Iron-Catalyzed Homo-Coupling of Simple and Functionalized Arylmagnesium Reagents

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Supporting Information

General procedures

Tetrahydrofuran (THF) was Purchased from Merck and freshly distilled from sodium/benzophenone under nitrogen before use.

Iron(III) chloride (anhydrous, 98%) was Purchased from Acros. It is used as a solution (0.1M) in THF stored under nitrogen atmosphere.

Other reagents were purchased from Acros, Aldrich and Avocado and used as received.

The Grignard reagents **1a-1g** and **1i-1j** were prepared in THF from the commercially available aryl bromide by the standard method and were titrated according to the procedure published by Watson¹, excepted in the case of 3-pyridylmagnesium bromide **1h**, which was titrated by reaction with isobutyraldehyde. The functionalized aryl Grignard reagents **1k-1q** were prepared as described above in the experimental procedures.

¹H, ¹³C and ¹⁹F NMR spectra were recorded on JEOL EX-270 and JEOL ECX-400 spectrometers. Chemical shifts δ are given in ppm from TMS as an internal reference.

Infrared spectra were recorded (liquids were analysed neat between NaCl plates, solids were analysed in solution in Nujol or as KBr pellets) on a Fourier transform Nicolet Impact 400 spectrometer at room temperature between 4000 and 600 cm⁻¹. Mass spectra were recorded on a Hewlett-Packard HP 5973 mass spectrometer.

Melting points were measured on a Büchi 530 apparatus and are given without correction.

Homo-coupling of aryl Grignard reagents 1a-1j (Table 1 and 2)

A dried four-necked flask equipped with a magnetic stirrer and a thermometer was charged under nitrogen with THF (20 mL), a solution of FeCl₃ in THF (0.1M, 3.0 mL, 0.3 mmol) and 0.6 equivalent of 1,2-dihalogenoethane (6 mmol). A solution of the aryl Grignard reagent **1** in THF (*c.a.* 1.0M, 10 mmol) was added at once *via* a syringe to the resulting yellow-green solution. The colour immediately changed to dark brown and the temperature increased. The resulting mixture was stirred at room temperature for 10 min then hydrolyzed with a 1M aqueous HCl solution (30 mL). After extraction with methylene chloride (3x50 mL), the combined organic layers were dried over anhydrous magnesium sulphate and concentrated under reduced pressure. The residue was purified by flash chromatography on silica gel to give the expected biaryl compound.

Homo-coupling of functionalized aryl Grignard reagents (1k-1o) (Table 3)

A dried four-necked flask equipped with a magnetic stirrer and a thermometer was charged under nitrogen with THF (20 mL) and 10 mmol of iodobenzonitrile (entries 1-3) or ethyl iodobenzoate (entries 4-5). Isopropylmagnesium bromide was added (1 M in THF, 11 mmol) at -40°C to the resulting solution. After stirring at -40°C for 30 min, a solution of FeCl₃ in THF (0.1M, 3.0 mL, 0.3 mmol,) and 1,2-dihalogenoethane (10 mmol) were added simultaneously and the resulting mixture was stirred at -40°C for 2 h (entries 1-3) or at room temperature for 1 h (entries 4-5) then hydrolyzed with a 1 M aqueous HCl solution (30 mL). After extraction with methylene chloride (3x50 mL), the combined organic layers were dried over anhydrous magnesium sulphate and

concentrated under reduced pressure. The residue was purified by flash chromatography on silica gel to give the expected biaryl compound.

Homo-coupling of 2-nitrophenylmagnesium bromide **1p** (Scheme 1)

A dried four-necked flask equipped with a magnetic stirrer and a thermometer was charged under nitrogen with THF (30 mL) and 2-iodonitrobenzene (2.49 g, 10 mmol). Phenylmagnesium bromide was added (1.0M in THF, 10.5 mmol) at -40°C to the resulting solution. After stirring at -40°C for 5 min, a solution of FeCl₃ in THF (0.1M, 3.0 mL, 0.3 mmol) and 1,2-dibromoethane (1.13 g, 10 mmol) were added simultaneously and the resulting mixture was stirred at room temperature for 30 min then hydrolyzed with a 10% aqueous NH₄Cl solution (80 mL). After extraction with ethyl acetate (3x50 mL), the combined organic layers were dried over anhydrous magnesium sulphate and concentrated under reduced pressure. The residue was purified by chromatography on silica gel (eluent: cyclohexane-AcOEt 70:30) to give 505 mg of **2p** (Yield: 41%).

Homo-coupling of 2-fluoro-3-carbethoxy-4-pyridylmagnesium bromide **1q** (Scheme 2)

*Preparation of 2-fluoro-3-iodopyridine **12***

In a dried four-necked flask equipped with a magnetic stirrer, a thermometer and an addition dropping funnel, LDA was prepared by stirring THF (250 mL), isopropylamine (14 mL, 0.1 mol) and *n*-butyllithium (1.60M in hexane, 62.5 mL, 0.1 mol) at 0°C for 15 min. The resulting mixture was cooled to -80°C and a solution of 2-fluoropyridine (9.71 g, 0.10 mol) in THF (80 mL) was added dropwise at -75°C. After stirring at -75°C for 4 h, zinc chloride (20.4 g, 0.15 mol) was added and the temperature was allowed to warm to 0°C. Iodine was then added and the resulting mixture was stirred at room temperature for 1 h then hydrolyzed with a 10% aqueous NaHSO₃ solution (100 mL). After extraction with ethyl ether (3x100 mL), the combined organic layers were washed with a 10% aqueous solution of NaHSO₃ (2x100 mL) then with brine (100 mL), dried over anhydrous magnesium sulphate and concentrated under reduced pressure. Crude material was purified by chromatography on silica gel (eluent : cyclohexane-AcOEt 90:10) to give 9 g of **12** (Yield: 40%).

