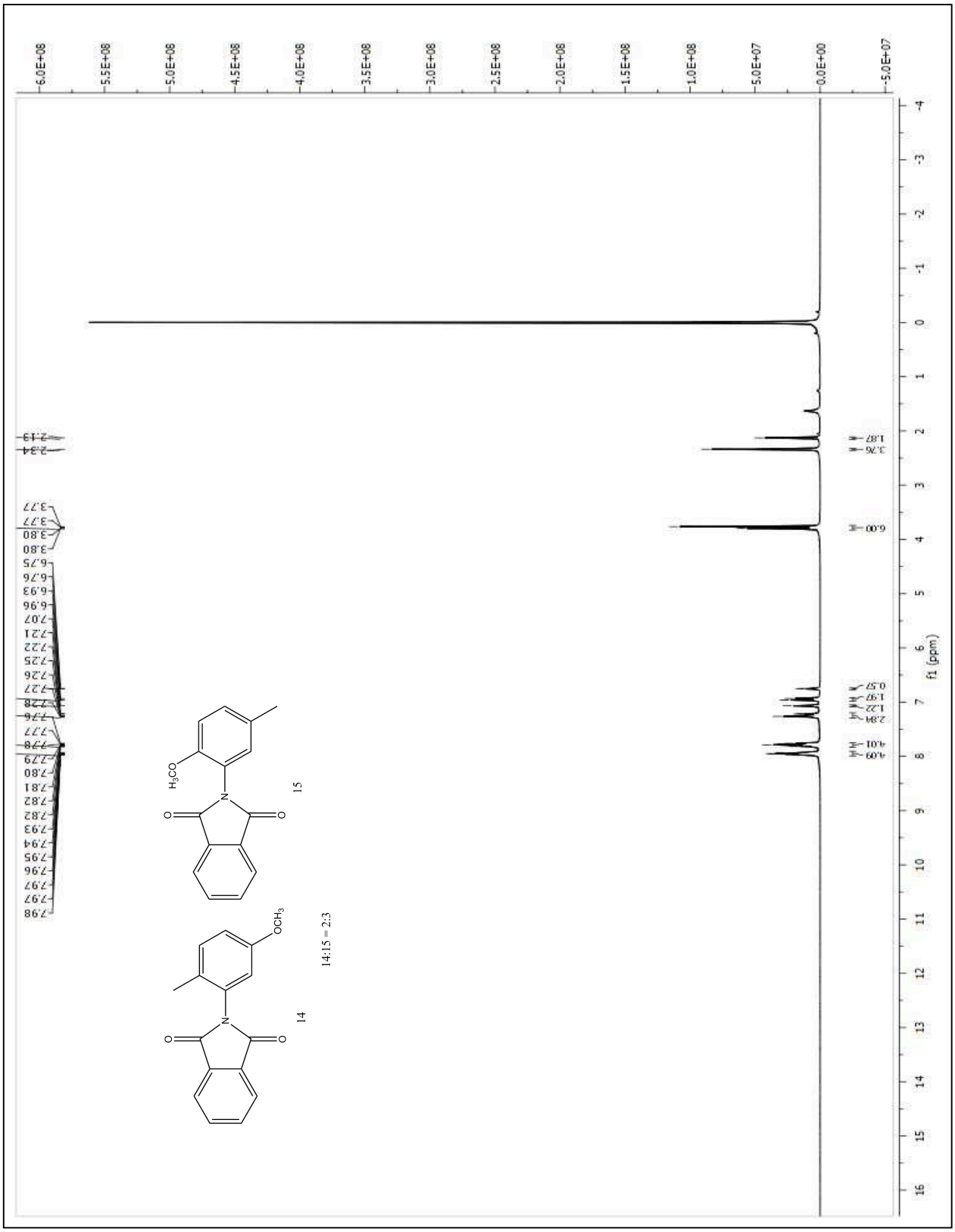
A magnetically stirred solution of phthalimide (0.10 g, 0.68mmol), iodobenzene diacetate (0.55 g, 1.7mmol) in 4 mL of 4-methylanisole was microwave heated at 145 ^{0}C for 3 h. The excess solvent from the mixture is removed at reduced pressure and crude product was purified by column chromatography to give a mixture of **14** and **15** (0.1287 g, 70 %, **14**:**15** = 2:3).

***R_f* -Value:** Hexane/Ethyl acetate (9:1 *v/v*) = 0.1714.

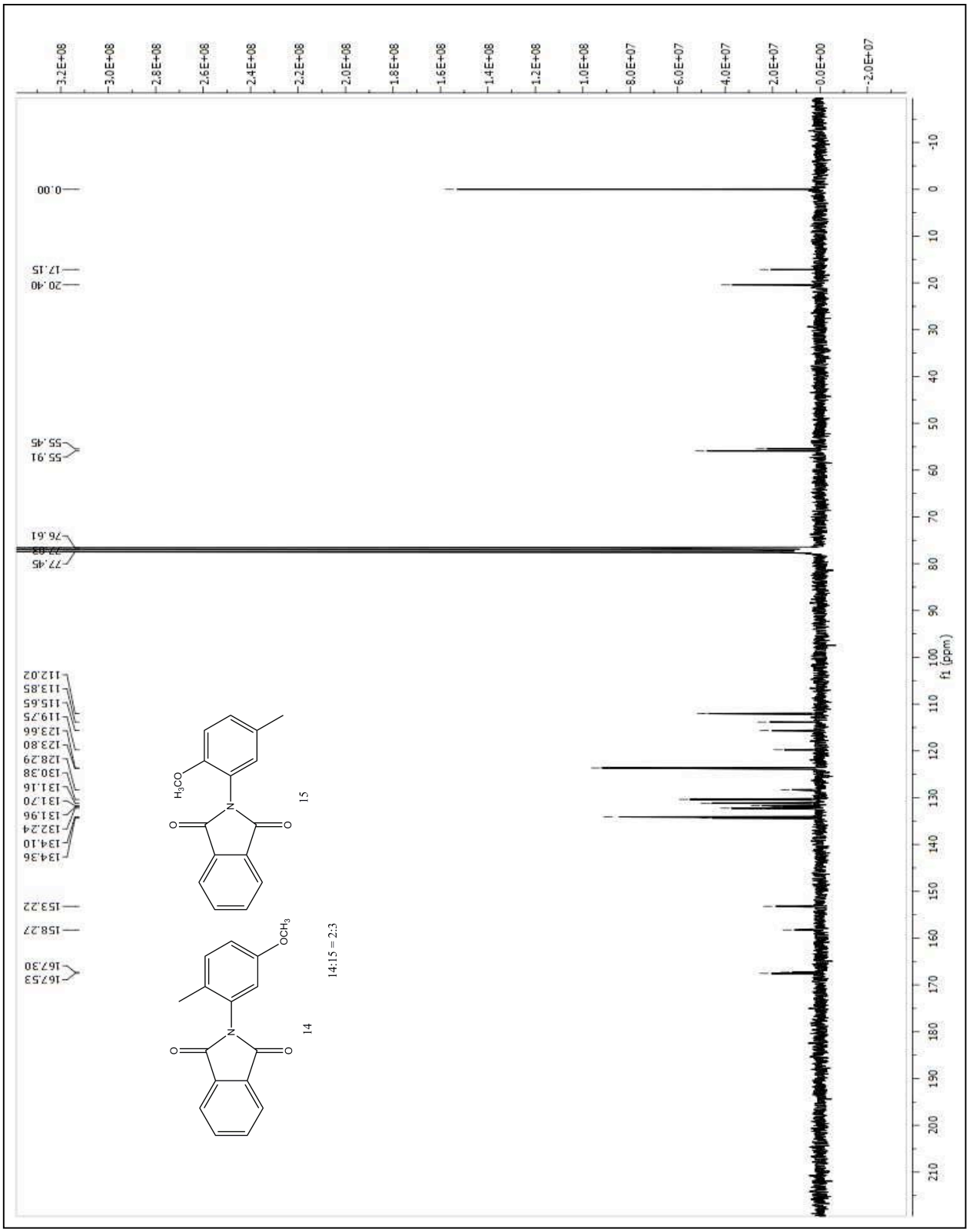
^{1}H NMR (300 MHz, $CDCl_3$): δ = 2.13 (s, 2H), 2.34 (s, 3H), 3.78 (dd, *J* = 9.6, 2.1 Hz, 6H), 6.75 (d, *J* = 2.5 Hz, 1H), 6.95 (d, *J* = 8.4 Hz, 2H), 7.07 (s, 1H), 7.20 – 7.30 (m, 3H), 7.76-7.82 (m, 4H), 7.91 – 7.99 (m, 4H).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 17.15, 20.40, 55.45, 55.91, 112.02, 113.85, 115.65, 119.75, 123.66, 123.80, 128.29, 130.38, 131.16, 131.70, 131.96, 132.24, 134.10, 134.36, 153.22, 158.27, 167.30, 167.53.

LRMS EI (m/z): [M+] calc'd for $C_{14}H_9NO_2$ 267.09, observed 267.10 m/z.

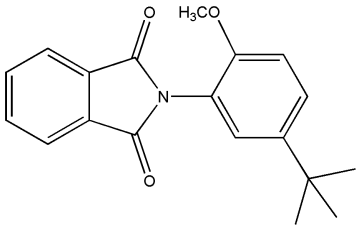


Spectra 19. ^{1}H NMR of Compound **14** and **15**



Spectra 20. ^{13}C NMR of Compound **14** and **15**

Synthesis of 2-(5-tert-butyl-2-methoxyphenyl) isoindoline-1,3-dione (16)