*Preparation of 2-fluoro-3-iodo-4-carbethoxypyridine **3q***

In a dried four-necked flask equipped with a magnetic stirrer, a thermometer and an addition dropping funnel, LDA was prepared by stirring THF (130 mL), isopropylamine (5.6 mL, 40 mmol) and *n*-butyllithium (1.6 M in hexane, 25.0 mL, 40 mmol) at 0°C for 15 min. The resulting mixture was cooled at -80°C and a solution of 2-fluoro-3-iodopyridine (**12**) (8.92 g, 40 mmol) in THF (60 mL) was added dropwise at -75°C. After stirring at -75°C for 1 h, a solution of magnesium bromide in THF prepared by reacting 1,2-dibromoethane (9 g, 48 mmol) with magnesium turnings (1.22 g, 50 mmol) in THF (120 mL) was added. The resulting solution was stirred at -75°C for 15 min then ethyl cyanoformate (5.5 mL, 56 mmol) was added and stirring was continued at room temperature for 1 h. The reaction mixture was then hydrolyzed by brine (100 mL). After extraction with ethyl acetate (3x100 mL), the combined organic layers were dried over MgSO₄ and concentrated under reduced pressure. Crude material was purified by chromatography on silica gel (eluent: cyclohexane-AcOEt 80:20) to give 7.82 g of **3q** (Yield : 66%).

*Homo-coupling of 2-fluoro-3-carbethoxy-4-pyridylmagnesium bromide **1q** (Scheme 2)*

A dried four-necked flask equipped with a magnetic stirrer and a thermometer was charged under nitrogen with THF (20 mL) and 2-fluoro-3-carbethoxy-4-iodopyridine **3q** (2.95 g, 10 mmol). The resulting solution was cooled to -40°C then a solution of isopropylmagnesium bromide in THF (c.a. 1 M, 11 mmol) was added at -40°C. After stirring at -40°C for 30 min, a solution of FeCl₃ in THF (0.1M, 3 mL, 0.3 mmol) and 1,2-diiodoethane (1.69 g, 6 mmol) were added simultaneously and the resulting mixture was stirred at room temperature for 1 h then hydrolyzed with brine (50 mL). After

extraction with chloroform (3x50 mL), the combined organic layers were washed with a 10% aqueous NaHSO₃ solution (50 mL), dried over anhydrous magnesium sulphate and concentrated under reduced pressure. The residue was purified by chromatography on silica gel (eluent: cyclohexane-AcOEt 70:30), to give 500 mg of **2q** (Yield: 33%).

Intramolecular coupling reaction : preparation of 1,9-dihydrophenanthrene 6 (Scheme 3)

Preparation of 2-bromo(bromomethyl)benzene 13

A flask equipped with a reflux condenser was charged with 2-bromotoluene (51.3 g, 0.3 mol), benzoyl peroxide (75%, 19.4 g, 0.06 mol), carbon tetrachloride (300 mL) and *N*-bromosuccinimide (53.4 g, 0.3 mol). The resulting mixture was refluxed for 2 h then cooled, washed with a 10% aqueous NaHCO₃ 10% solution then with brine (100 mL), dried over anhydrous magnesium sulphate and concentrated under reduced pressure. Crude material was distilled under reduced pressure (b.p. 74°C/0.30 Torr) to give 47 g of **13** (Yield : 63%).

Preparation of 1,2-bis(2-bromophenyl)ethane 14

A dried four-necked flask equipped with a magnetic stirrer, a thermometer and a reflux condenser was charged under nitrogen with THF (100 mL) and magnesium turnings (1.82 g, 72 mmol). A solution of 2-bromo(bromomethyl)benzene **13** (31.6 g, 120 mmol) in THF (40 mL) was added dropwise under stirring (exothermic) then the resulting suspension was stirred at room temperature for 2 h. The reaction mixture was then hydrolyzed with a 1 M HCl solution (150 mL). After extraction with ethyl ether (3x100 mL), the combined organic layers were dried over anhydrous magnesium sulphate and concentrated under reduced pressure. Crude material was distilled under reduced pressure to eliminate the solvents and the resulting solid was then washed with pentane to give 6.45 g of **14** (Yield: 32%).

Preparation of 1,2-bis(2-iodophenyl)ethane 4

A dried four-necked flask equipped with a magnetic stirrer, a thermometer and a addition dropping funnel was charged under nitrogen with 1,2-bis(2-bromophenyl)ethane **14** (8.5 g, 25 mmol) and THF (70 mL). The resulting solution was cooled to -75°C and *n*-butyllithium (1,6M in hexane, 35.5 mL, 55 mmol) was added at -75°C then the mixture was stirred for 15 min. A solution of iodine (25.4 g, 100 mmol) in THF (110 mL) was added at -75°C and the resulting mixture was stirred at room temperature for 1 h then hydrolyzed with a 1 M HCl solution (150 mL). After extraction with methylene chloride (3x70 mL), the combined organic layers were washed with a 10% aqueous NaHSO₃ solution (2x70 mL) then with brine (70 mL) then dried over anhydrous magnesium sulphate and concentrated under reduced pressure. Crude material was purified by flash chromatography on silica gel (eluent: cyclohexane-methylene chloride 99:1), to give 8.4 g of **4** (Yield: 77%).

Intramolecular coupling reaction (Scheme 3)

A dried four-necked flask equipped with a magnetic stirrer and a thermometer was charged under nitrogen with THF (15 mL) and 1,2-bis(2-iodophenyl)ethane **4** (1.40 g, 5 mmol). A solution of isopropylmagnesium bromide in THF (1.0M, 11 mL, 11 mmol,) was added then the resulting solution was stirred at room temperature for 60 min. A solution of FeCl₃ in THF (0.1M, 3mL, 0.3 mmol) and 1,2-dibromoethane (0.94 g, 5 mmol) were added simultaneously. The resulting mixture was stirred at room temperature for 60 min then hydrolyzed with a 1 M HCl solution (50 mL). After extraction with methylene chloride (3x40 mL), the combined organic layers were dried over anhydrous magnesium sulphate and concentrated under reduced pressure. The crude material was purified by chromatography on silica gel (eluent: pentane) to give 0.69 g of **6** (yield: 76%).

Synthesis of *N*-methylocrinasiadine **11** (Schemes 4 and 5)

6-bromopiperonal **7**, 6-iodopiperonal **8** and 6-iodo-3,4-methylenedioxybenzoic acid **15** were prepared in respectively 54%, 80% and 59% yields according to procedures described in the literature.^{15, 16, 21}

Preparation of 2,2'-diiodo-4,5-methylenedioxybenzanilide 16

A dried four-necked flask equipped with a magnetic stirrer and a thermometer was charged under nitrogen with methylene chloride (60 mL), DMF (0.1 mL) and 6-iodo-3,4-methylenedioxybenzoic acid **15** (6.0 g, 20 mmol). Oxalyl chloride (3.7 mL, 40 mmol) was added and, after stirring for 2 h, the resulting solution was concentrated under reduced pressure to give 6.8 g of the corresponding carboxylic acid chloride as a pale yellow solid.

A dried four-necked flask equipped with a magnetic stirrer, a thermometer and an addition dropping funnel was charged under nitrogen with methylene chloride (40 mL), 2-iodoaniline (4.38 g, 20 mmol) and triethylamine (3.1 mL, 22 mmol). The mixture was cooled to 0°C then a solution of the previously prepared acid chloride in methylene chloride (50 mL) was added dropwise at 0°C and stirring was continued for 3 h at room temperature. The reaction mixture was then hydrolyzed with water (100 mL). After extraction with chloroform (3x80 mL), the combined organic layers were dried over anhydrous magnesium sulphate and concentrated under reduced pressure. Crude material was purified by flash chromatography on silica gel (eluent : CH₂Cl₂) to give 2.41 g of **16** (Yield: 77%).

*Preparation of 2,2'-diiodo-*N*-methyl-4,5-methylenedioxybenzanilide 9*

A dried four-necked flask equipped with a magnetic stirrer and a thermometer was charged under nitrogen with THF (30 mL) and sodium hydride (95%, 250 mg, 9.6 mmol). The resulting suspension was cooled to 0°C and a solution of 2,2'-diiodo-4,5-methylenedioxybenzanilide **16** (3.92 g, 8 mmol) in methylene chloride (50 mL) was added dropwise at 0°C. After stirring for 15 min at 0°C, methyl iodide (650 µL, 10.4 mmol) was added. The resulting mixture was stirred at room temperature for 2 h then hydrolyzed with water (60 mL). After extraction with chloroform (3x60 mL), the combined organic layers were dried over anhydrous magnesium sulphate and concentrated under reduced pressure. Crude material was purified by flash chromatography on silica gel (eluent: CH₂Cl₂-AcOEt 97:3) to give 3.47 g of **9** (Yield: 86%).

*Preparation of *N*-methylocrinasiadine 11*

A dried four-necked flask equipped with a magnetic stirrer and a thermometer was charged under nitrogen with THF (26 mL) and 2,2'-diiodo-*N*-methyl-4,5-methylenedioxybenzanilide **9** (2.03 g, 4.0 mmol). The resulting mixture was cooled to -30°C. A solution of isopropylmagnesium bromide in THF (c.a. 1.0M, 8.8 mmol) was added at -25°C. After stirring at -25°C for 30 min, a solution of FeCl₃ in THF (0.1M, 2.4 mL, 0.24 mmol, 6 mol %) and 1,2-dibromoethane (0.75 g, 4 mmol) were added simultaneously. The resulting mixture was stirred at room temperature for 1 h then hydrolyzed with brine (50 mL). After extraction with chloroform (3x60 mL), the combined organic layers were washed with a 10% aqueous NaHSO₃ solution (50 mL), dried over anhydrous magnesium sulphate and concentrated under reduced pressure. Crude material was purified by flash chromatography on silica gel (eluent: cyclohexane-AcOEt 60:40) then recrystallized from ethanol to give 400 mg of *N*-methylocrinasiadine (Yield: 39%).

SPECTROSCOPIC DATA

All the known compounds gave satisfactory spectroscopic values which are given below and are analogue to spectroscopic data reported in the literature. Elemental analysis or HRMS are given for each new compound.

Biphenyl²(2a): white solid; **m.p.** 67-69°C (Lit. 69-71°C); **IR (KBr)** ν = 3031, 1941, 1880, 1573, 1481, 1342, 1173, 1081, 902, 743, 702 cm^{-1} ; **¹H NMR (400 MHz, CDCl₃, 25°C)** δ : 7.34 (1H, t, $J^3(\text{H})=7.3\text{Hz}$), 7.43 (2H, d, $J^3(\text{H})=7.8\text{Hz}$), 7.59 (2H, d, $J^3(\text{H})=7.4\text{Hz}$); **¹³C NMR (100 MHz, CDCl₃, 25°C)** δ : 127.1 (CH), 127.2 (CH), 128.7 (CH), 141.2 (C); **MS (EI, 70eV) m/z (%)** : 154 (100) [M^+], 76 (15) [$\text{M}^+-\text{C}_6\text{H}_6$].