16

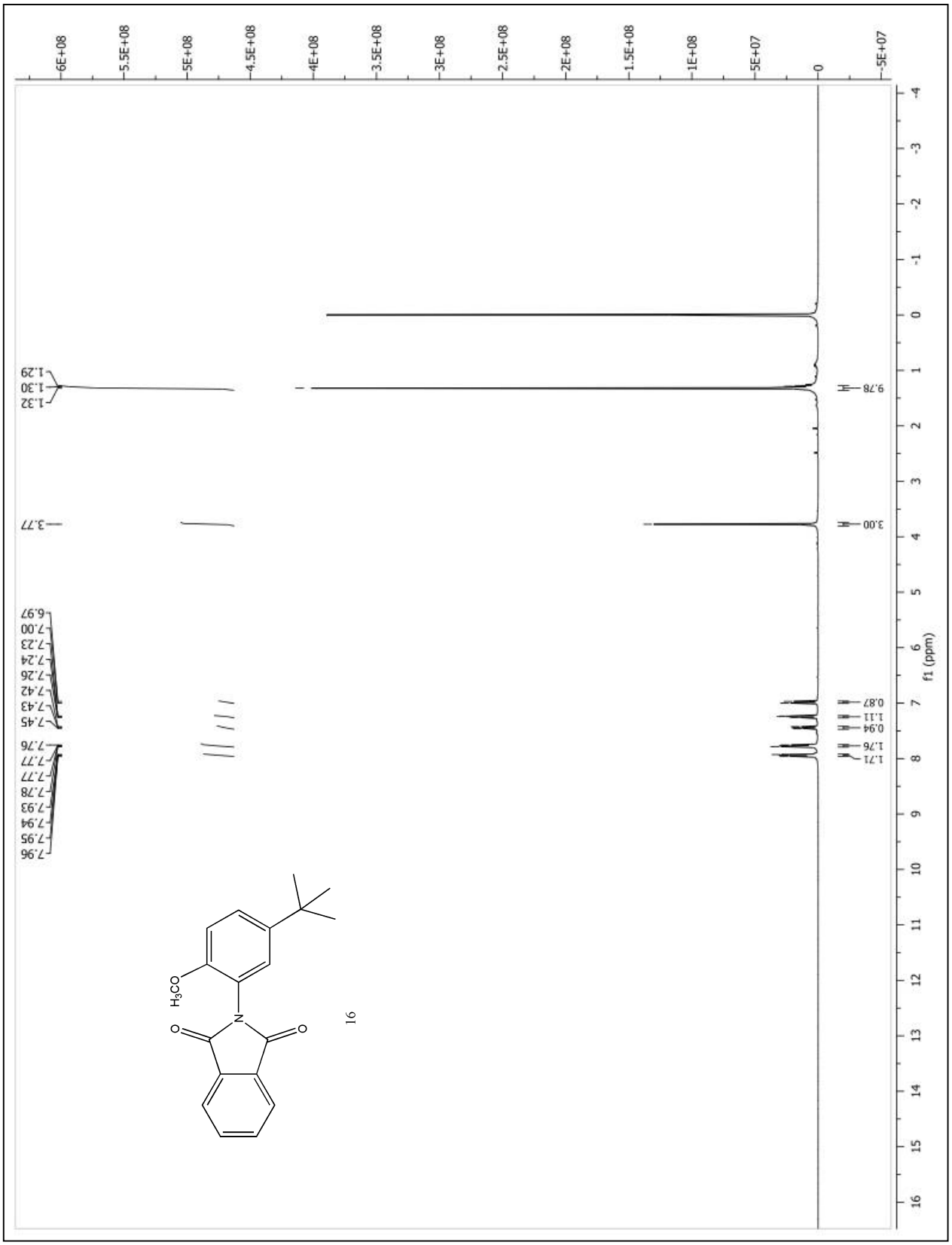
A magnetically stirred solution of phthalimide (0.10 g, 0.68mmol), iodobenzene diacetate (0.55 g, 1.7mmol), 1-*tert*-butyl-4-methoxybenzene (0.22 g, 1.359 mmol) in 4 mL of acetonitrile was microwave heated at 145 ^{0}C for 3 h. The excess solvent from the mixture is removed at reduced pressure and crude product was purified by column chromatography to give pure **16** (0.117 g, 56 %).

***R*$_f$ -Value:** Hexane/Ethyl acetate (9:1 *v/v*) = 0.11.

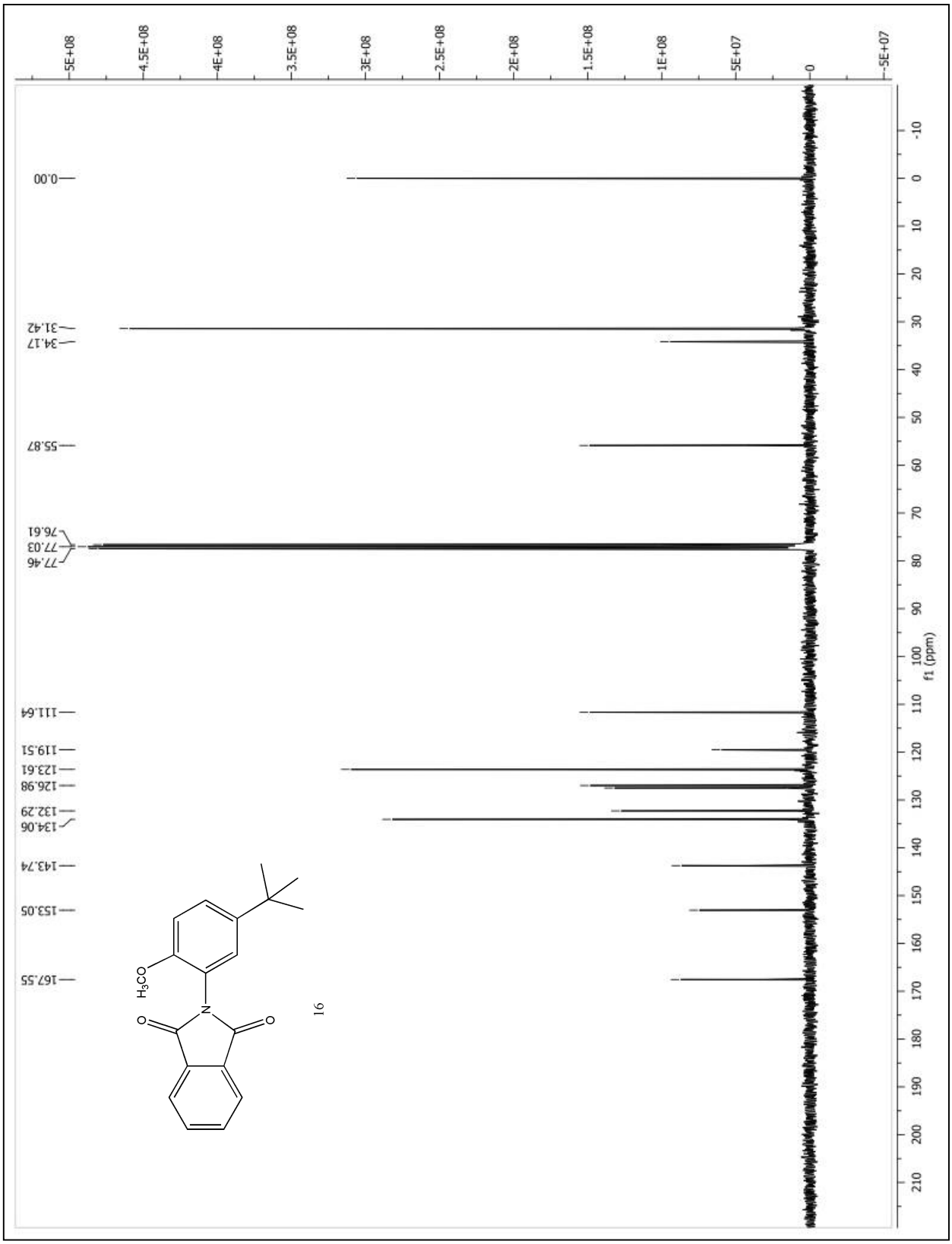
^{1}H NMR (300 MHz, CDCl$_3$): δ = 1.29-1.32 (m, 9H), 3.77 (s, 3H), 6.98 (d, *J* = 9 Hz, 1H), 7.25 (m, 1H) 7.44 (dd, *J* = 3 Hz, 3 Hz, 1 H) 7.77 (dd, *J* = 3 Hz, 3 Hz, 2H), 7.94 (dd, *J* = 3 Hz, 3 Hz, 2H).

^{13}C NMR (75 MHz, CDCl$_3$): δ = 31.42, 34.17, 55.87, 111.64, 119.51, 123.61, 126.98, 132.29, 134.06, 143.74, 153.05, 167.55.

LRMS EI (m/z): [M+] calc'd for $C_{19}H_{19}NO_3$ 309.14, observed 309.12 m/z.

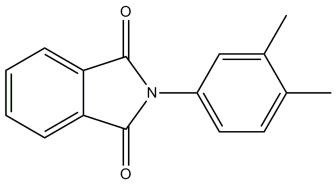


Spectra 21. ^{1}H NMR of Compound **16**

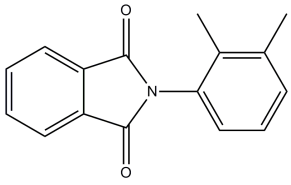


Spectra 22. ^{13}C NMR of Compound **16**

Synthesis of 12 and 13



12



13

12:13 = 3:4

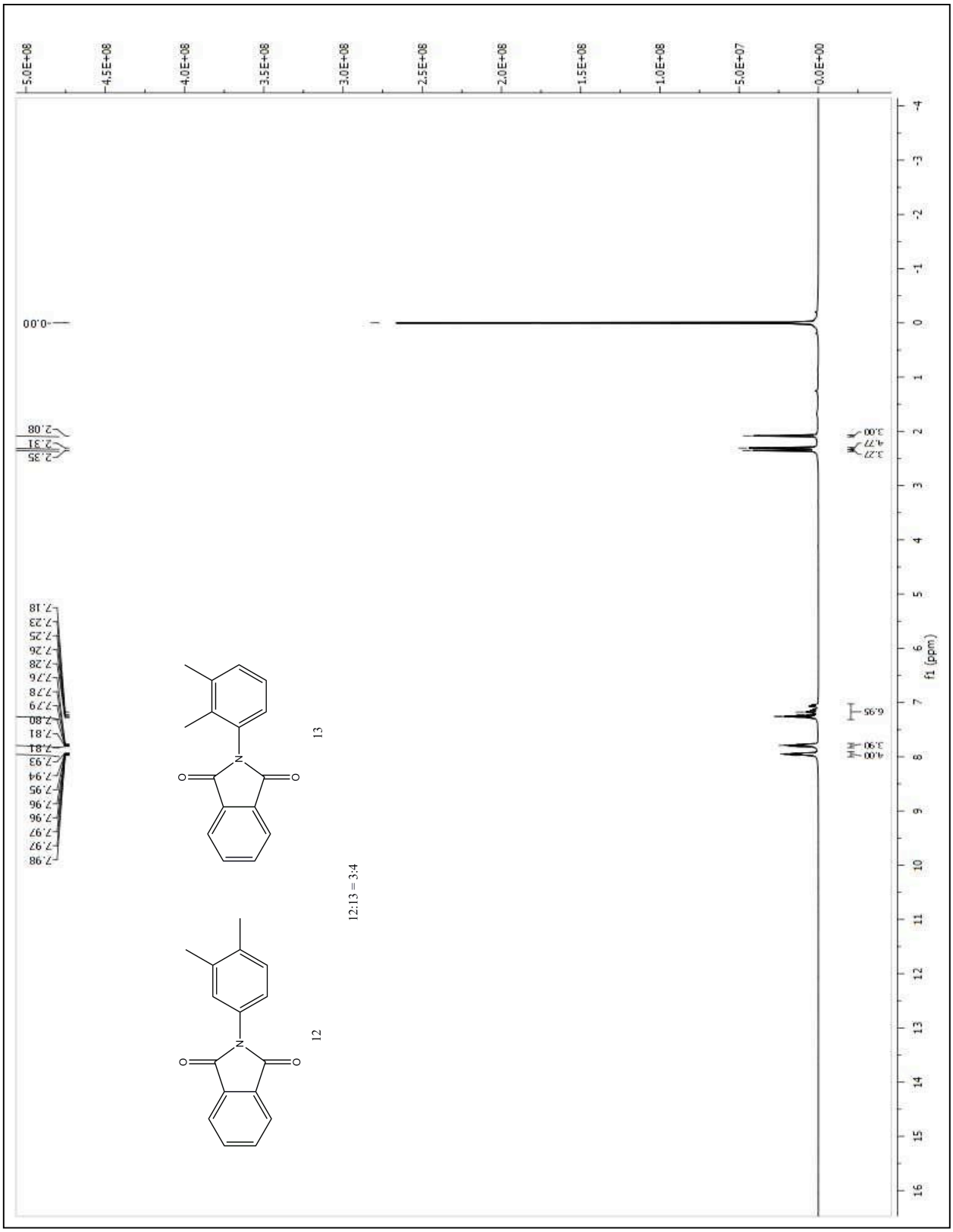
A magnetically stirred solution of phthalimide (0.10 g, 0.68mmol), iodobenzene diacetate (0.55 g, 1.7mmol) in 4 mL of *o*-xylene was microwave heated at 145 ^{0}C for 3 h. The excess solvent from the mixture is removed at reduced pressure and crude product was purified by column chromatography to give a mixture of **12** and **13** (0.1333 g, 80 %, **12**:**13** = 3:4).