4,4'-Dimethoxybiphenyl²(2b): white solid; **m.p.** 178-179°C (Lit. 179-180°C); **IR (KBr)** ν = 1614, 1506, 1276, 1250, 1178, 1040, 1015, 825, 805 cm^{-1} ; **¹H NMR (400 MHz, CDCl₃, 25°C)** δ : 3.83 (3H, s), 6.95 (2H, dm, $J^3(\text{H})=9.1\text{Hz}$), 7.47 (2H, dm, $J^3(\text{H})=9.2\text{Hz}$); **¹³C NMR (100 MHz, CDCl₃, 25°C)** δ : 55.3 (CH₃), 114.1 (CH), 127.7 (CH), 133.4 (C), 158.7 (C); **MS (EI, 70eV) m/z (%)** : 214 (100) [M^+], 199 (85) [M^+-CH_3], 171 (26), 156 (09), 139 (08), 128 (15).

3,3'-Dimethoxybiphenyl³(2c): colourless oil; **IR (Neat)** ν = 3058, 3006, 2950, 2838, 1598, 1575, 1481, 1415, 1279, 1237, 1204, 1171, 1052, 1035, 857, 777, 702 cm^{-1} ; **¹H NMR (400 MHz, CDCl₃, 25°C)** δ : 3.82 (3H, s), 6.88 (1H, dd, $J^3=8.2\text{Hz}$, $J^4=2.3\text{Hz}$), 7.12 (1H, dd, $J^4=2.3\text{Hz}$, $J^4=1.8\text{Hz}$), 7.16 (1H, d, $J^3=7.8\text{Hz}$), 7.32 (1H, dd, $J^3=8.2\text{Hz}$, $J^3=7.8\text{Hz}$); **¹³C NMR (100 MHz, CDCl₃, 25°C)** δ : 55.2, 112.7, 112.9, 119.6, 129.7, 142.5, 159.8; **MS (EI, 70eV) m/z (%)** : 214 (100) [M^{++}], 171 (23), 153 (07), 139 (10), 128 (15), 115 (06); **Elemental Analysis** calculated: C 78.48 %, H 6.59 %, O 14.93 %; found : C 78.44 %, H 6.35 %.

4,4'-bis(dimethylamino)biphenyl⁴(2d): white solid; **m.p.** 193-194°C (Lit. 195°C); **IR (Nujol)** ν = 2720, 1609, 1352, 1224, 1189, 1055, 943, 805, 728 cm^{-1} ; **¹H NMR (400 MHz, CDCl₃, 25°C)** δ : 2.96 (6H, s), 6.79 (2H, d, $J^3(\text{H})=8.2\text{Hz}$), 7.45 (2H, d, $J^3(\text{H})=9.2\text{Hz}$); **¹³C NMR (100 MHz, CDCl₃, 25°C)** δ : 40.8 (CH₃), 113.1 (CH), 126.9 (CH), 129.8 (C), 149.2 (C); **MS (EI, 70eV) m/z (%)** : 240 (100) [M^+], 225 (30) [M^+-CH_3], 152 (10), 119 (24).

4,4'-difluorobiphenyl²(2e): white solid; **m.p.** 89-90°C (Litt. 90-93°C); **IR (Nujol)** ν = 3073, 1890, 1603, 1501, 1322, 1235, 1112, 825 cm^{-1} ; **¹H NMR (400 MHz, CDCl₃, 25°C)** δ : 7.07-7.15 (2H, m), 7.44-7.51 (2H, m); **¹³C NMR (100 MHz, CDCl₃, 25°C)** δ : 115.7 (d, $J^2(\text{C-F})=21,1\text{Hz}$), 128.6 (d, $J^3(\text{C-F})=8,6\text{Hz}$), 136.4 (d, $J^4(\text{C-F})=2,9\text{Hz}$), 162.4 (d, $J^1(\text{C-F})=246,6\text{Hz}$); **¹⁹F NMR (376 MHz, CDCl₃, 25°C)** δ : -115,6 (s); **MS (IE, 70eV) m/z (%)** : 190 (100) [M^+], 170 (09) [M^+-HF].

1,1'-binaphthyl²(2f): white solid; **m.p.** 143-144°C (Lit. 158-160°C); **IR (Nujol)** ν = 3048, 1583, 1506, 1015, 968, 805, 779, 682 cm^{-1} ; **¹H NMR (400 MHz, CDCl₃, 25°C)** δ : 7.24-7.31 (1H, m), 7.39 (1H, d, $J^3(\text{H})=8.2\text{Hz}$), 7.42-7.52 (2H, m), 7.58 (1H, t, $J^3(\text{H})=7.6\text{Hz}$), 7.91-7.98 (2H, m); **¹³C NMR (100 MHz, CDCl₃, 25°C)** δ : 125.5 (CH), 125.9 (CH), 126.1 (CH), 126.7 (CH), 127.9 (CH), 128.0 (CH), 128.3 (CH), 133.0 (C), 133.6 (C), 138.6 (C); **MS (EI, 70eV) m/z (%)** : 254 (100) [M^+], 126 (16).

2,2'-binaphthyl²(2g): white solid; **m.p.** 183-184°C (Lit. 183-185°C); **IR (Nujol)** ν = 3048, 1594, 1276, 1132, 968, 886, 856, 810, 738 cm^{-1} ; **¹H NMR (400 MHz, CDCl₃, 25°C)** δ : 7.46-7.56 (2H, m), 7.86-7.98 (4H, m), 8.17 (1H, s); **¹³C NMR (100 MHz, CDCl₃, 25°C)** δ : 125.7 (CH), 126.0 (CH), 126.1 (CH), 126.3 (CH), 127.7 (CH), 128.2 (CH), 128.5 (CH), 132.7 (C), 133.7 (C), 138.4 (C); **MS (EI, 70eV) m/z (%)** : 254 (100) [M^+], 126 (16).

3,3'-bipyridyl⁵(2h): orange oil; **m.p.** 230-231°C (Picrate) (Lit. 232°C); **IR (Neat)** ν = 3032, 1588, 1562, 1465, 1424, 1399, 1332, 1199, 1025, 999, 794, 717 cm^{-1} ; **¹H NMR (270 MHz, CDCl₃, 25°C)** δ : 7.38-7.45 (1H, m), 7.89 (1H, ddd, $J^3=7.8\text{Hz}$, $J^4=2.3\text{Hz}$, $J^4=1.8\text{Hz}$), 8.65 (1H, dd, $J^3=4.8\text{Hz}$, $J^4=1.8\text{Hz}$), 8.85 (2H, d, $J^4=2.3\text{Hz}$); **¹³C NMR (68 MHz, CDCl₃, 25°C)** δ : 123.5, 133.1, 134.1, 147.8, 149.0; **MS (EI, 70eV) m/z (%)** : 156 (100) [M^{+}], 129 (11), 128 (10), 103 (09), 76 (10), 50 (08).

2,2'-Dimethoxybiphenyl⁶(2i): white solid; **m.p.** 155-156°C (Lit. 154-156°C); **IR (Nujol)** ν = 1588, 1506, 1429, 1310, 1281, 1255, 1240, 1220, 1168, 1112, 1061, 1025, 999, 933, 764 cm^{-1} ; **¹H NMR (400 MHz, CDCl₃, 25°C)** δ : 3.77 (3H, s), 6.93-7.04 (2H, m), 7.21-7.38 (2H, m); **¹³C NMR (100 MHz, CDCl₃, 25°C)** δ : 55.6 (CH₃), 111.0 (CH), 120.3 (CH), 127.7 (C), 128.6 (CH), 131.4 (CH), 157.0 (C); **MS (EI, 70eV) m/z (%)** : 214 (100) [M^{+}], 199 (15) [$\text{M}^{+}-\text{CH}_3$], 184 (32), 168 (21), 155 (07), 139 (12), 115 (07), 102 (04), 91 (03).

2,2',4,4',6,6'-hexamethylbiphenyl⁷(2j): white solid; **m.p.** 99-100°C (Lit. 100°C); **IR (Nujol)** ν = 2730, 1609, 1009, 851 cm^{-1} ; **¹H NMR (400 MHz, CDCl₃, 25°C)** δ : 1.86 (6H, s), 2.33 (3H, s), 6.93 (2H, s); **¹³C NMR (100 MHz, CDCl₃, 25°C)** δ : 19.8 (CH₃), 21.1 (CH₃), 128.2 (CH), 135.5 (C), 136.0 (C), 137.0 (C); **MS (EI, 70eV) m/z (%)** : 238 (86) [M^{+}], 223 (100) [$\text{M}^{+}-\text{CH}_3$], 208 (47), 193 (34), 178 (11), 165 (09).

4,4'-dicyanobiphenyl⁸(2k): white solid; **m.p.** 233-234°C (Lit. 233-234°C); **IR (KBr)** ν = 2227, 1604, 1490, 1397, 1180, 859, 818, 545 cm^{-1} ; **¹H NMR (400 MHz, CDCl₃, 25°C)** δ : 7.68-7.75 (2H, m), 7.75-7.82 (2H); **¹³C NMR (100 MHz, CDCl₃, 25°C)** δ : 112.4, 118.4, 127.9, 132.9, 143.5; **MS (EI, 70eV) m/z (%)** : 204 (100) [M^{+}], 177 (10) [$\text{M}^{+}-\text{HCN}$].

3,3'-dicyanobiphenyl⁹(2l): white solid; **m.p.** 200-201°C; **IR (KBr)** ν = 2232, 1602, 1580, 1475, 1401, 1336, 1270, 1195, 1101, 898, 813, 791, 688, 589, 565 cm^{-1} ; **¹H NMR (400 MHz, CDCl₃, 25°C)** δ : 7.62 (1H, dd, $J^3=7.8\text{Hz}$, $J^3=7.8\text{Hz}$), 7.72 (1H, ddd, $J^3=7.8\text{Hz}$, $J^4=1.4\text{Hz}$, $J^4=1.4\text{Hz}$), 7.81 (1H, ddd, $J^3=7.8\text{Hz}$, $J^4=1.4\text{Hz}$, $J^4=1.4\text{Hz}$), 7.85 (1H, d, $J^4=1.4\text{Hz}$); **¹³C NMR (100 MHz, CDCl₃, 25°C)** δ : 113.4, 118.3, 130.0, 130.6, 131.4, 131.8, 140.1; **MS (EI, 70eV) m/z (%)** : 204 (100) [M^{+}], 177 (12) [$\text{M}^{+}-\text{HCN}$], 150 (04) [$\text{M}^{+}-2\text{HCN}$].

2,2'-dicyanobiphenyl¹⁰ (2m): white solid; **m.p.** 173-174°C; **IR (KBr)** ν = 2227, 1594, 1562, 1482, 1429, 1266, 1200, 1064, 975, 768, 735, 562, 540 cm^{-1} ; **¹H NMR (400 MHz, CDCl₃, 25°C)** δ : 7.55-7.61 (2H, m), 7.73 (1H, ddd, $J^3=7.8\text{Hz}$, $J^3=7.8\text{Hz}$, $J^4=1.4\text{Hz}$), 7.81-7.86 (1H, m); **¹³C NMR (100 MHz, CDCl₃, 25°C)** δ : 112.3, 117.5, 129.2, 130.5, 132.8, 133.5, 141.5; **MS (EI, 70eV) m/z (%)** : 204 (100) [M^{+}], 177 (18) [$\text{M}^{+}-\text{HCN}$], 150 (06) [$\text{M}^{+}-2\text{HCN}$]; **Elemental Analysis** calculated : C 82.33 %, H 3.95 %, N 13.72 %; found C 82.16 %, H 3.78 %, N 13.63 %.