***R*$_f$ -Value:** Hexane/Ethyl acetate (9:1 *v/v*) = 0.22.

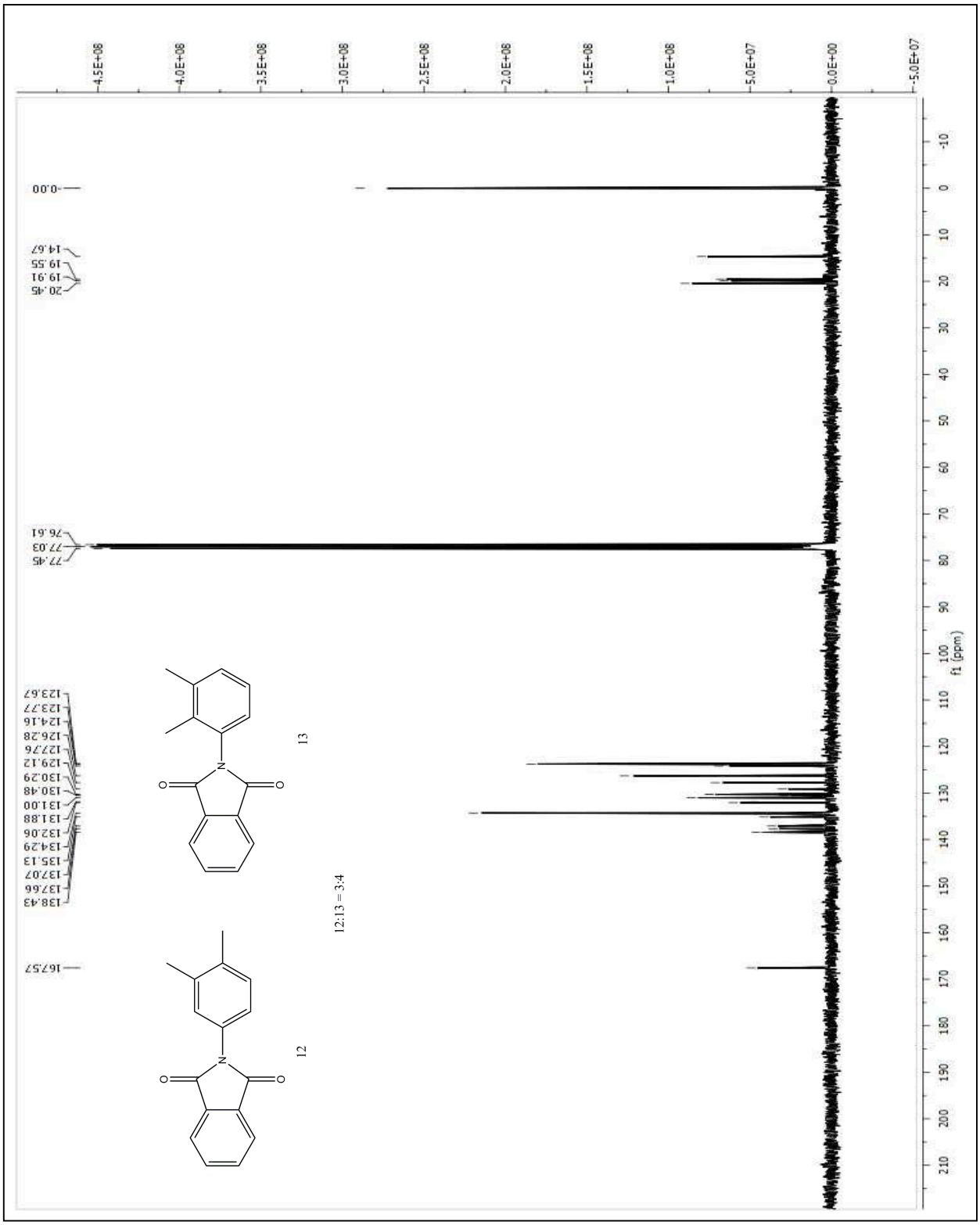
^{1}H NMR (300 MHz, CDCl$_3$): δ = 2.08 (s, 3H), 2.31 (s, 5H), 2.35 (s, 3H), 7.23 – 7.28 (m, 7H), 7.76 – 7.81 (m, 4H), 7.93-7.98 (m, 4H).

^{13}C NMR (75 MHz, CDCl$_3$): δ = 14.67, 19.55, 19.91, 20.45, 123.67, 123.77, 124.16, 126.28, 127.76, 129.12, 130.29, 130.48, 131.00, 131.88, 132.06, 134.29, 135.13, 137.07, 137.66, 138.43, 167.57.

LRMS EI (m/z): [M+] calc'd for $C_{14}H_9NO_2$ 251.09, observed 251.10 m/z.

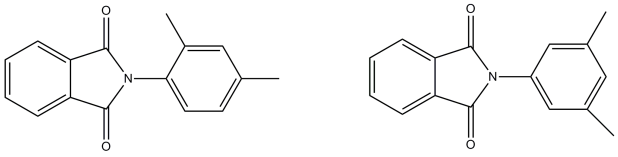


Spectra 23. ^{1}H NMR of Compound **12** and **13**



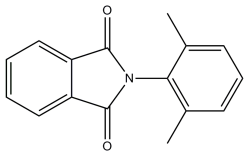
Spectra 24. ^{13}C NMR of Compound **12** and **13**

Synthesis of 17, 18, 19



17

18



19

17:18:19 = 4:3:4

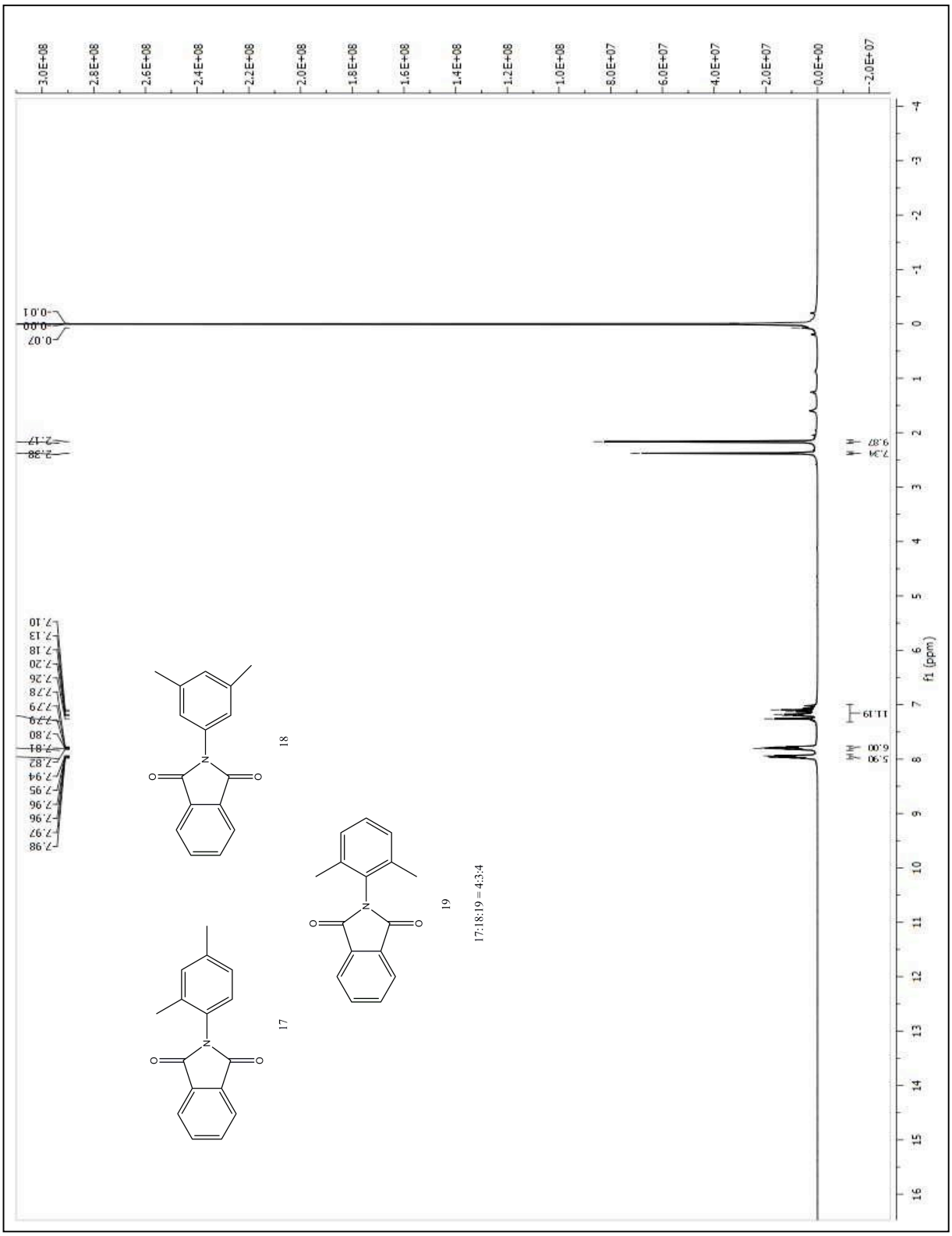
A magnetically stirred solution of phthalimide (0.10 g, 0.68mmol), iodobenzene diacetate (0.55 g, 1.7mmol) in 4 mL of *m*-xylene was microwave heated at 145 ^{0}C for 3 h. The excess solvent from the mixture is removed at reduced pressure and crude product was purified by column chromatography to give a mixture of **17**, **18** and **19** (0.128 g, 75%, **17**:**18**:**19** = 4:3:4).