4,4'-dicarbethoxybiphenyl⁸(2n): white solid; **m.p.** 223-224°C (Lit. 224-226°C); **IR (KBr)** ν = 1715, 1608, 1561, 1485, 1453, 1401, 1373, 1279, 1185, 1110, 1026, 1007, 885, 852, 758, 697 cm^{-1} ; **¹H NMR (400 MHz, CDCl₃, 25°C)** δ : 1.42 (3H, t, $J^3=7.3\text{Hz}$), 4.41 (2H, q, $J^3=7.3\text{Hz}$), 7.68 (2H, d, $J^3=8.7\text{Hz}$), 8.14 (2H, d, $J^3=8.7\text{Hz}$); **¹³C NMR (100 MHz, CDCl₃, 25°C)** δ : 14.3, 61.0, 127.1, 129.9, 130.1, 144.2, 166.2; **MS (EI, 70eV)** 298 (61) [M^{+}], 270 (07) [$\text{M}^{+}-\text{CO}$], 253 (100) [$\text{M}^{+}-\text{OC}_2\text{H}_5$], 242 (11), 225 (29) [$\text{M}^{+}-\text{COOC}_2\text{H}_5$], 180, (05), 152 (38) [$\text{M}^{+}-2\text{COOC}_2\text{H}_5$], 104 (05), 76 (05).

3,3'-dicarbethoxybiphenyl¹¹ (2o): white solid; **m.p.** 68-69°C; **IR (KBr)** ν = 3002, 2978, 1730, 1594, 1479, 1391, 1372, 1309, 1246, 1106, 1087, 1029, 893, 743, 695 cm^{-1} ; **¹H NMR (400 MHz, CDCl₃, 25°C)** δ : 1.43 (3H, t, $J^3=7.3\text{Hz}$), 4.42 (2H, q, $J^3=7.3\text{Hz}$), 7.54 (1H, dd, $J^3=7.8\text{Hz}$, $J^3=7.8\text{Hz}$), 7.81 (1H, ddd, $J^3=7.8\text{Hz}$, $J^4=1.8\text{Hz}$, $J^4=1.8\text{Hz}$), 8.06 (1H, ddd, $J^3=7.8\text{Hz}$, $J^4=1.8\text{Hz}$, $J^4=1.8\text{Hz}$), 8.31 (1H, dd, $J^4=1.8\text{Hz}$, $J^4=1.8\text{Hz}$); **¹³C NMR (100 MHz, CDCl₃, 25°C)** δ : 14.3, 61.1, 128.2, 128.7, 128.9, 131.1, 131.5, 140.4, 166.4; **MS (EI, 70eV) m/z (%)** : 298 (78) [M^{+}], 270 (08)

[M⁺-CO], 253 (100) [M⁺-OC₂H₅], 242 (09), 225 (20) [M⁺-COOC₂H₅], 152 (46) [M⁺-2COOC₂H₅], 104 (07), 76 (07); **HRMS**: calculated for C₁₈H₁₈O₄: 298.1205, found 298.1207; **Elemental Analysis** calculated: C 72.47 %, H 6.08 %, O 21.45 %; found C 72.46 %, H 6.07 %, O 21.47 %.

2,2'-dinitrobiphenyl¹²(2p): beige solid; **m.p.** 120-122°C (Lit. 123-128°C); **IR (Nujol)** ν = 1600, 1567, 1520, 1353, 1299, 1254, 850, 786, 756, 746, 680, 588 cm⁻¹; **¹H NMR (400 MHz, CDCl₃, 25°C)** δ : 7.29 (1H, dd, J³=7.4Hz, J⁴=1.4Hz), 7.58 (1H, ddd, J³=8.2Hz, J³=7.4Hz, J⁴=1.4Hz), 7.68 (1H, ddd, J³=7.4Hz, J³=7.4Hz, J⁴=1.4Hz), 8.20 (1H, dd, J³=8.2Hz, J⁴=1.4Hz); **¹³C NMR (100 MHz, CDCl₃, 25°C)** δ : 124.7, 129.1, 130.9, 133.4, 134.1, 147.0; **MS (EI, 70eV) m/z (%)**: 198 (100) [M⁺-NO₂], 180 (28), 168 (41), 152 (33), 139 (47), 126 (09), 115 (28), 102 (05), 89 (08), 76 (10), 63 (13), 51 (06).

2-fluoro-3-carbethoxy-4-iodopyridine (3q): colorless oil; **IR (Neat)** ν = 2995, 1739, 1592, 1551, 1448, 1412, 1372, 1293, 1236, 1182, 1124, 1068, 1021, 890, 702 cm⁻¹; **¹H NMR (400 MHz, CDCl₃, 25°C)** δ : 1.43 (3H, t, J³=7.3Hz), 4.47 (2H, q, J³=7.3Hz), 7.71 (1H, d, J³=5.0Hz), 7.92 (1H, d, J³=5.0Hz); **¹³C NMR (100 MHz, CDCl₃, 25°C)** δ : 13.9, 62.6, 107.1 (d, J³(C-F)=2.9Hz), 123.0 (d, J²(C-F)=34.5Hz), 132.0 (d, J⁴(C-F)=4.8Hz), 148.2 (d, J³(C-F)=16.3Hz), 158.6 (d, J¹(C-F)=244Hz), 163.4 (d, J³(C-F)=4.8Hz); **RMN ¹⁹F (376 MHz, CDCl₃, 25°C)** δ : -65.4 (s); **MS (EI, 70eV) m/z (%)**: 295 (47) [M⁺], 267 (54) [M⁺-C₂H₄], 250 (100) [M⁺-OC₂H₅], 222 (20) [M⁺-COOC₂H₅], 195 (09), 123 (07), 95 (07), 68 (11); **HRMS**: calculated for C₈H₇FINO₂: 294.9506, found 294.9502. **Elemental Analysis** calculated: 32.57 %, H 2.39 %, N 4.75 %; found C 32.34 %, H 2.31 %, N 4.64 %.

2,2'-difluoro-3,3'-dicarbethoxy-4,4'-bipyridyl (2q): yellow solid; **m.p.** 84-86°C; **IR (KBr)** ν = 2983, 1725, 1598, 1542, 1476, 1411, 1382, 1327, 1293, 1242, 1180, 1152, 1081, 1021, 852, 796, 664, 589 cm⁻¹; **¹H NMR (400 MHz, CDCl₃, 25°C)** δ : 1.12 (3H, t, J³=7.1Hz), 4.18 (2H, q, J³=7.1Hz), 7.16 (1H, d, J³=5.0Hz), 8.38 (1H, d, J³(H)=5.0Hz); **¹³C NMR (100 MHz, CDCl₃, 25°C)** δ : 13.4, 62.0, 114.4 (d, J²(C-F)=30.7Hz), 121.7 (d, J⁴(C-F)=4.8Hz), 149.1 (d, J³(C-F)=16.3Hz), 150.1, 160.4 (d, J¹(C-F)=160.5Hz), 162.3 (d, J³(C-F)=5.8Hz); **¹⁹F NMR (376 MHz, CDCl₃, 25°C)** δ : -65.0 (s); **MS (CI, CH₄) m/z (%)**: 377 (10), 365 (21), 337 (100) [MH⁺], 317 (07) [M⁺-F], 291 (78) [M⁺-OC₂H₅], 263 (11) [M⁺-COOC₂H₅], 245 (05), 219 (05); **HRMS**: calculated for C₁₆H₁₅F₂N₂O₄: 337.1000, found 337.0997; **Elemental Analysis** calculated: 57.14 %, H 4.20 %, N 8.33 %, F 11.30 %; found C 57.30 %, H 4.22 %, N 8.26 %, F 11.26 %.

1,2-bis(2-iodophenyl)ethane¹³(4): white solid; **m.p.** 101-103°C; **IR (KBr)** ν = 2946, 2932, 2861, 1462, 1428, 1293, 1204, 1152, 1091, 1048, 1012, 951, 861, 753, 716, 646, 538 cm⁻¹; **¹H NMR (400 MHz, CDCl₃, 25°C)** δ : 2.99 (2H, s), 6.90 (1H, ddd, J³=7.8Hz, J³=7.8Hz, J⁴=1.8Hz), 7.19-7.31 (2H, m), 7.83 (1H, d, J³=7.8Hz); **¹³C NMR (100 MHz, CDCl₃, 25°C)** δ : 41.2, 100.6, 128.0, 128.3, 129.7, 139.4, 143.6; **MS (EI, 70eV) m/z (%)**: 434 (44) [M⁺], 307 (07) [M⁺-I], 217 (100) [M⁺-C₆H₄ICH₂], 180 (67) [M⁺-2I], 165 (15), 152 (06), 90 (23) [M⁺-C₆H₄ICH₂-I], 63 (05).

9,10-dihydrophenanthrene¹⁴(6): colorless liquid; **IR (Neat)** ν = 3068, 3030, 2941, 2898, 2838, 2359, 1489, 1458, 1449, 779, 749, 730, 622 cm⁻¹; **¹H NMR (400 MHz, CDCl₃, 25°C)** δ : 2.87 (2H, s), 7.20-7.26 (2H, m), 7.26-7.33 (1H, m), 7.74 (1H, d, J³=7.8Hz); **¹³C NMR (100 MHz, CDCl₃, 25°C)** δ : 29.0, 123.7, 126.9, 127.3, 128.1, 134.5, 137.4; **MS (EI, 70eV) m/z (%)**: 180 (100) [M⁺], 165 (33) [M⁺-CH₃], 152 (12) [M⁺-C₂H₄], 89 (10), 76 (05).

6-bromopiperonal¹⁵(7): white solid, **m.p.** 124-126°C (Lit. 131°C); **IR (KBr)** ν = 2866, 1678, 1617, 1598, 1490, 1415, 1392, 1345, 1265, 1218, 1115, 1030, 979, 930, 890, 842, 782, 679, 602, 517 cm⁻¹; **¹H NMR (400 MHz, CDCl₃, 25°C)** δ : 6.09 (2H, s), 7.05 (1H, s), 7.34 (1H, s), 10.17 (1H, s); **¹³C NMR (100 MHz, CDCl₃, 25°C)** δ : 102.7, 108.0, 113.2, 121.5, 127.9, 148.1, 153.3,

190.3; **MS (EI, 70eV) m/z (%)** : 229 (100) [M^{+}], 199 (24) [M^{+} -CHO], 169 (05), 143 (11), 120 (10), 90 (05), 62 (23), 50 (05).

6-iodopiperonal¹⁶(8): yellow solid, **m.p.** 102-103°C (Lit. 102-104°C); **IR (KBr)** ν = 2894, 1674, 1612, 1594, 1508, 1486, 1406, 1386, 1345, 1256, 1115, 1045, 970, 932, 876, 847, 786, 663, 607 cm^{-1} ; **¹H NMR (400 MHz, CDCl₃, 25°C)** δ : 6.09 (2H, s), 7.32 (1H, s), 7.35 (1H, s), 9.87 (1H, s); **¹³C NMR (100 MHz, CDCl₃, 25°C)** δ : 93.3, 102.6, 108.7, 119.3, 129.5, 149.1, 153.4, 194.4; **MS (EI, 70eV) m/z (%)** : 276 (100) [M^{+}], 247 (09) [M^{+} -CHO], 148 (12) [M^{+} -I], 120 (18) [M^{+} -CHO-I], 62 (11).