***R*$_f$ -Value:** Hexane/Ethyl acetate (9:1 *v/v*) = 0.1666.

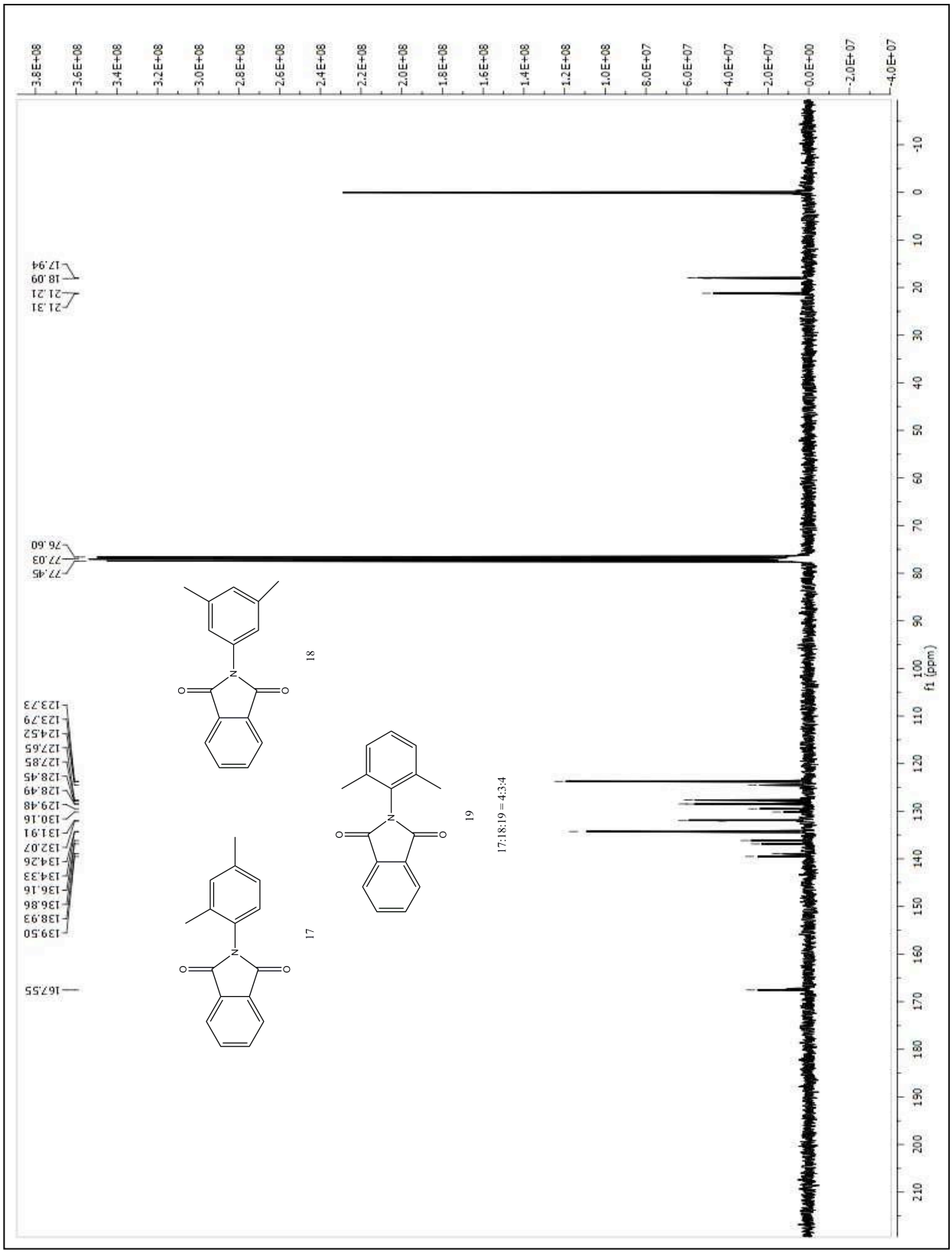
^{1}H NMR (300 MHz, CDCl$_3$): δ = 2.17 (s, 9H), 2.38 (s, 7H), 7.32 – 6.99 (m, 11H), 7.76 – 7.84 (m, 6H), 7.92 – 8.00 (m, 6H).

^{13}C NMR (75 MHz, CDCl$_3$): δ = 17.94, 18.09, 21.21, 21.31, 123.73, 123.79, 124.52, 127.65, 127.85, 128.45, 128.49, 129.48, 130.16, 131.91, 132.07, 134.26, 134.33, 136.16, 136.86, 138.93, 139.50, 167.55.

LRMS EI (m/z): [M+] calc'd for $C_{14}H_9NO_2$ 251.09, observed 251.10 m/z.

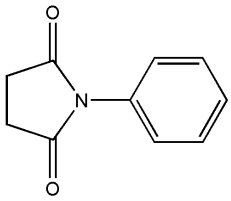


Spectra 25. ^{1}H NMR of Compound **17**, **18**, **19**



Spectra 26. ^{13}C NMR of Compound **17**, **18**, **19**

Synthesis of 1-phenylpyrrolidine-2,5-dione (20)



20

A magnetically stirred solution of succinimide (0.10 g, 1.009 mmol), iodobenzene diacetate (0.81 g, 2.522 mmol), in 4 mL of benzene was microwave heated at 145 ^{0}C for 3 h. The excess solvent from the mixture is removed at reduced pressure and the crude product was purified by column chromatography to give pure **20** (0.0982 g, 83 %). The NMR spectra matched with that of previously published.[1]

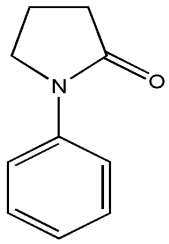
***R_f* -Value:** Hexane/Ethyl acetate (1:1 *v/v*) = 0.44.

^{1}H NMR (400 MHz, $CDCl_3$): δ = 2.9 (s, 4H), 7.28 (d, *J* = 7.2 Hz, 2H), 7.39-7.4 (m,1H), 7.49-7.5 (m,2H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 28.4, 126.4, 128.6, 129.2, 131.8, 176.2.

LRMS EI (m/z): [M+] calc'd for $C_{10}H_9NO_2$ 175.06, observed 175.10 m/z.

Synthesis of 1-phenylpyrrolidin-2-one (21)



21

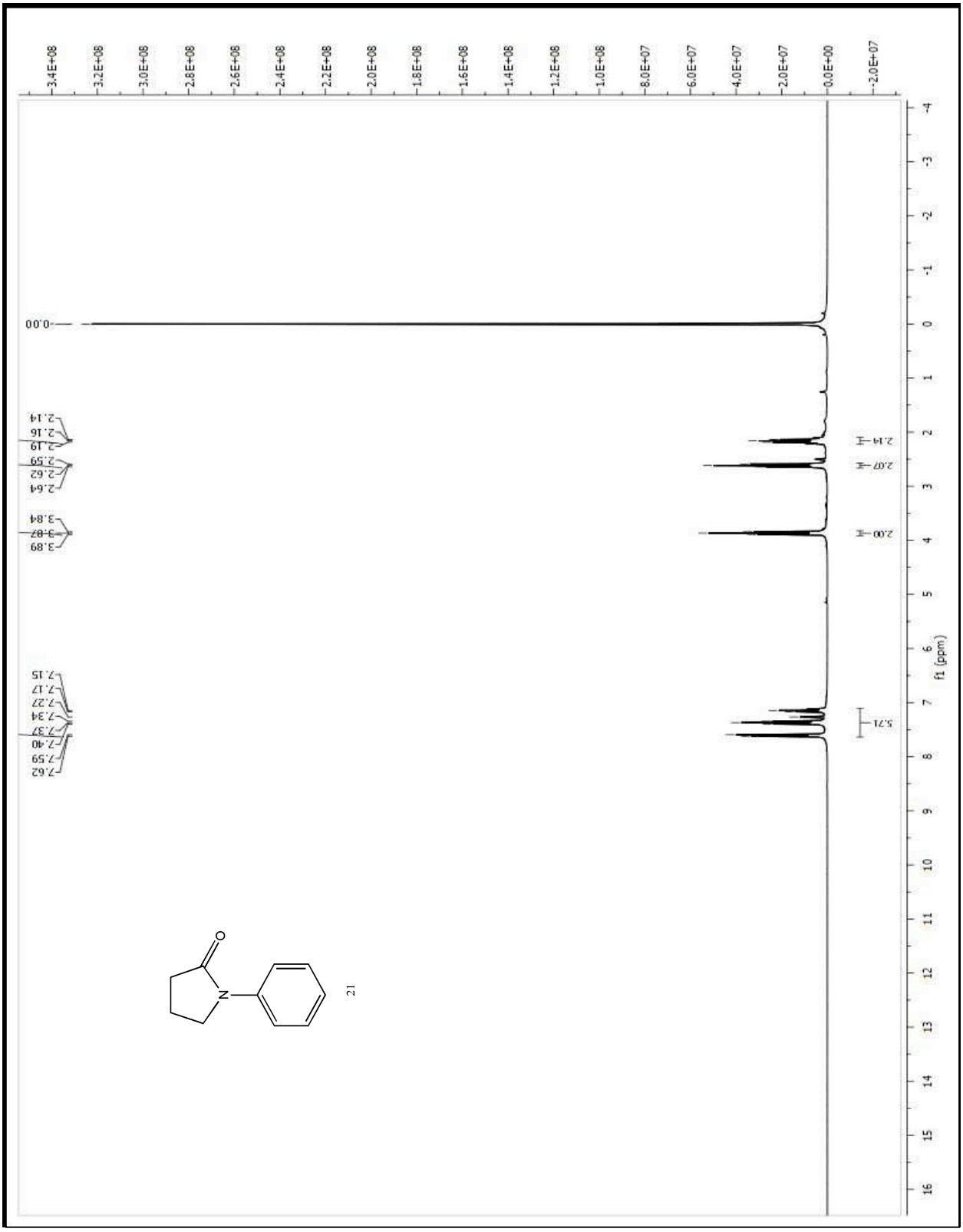
A magnetically stirred solution of 2-pyrolidinone (0.10 g, 1.1749 mmol), (Diacetoxyiodo) benzene (0.95 g, 2.937 mmol), in 4 mL of benzene was microwave heated at 145 ^{0}C for 3h. The excess solvent from the mixture is removed at reduced pressure and the crude product was purified by column chromatography to give pure **21** 0.0303 g (16 %)

***R*$_f$ -Value:** Hexane/Ethyl acetate (1:1 *v/v*) = 0.45.

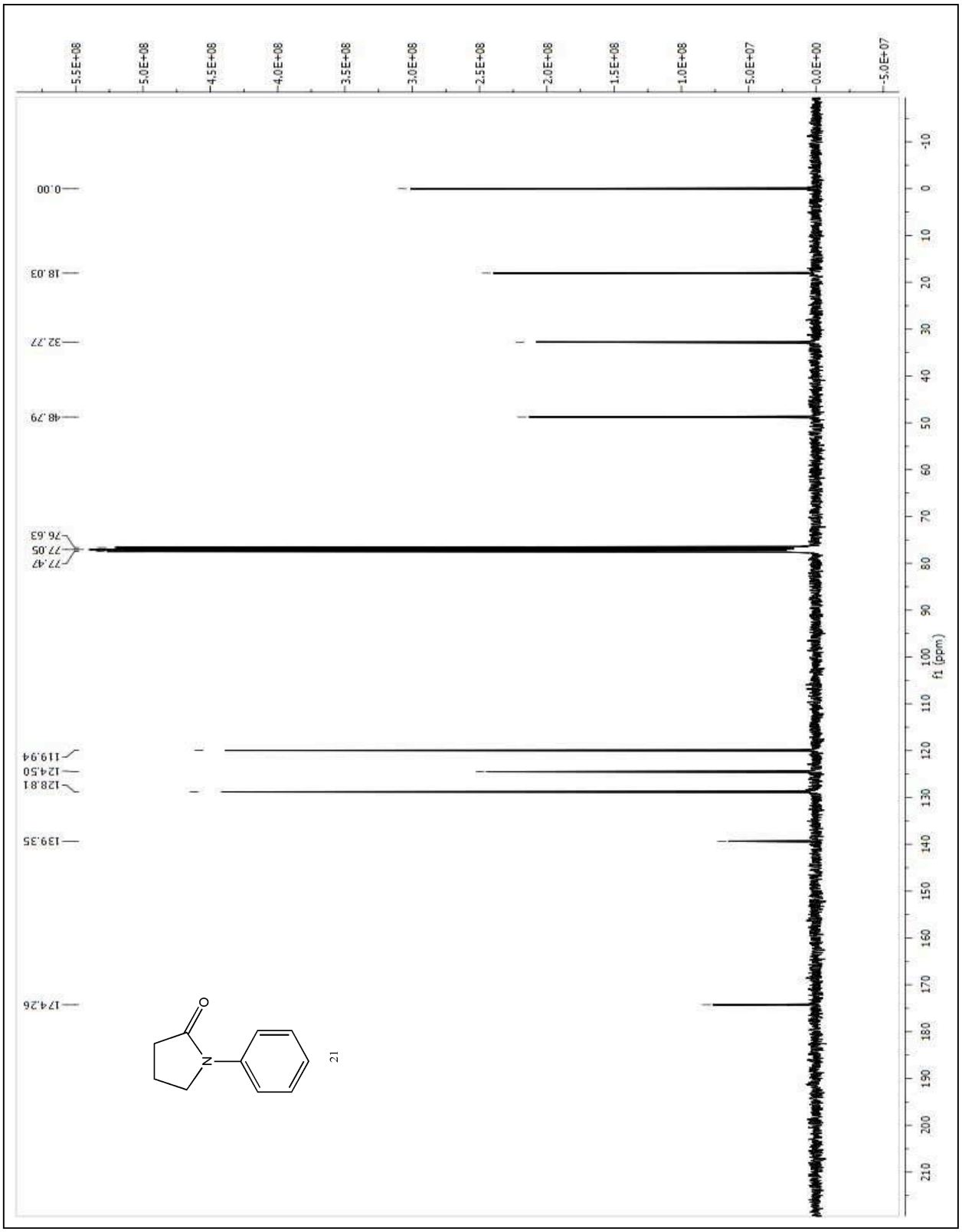
^{1}H NMR (300 MHz, CDCl$_3$): δ = 2.16 (m, 2H), 2.61 (t, *J* = 15 Hz, 2H), 3.87 (t, *J* = 15 Hz, 2H), 7.15-7.62 (m, 5H).

^{13}C NMR (75 MHz, CDCl$_3$): δ = 18.03, 32.77, 48.79, 119.94, 124.50, 128.81, 139.35, 174.26.

LRMS EI (m/z): [M+] calc'd for $C_{10}H_{11}NO_2$, 161.08, observed 161.10 m/z.

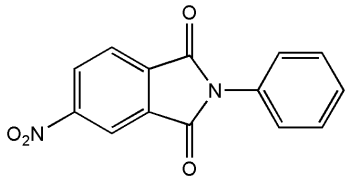


Spectra 27. ^{1}H NMR of Compound **21**



Spectra 28. ^{13}C NMR of Compound **21**

Synthesis of 5-nitro-2-phenylisoindoline-1, 3-dione (22)



22

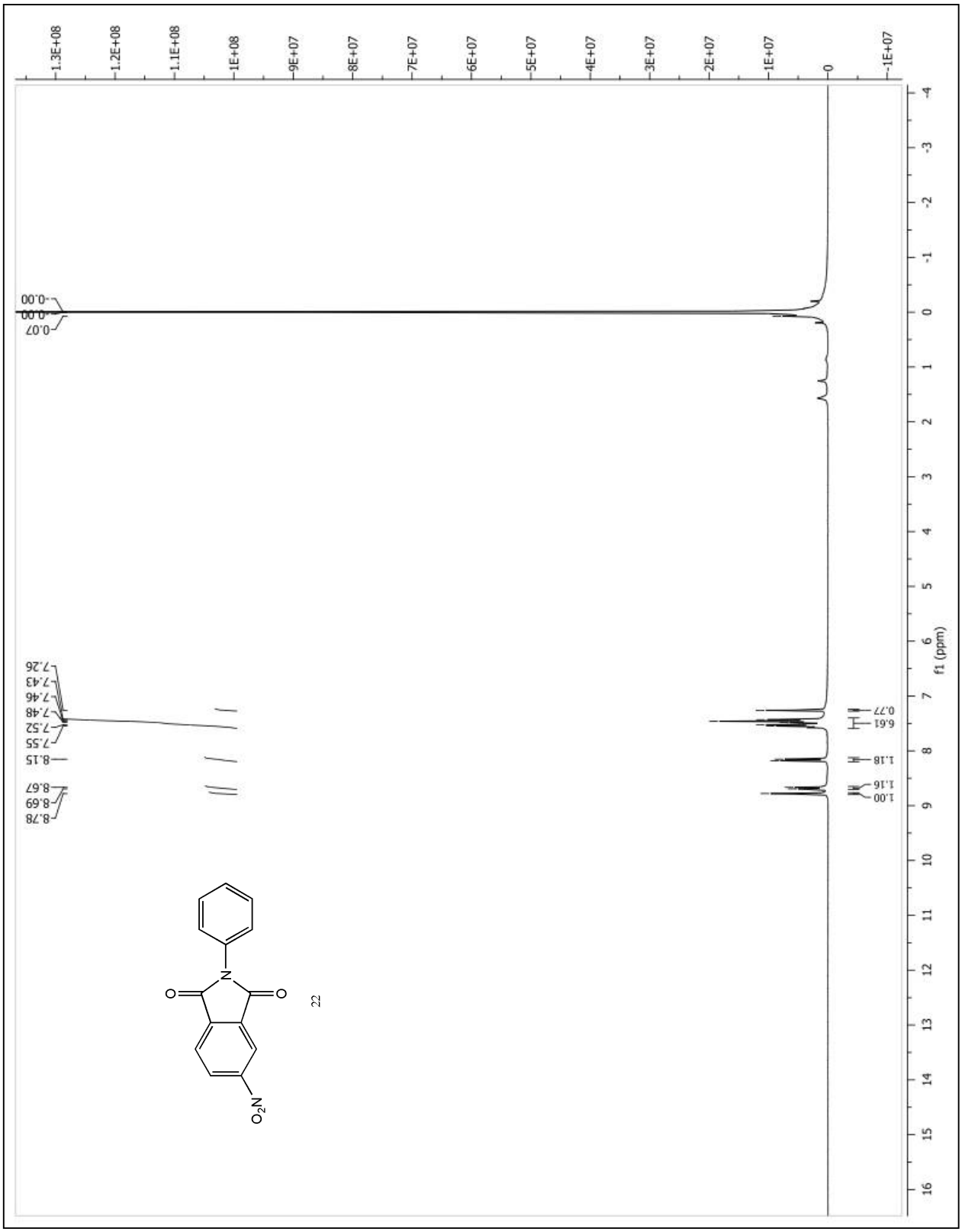
A magnetically stirred solution of 5-nitroisoindoline-1,3-dione (0.10 g, 0..5220 mmol), iodobenzene diacetate (0.42 g, 1.30 mmol), in 4 mL of benzene was microwave heated at 145 ^{0}C for 3h. The excess solvent from the mixture is removed at reduced pressure and the crude product was purified by column chromatography to give pure **22** (0.0557 g, 40 %).

***R_f*-Value:** Hexane/Ethyl acetate (8:2 *v/v*) = 0.33.

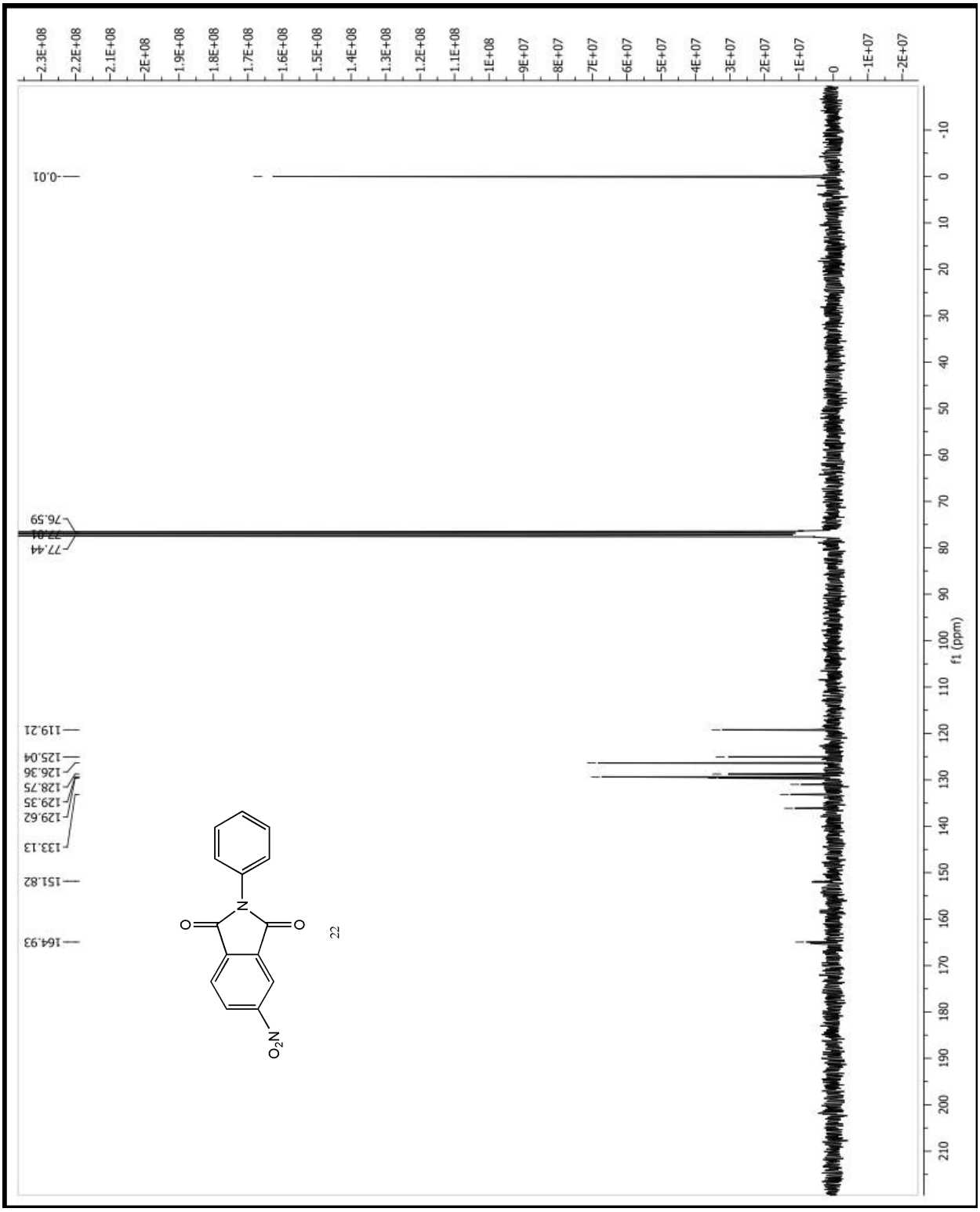
^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.26-7.55 (m, 5H), 8.17 (d, *J* = 9Hz, 1H), 8.68 (d, *J* = 6Hz, 1H), 8.78 (s, 1H).

^{13}C NMR (75 MHz, $CDCl_3$): δ= 119.21, 125.04, 126.36, 128.75, 129.35, 129.62, 133.13, 151.82, 164.93.

LRMS EI (m/z): [M+] calc'd for $C_{14}H_8N_2O_4$ 268.05, observed 268.0 m/z.

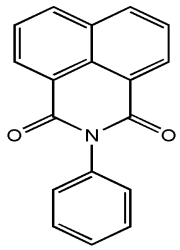


Spectra 29. ^{1}H NMR of Compound **22**



Spectra 30. ^{13}C NMR of Compound **22**

Synthesis of N-phenyl-1,8-naphthalimide (23)



23

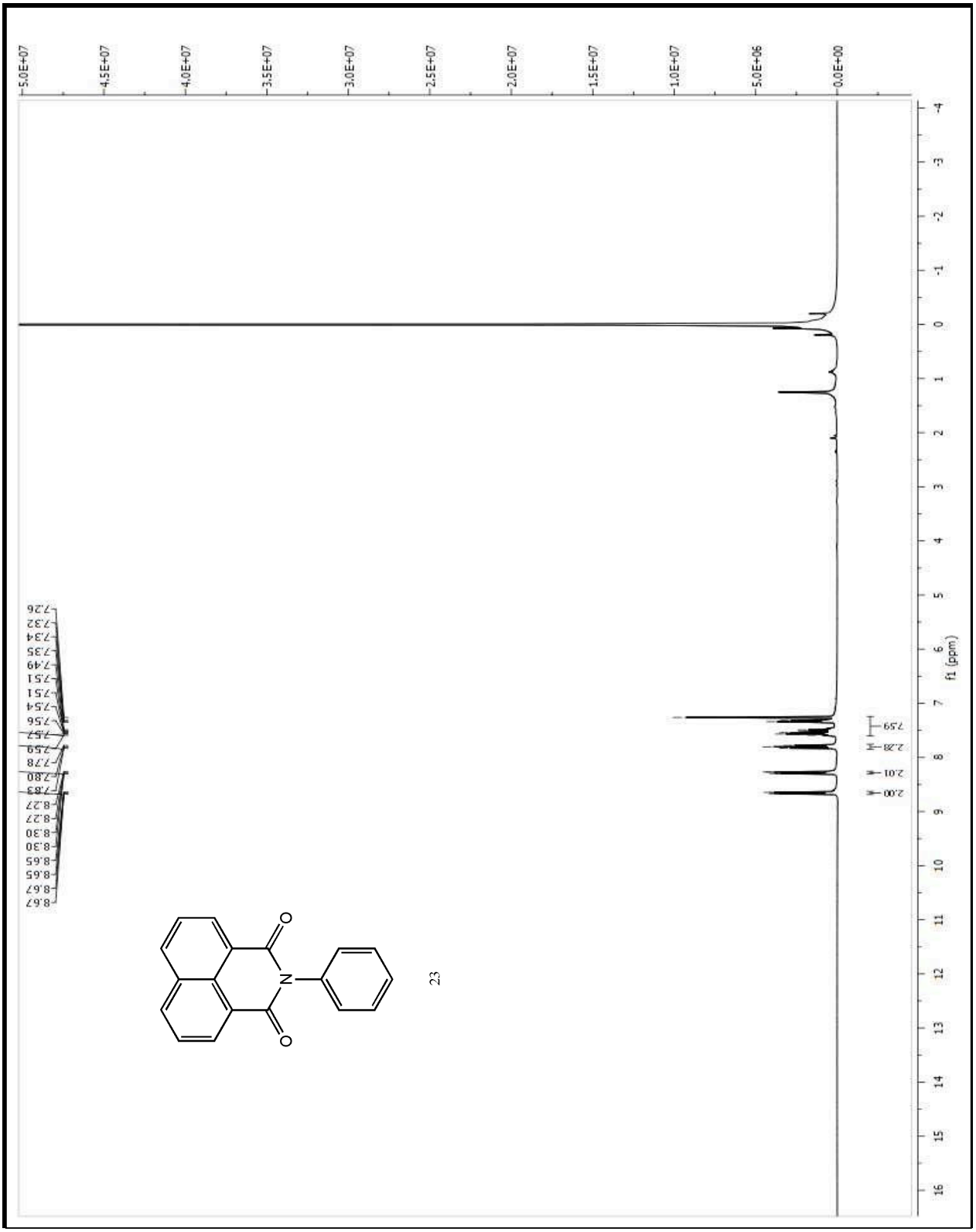
A magnetically stirred solution of 1,8- naphthalimide (0.10 g, 0.507 mmol), iodobenzene diacetate (0.41 g, 1.267 mmol), in 4 mL of benzene was microwave heated at 145 ^{0}C for 3 h. The excess solvent from the mixture is removed at reduced pressure and the crude product was purified by column chromatography to give pure **23** (0.0356 g, 27 %).

***R_f* -Value:** Hexane/Ethyl acetate (7:3 *v/v*) = 0.34.

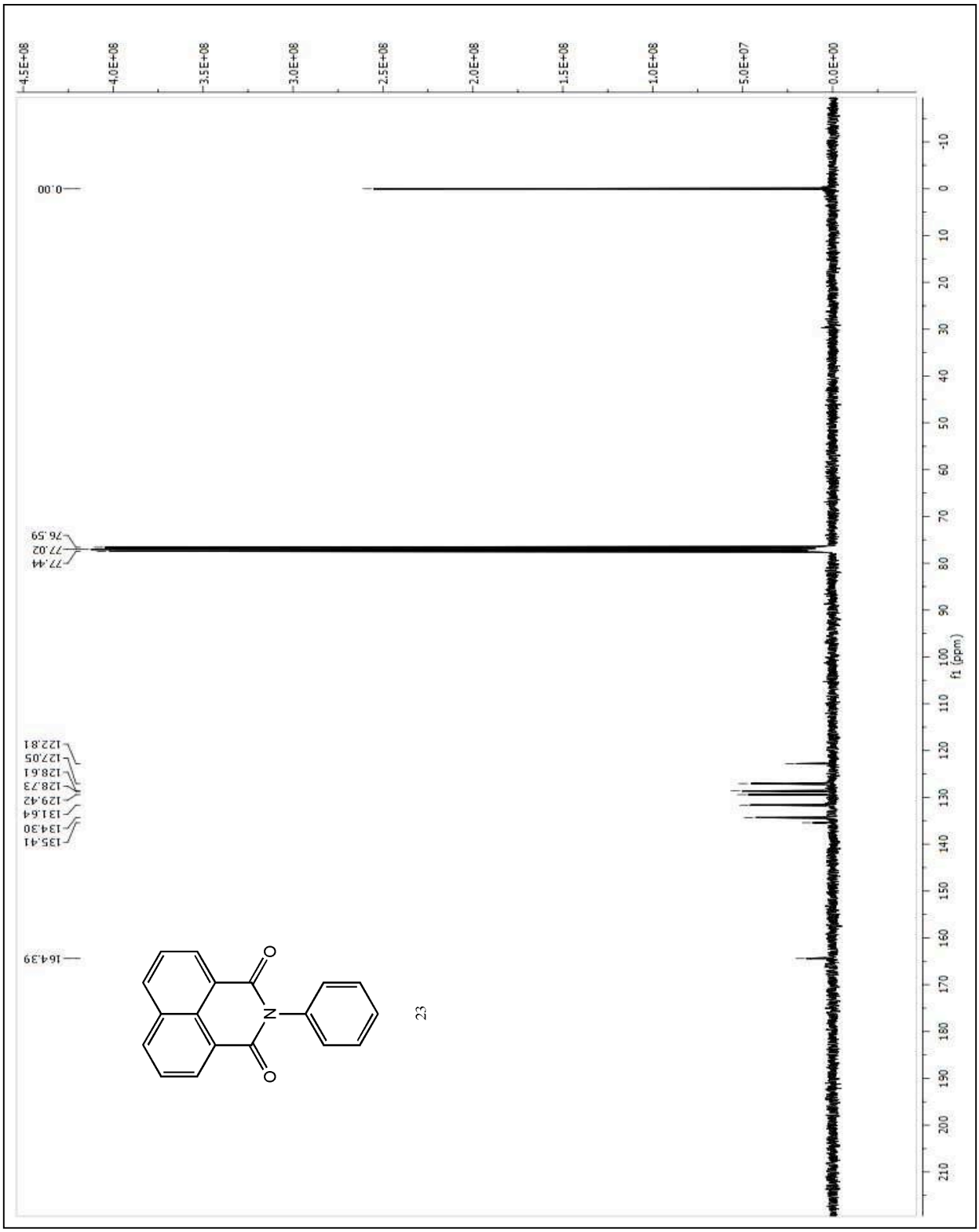
^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.32-7.59 (m, 5H), 7.78-7.83 (m, 2H), 8.28 (d, *J* = 9 Hz, 2H), 8.66 (d, *J* = 6 Hz, 2H).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 122.81, 127.05, 128.61, 128.73, 129.42, 131.64, 134.30, 135.41, 164.39.

LRMS EI (m/z): [M+] calc'd for $C_{18}H_{11}NO_2$, 273.08, observed 273.10 m/z.

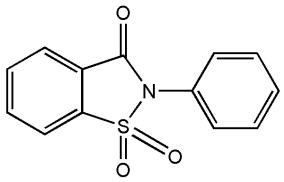


Spectra 31. ^{1}H NMR of Compound **23**



Spectra 32. ^{13}C NMR of Compound **23**

Synthesis of N-phenyl-1, 1-Dioxo-1, 2-benzothiazol-3-one (24)



24

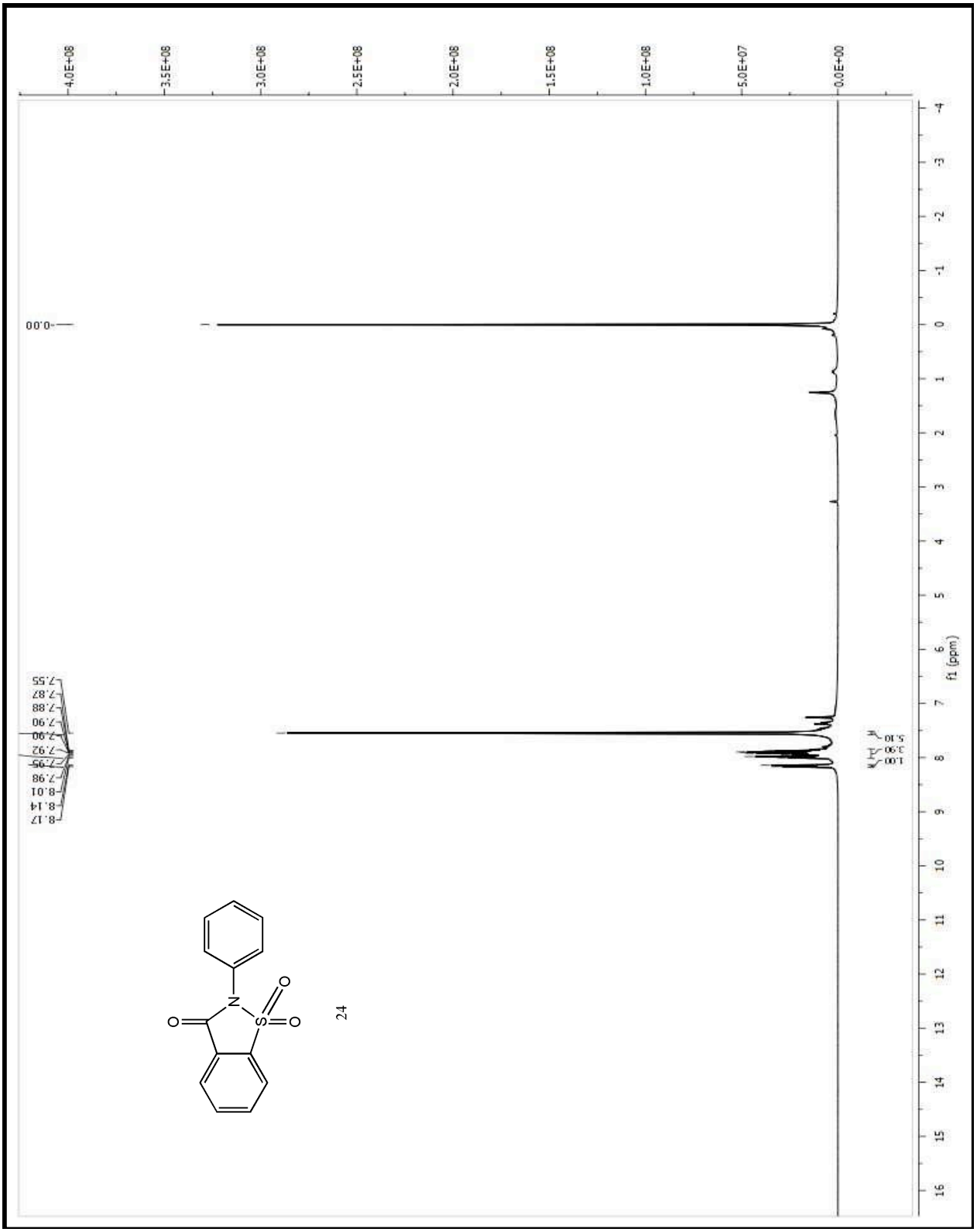
A magnetically stirred solution of 1,1-dioxo-1,2-benzothiazol-3-one (0.10 g, 0.546 mmol), iodobenzene diacetate (0.44 g, 1.365 mmol), in 4 mL of benzene was microwave heated at 145 ^{0}C for 3 h. The excess solvent from the mixture is removed at reduced pressure and the crude product was purified by column chromatography to give pure **24 (**0.037 g, 26 %).

***R_f*-Value:** Hexane/Ethyl acetate (7:3 *v/v*) = 0.26.

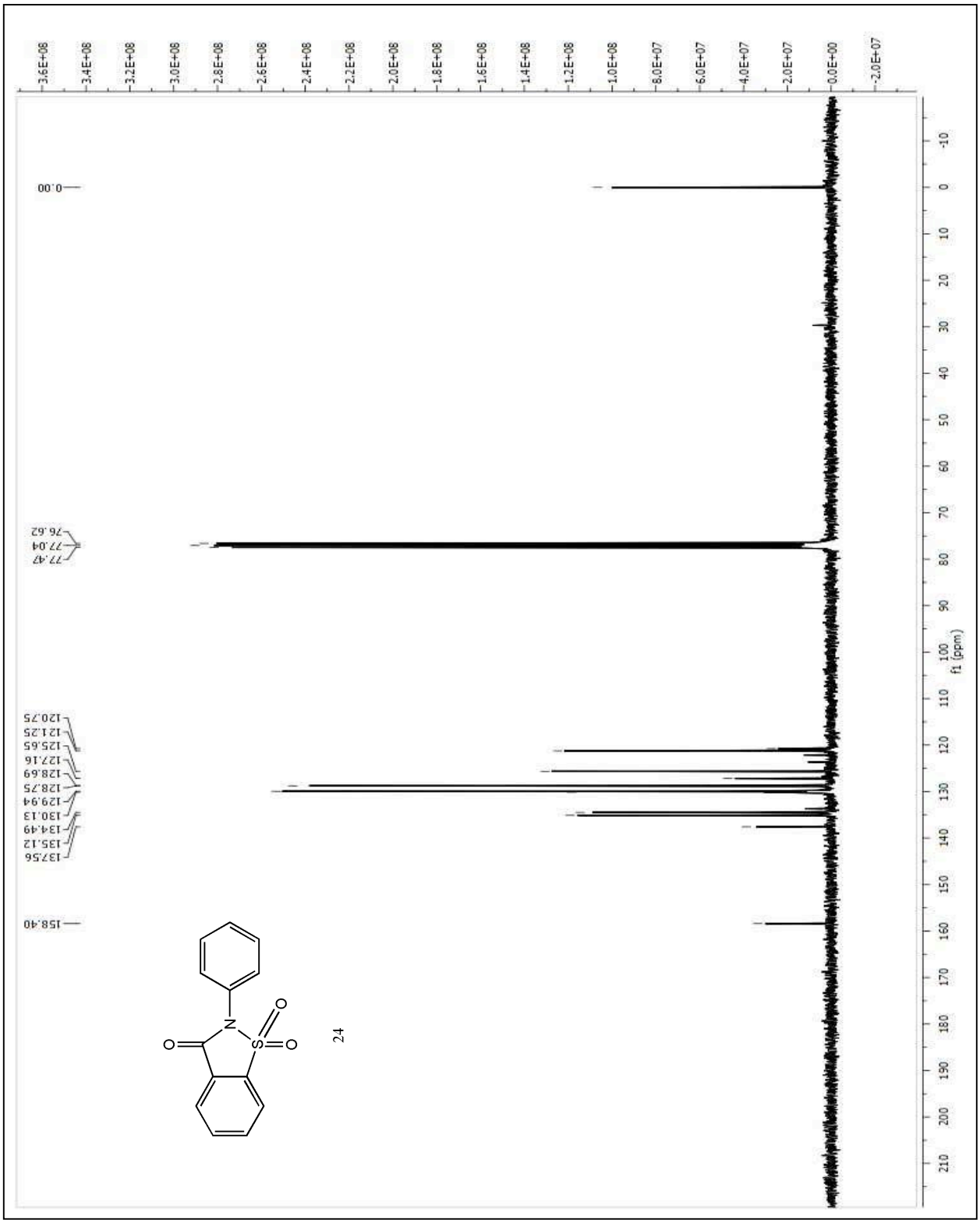
^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.55 (s, 5H), 7.87-8.01(m, 3H), 8.15 (d, *J* = 9 Hz, 1H).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 120.75, 121.25, 125.65, 127.16, 128.69, 128.75, 129.94, 130.13, 134.49, 135.12, 137.56, 158.40.

LRMS EI (m/z): [M+] calc'd for $C_{13}H_9NO_3S$, 259.03, observed 259.0 m/z.



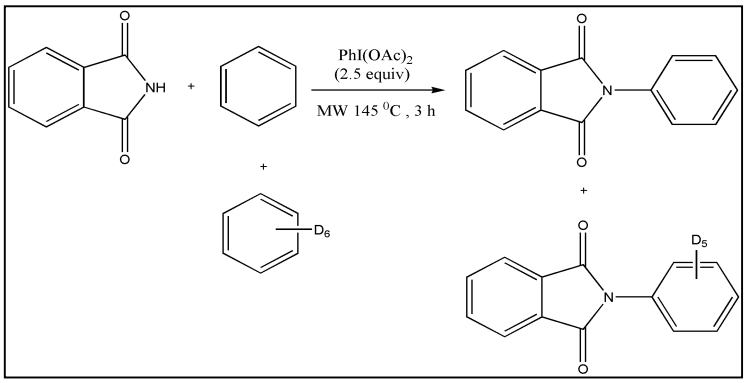
Spectra 33. ^{1}H NMR of Compound **24**



Spectra 34. ^{13}C NMR of Compound **24**

KIE experiments

1) Benzene and benzene-d_6

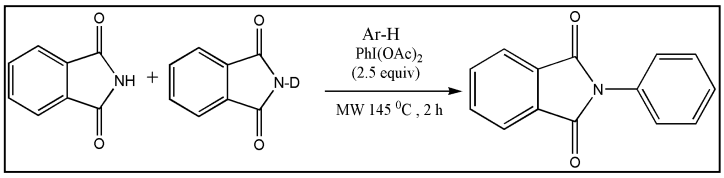


A magnetically stirred solution of phthalimide (0.10 g, 0.68mmol), iodobenzene diacetate (0.55 g, 1.7mmol) with equimolar amounts of benzene (2 mL, 22.5 mmol) and benzene-d_6 (2 mL, 22.5 mmol) was microwave heated at 145 ^{0}C for 3 h. The reaction was then cooled, and an aliquot was removed and analyzed by GC/MS.

GC/MS Conditions: J & W Scientific DB-1, capillary 25.0m x 200µm x 0.33µm, 1.3 mL/min, 40 ^{o}C, hold 0.50min, 12 ^{o}C/min to 320 ^{o}C, hold 6.0min.

$\mathbf{k_H/k_D}$ = 1.03

2) Phthalimide and phthalimide-*d*



A magnetically stirred equimolar mixture of phthalimide (0.05 g, 0.339 mmol) and phthalimide -*d* (0.05 g, 0.339 mmol), iodobenzene diacetate (0.27 g, 0.849 mmol) with 4 mL of benzene was microwave heated at 145 ^{0}C for 2 h. The reaction was then cooled, and an aliquot was removed and analyzed by GC/MS.

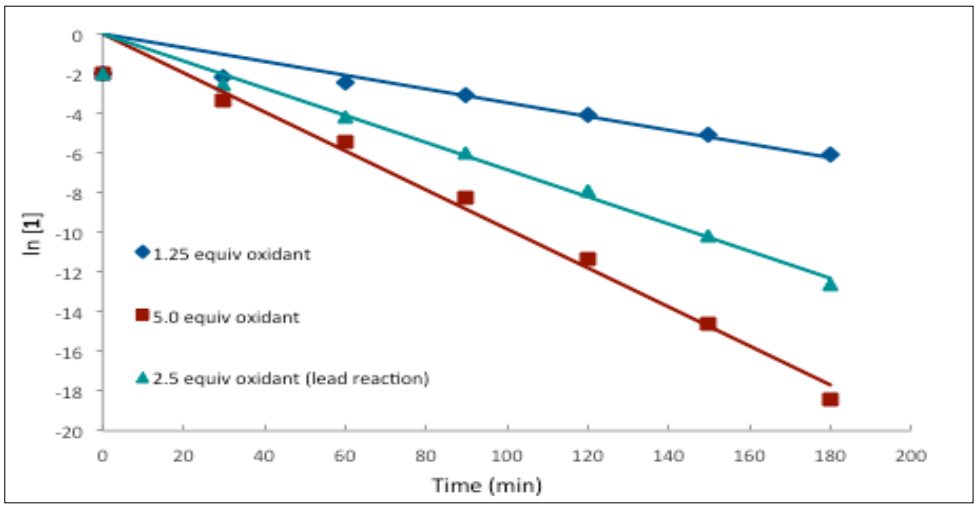
GC/MS Conditions: J & W Scientific DB-1, capillary 25.0 m x 200 µm x 0.33 µm, 1.3 mL/min, 40 ^{o}C, hold 0.50 min, 12 ^{o}C/min to 320 ^{o}C, hold 6.0 min.

$\mathbf{k_H/k_D}$ = 0.98

Kinetic experiments

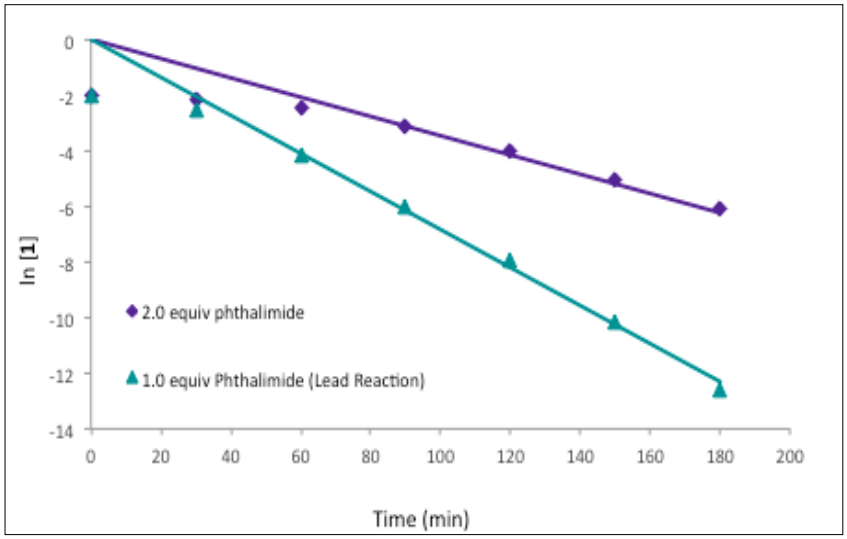
1) Order in oxidant

The order in the oxidant iodobenzene diacetate was determined by studying the conversion of starting material (**1**) with three different iodobenzene diacetate concentrations. A magnetically stirred solution of phathimide (0.10 g, 0.68 mmol), iodobenzene diacetate (0.85 mmol, 1.7 mmol or 3.4 mmol) in 4 mL of benzene was microwave heated at 145 ^{0}C for 3 h. The reaction was monitored over a time interval of 30 min. The conversion of starting material (**1**) from time t = 0 min to time t =180 min was calculated by GC-MS using dodecane as an internal standard. A log plot of concentration of (**1**) versus time gave a straight line indicating the first order dependence on oxidant.

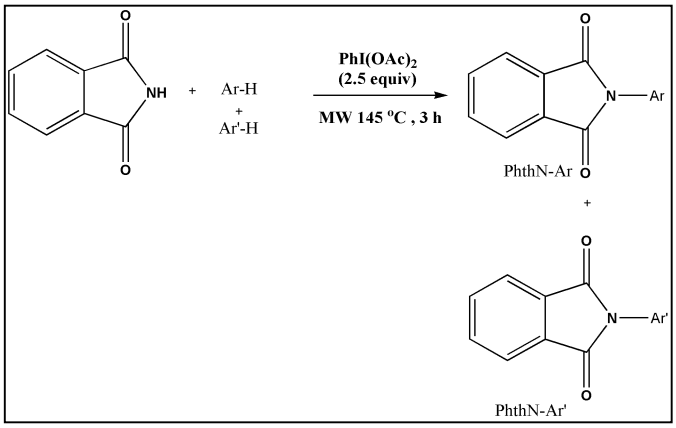


2) Order in Substrate

The order in the substrate (**1**) was determined by studying the conversion of starting material (**1**) with varying amounts of (diacetoxyiodo)benzene. A magnetically stirred solution of phthalimide (0.68 mmol or 1.4 mmol), (diacetoxyiodo)dbenzene (1.7mmol) in 4 mL of benzene was microwave heated at 145 ^{0}C for 3 h. The reaction was monitored over a time interval of 30 min. The conversion of starting material (**1**) from time t = 0 min to time t =180 min was calculated by GC/MS using dodecane as an internal standard. A log plot of concentration of **1** versus time gave a straight line indicating the first order dependence on substrate **1**.



Competition experiment

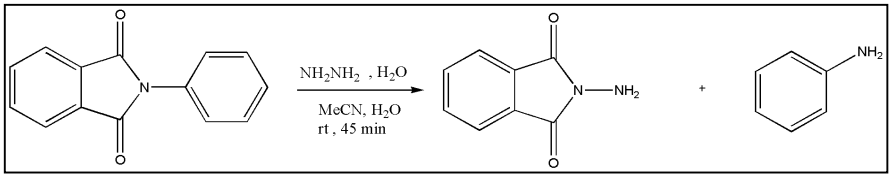


A magnetically stirred solution of phthalimide (0.10 g, 0.68mmol), iodobenzene diacetate (0.55 g, 1.7 mmol) with equimolar amounts of Ar-H (2.0 mL) and Ar'-H (2.0 mL,) was microwave heated at 145 ^{0}C for 3 h. The reaction was then cooled, and an aliquot was removed and analyzed by GC/MS.

Entry	Ar-H	Ar'-H	PhthN-Ar	PhthN-Ar'
1	*p*-xylene	benzene	71%	29%
2	*p*-xylene	*p*-difluoro-benzene	96%	4%
3	benzene	*p*-difluoro-benzene	86%	14%

The ratios of the products were determined by GC/MS.

Phthalimide Deprotection



N-Hydrazine hydrate (0.15 g, 4.5 mmol) and 3 mL of water was added to a solution of *N*-phenyl phthalimide (0.10 g, 0.45 mmol) in 5 mL acetonitrile. The mixture was stirred for 45 min at room temperature until TLC shows the complete conversion of *N*-phenylphthalimide. An aliquot was removed from the reaction mixture and analyzed by GC/MS.[3]

References

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