2,2'-diiodo-N-methyl-4,5-methylenedioxybenzanilide (9): white solid, **m.p.** 213-214°C; **IR (KBr)** ν = 1645, 1575, 1505, 1485, 1425, 1387, 1359, 1303, 1237, 1124, 1101, 1040, 922, 865, 847, 763, 725, 683, 566 cm^{-1} ; Owing to the restricted rotation around the amide bond, two rotamers are observed (ratio 70 :30) at 25°C in CDCl₃ : **¹H NMR (400 MHz, CDCl₃, 25°C)** δ : 3.12 (0.9H, s) and 3.38 (2.1H, s), 5.85, 5.87 (1.4H, 2d, $J^2=1.4\text{Hz}$) and 6.03, 6.05 (0.6H, 2d, $J^2=1.4\text{Hz}$), 6.86 (0.7H, s) and 7.01 (0.3H, s), 6.92 (0.7H, ddd, $J^3=7.8\text{Hz}$, $J^3=7.3\text{Hz}$, $J^4=1.8\text{Hz}$) and 7.06-7.13 (0.3H, m), 7.10 (0.7H, s) and 7.27 (0.3H, s), 7.22 (0.7H, ddd, $J^3=7.8\text{Hz}$, $J^3=7.3\text{Hz}$, $J^4=1.4\text{Hz}$) and 7.44-7.54 (0.3H, m), 7.48 (1H, dd, $J^3=8.2\text{Hz}$, $J^3=7.8\text{Hz}$, $J^4=1.8\text{Hz}$), 7.82 (0.7H, dd, $J^3=7.8\text{Hz}$, $J^4=1.4\text{Hz}$) and 7.93 (0.3H, d, $J^3=7.3\text{Hz}$); **¹³C NMR (100 MHz, CDCl₃, 25°C)** δ : 36.9 and 39.4, 81.0 and 83.1, 98.1 and 98.7, 101.7 and 102.0, 107.2 and 107.7, 118.4, 118.7, 128.6, 129.48, 129.50, 129.59, 129.9, 135.2 and 135.4, 139.7 and 140.0, 144.8, 145.6, 147.5, 148.47, 148.53, 148.9, 169.4 and 169.5; **MS (EI, 70eV) m/z (%)** : 507 (06) [M^{+}], 380 (100) [M^{+} -I], 275 (90) [M^{+} -IC₆H₄NCH₃], 253 (15), 247 (11) [M^{+} -IC₆H₄NCH₃-CO], 120 (12), 62 (05); **HRMS** : calculated for C₁₅H₁₂O₃Ni₂ : 507.8907, found 507.8904. **Elemental Analysis** calculated : C 35.53 %, H 2.19 %, N 2.76 % ; found C 35.32 %, H 2.18 %, N 2.67 %.

N-methylcrinasiadine¹⁷(11): pale yellow solid, **m.p.** 241-243°C (Lit. 247.5-248.5°C); **IR (KBr)** ν = 2922, 1658, 1622, 1608, 1499, 1463, 1415, 1400, 1350, 1317, 1264, 1250, 1209, 1099, 1039, 937, 880, 847, 753, 683, 627, 594 cm^{-1} ; **¹H NMR (400 MHz, CDCl₃, 25°C)** δ : 3.81 (3H, s), 6.13 (2H, s), 7.30 (1H, ddd, $J^3=7.6\text{Hz}$, $J^3=7.6\text{Hz}$, $J^4=0.9\text{Hz}$), 7.40 (1H, d, $J^3=8.3\text{Hz}$), 7.52 (1H, ddd, $J^3=8.7\text{Hz}$, $J^3=6.9\text{Hz}$, $J^4=1.4\text{Hz}$), 7.62 (1H, s), 7.91 (1H, s), 8.09 (1H, dd, $J^3=7.8\text{Hz}$, $J^4=1.2\text{Hz}$); **¹³C NMR (100 MHz, CDCl₃, 25°C)** δ : 30.0, 100.4, 101.9, 107.0, 115.0, 119.2, 121.3, 122.3, 122.9, 128.9, 130.4, 137.4, 148.4, 152.1, 160.9; **MS (EI, 70eV) m/z (%)** : 253 (100) [M^{+}], 224 (10), 166 (06), 139 (07); **HRMS** : calculated for C₁₅H₁₁O₃N : 253.0739, found 253.0744.

2-fluoro-3-iodopyridine¹⁸(12): white solid ; **m.p.** 42-43°C (Lit. 42°C); **IR (KBr)** ν = 1582, 1551, 1425, 1401, 1251, 1063, 1021, 847, 804, 739, 650, 569 cm^{-1} ; **¹H NMR (400 MHz, CDCl₃, 25°C)** δ : 6.94-7.01 (1H, m), 8.14-8.21 (2H, m); **¹³C NMR (100 MHz, CDCl₃, 25°C)** δ : 76.0 (d, $J^2(\text{C-F})=43\text{Hz}$), 122.7 (d, $J^4(\text{C-F})=3.8\text{Hz}$), 147.2 (d, $J^3(\text{C-F})=13.4\text{Hz}$), 150.1 (d, $J^3(\text{C-F})=2.9\text{Hz}$), 162.0 (d, $J^1(\text{C-F})=235\text{Hz}$); **RMN ¹⁹F (376 MHz, CDCl₃, 25°C)** δ : -54.4 (s); **MS (EI, 70eV) m/z (%)** : 223 (100) [M^{+}], 127 (06) [M^{+} -C₅H₃NF], 96 (22) [M^{+} -I], 76 (10) [M^{+} -I-HF], 73 (10).

2-bromo-1-(bromomethyl)benzene¹⁹(13): colourless liquid ; **b.p.** 74°C under 0.30 Torr (Lit. 120-130°C under 13 Torr); **IR (Neat)** ν = 661, 728, 758, 1030, 1051, 1224, 1445, 1482, 1568, 1690, 3063 cm^{-1} ; **¹H NMR (400 MHz, CDCl₃, 25°C)** δ : 4.60 (2H, s), 7.16 (1H, ddd, $J^3=7.8\text{Hz}$, $J^3=7.8\text{Hz}$, $J^4=1.4\text{Hz}$), 7.29 (1H, dd, $J^3=7.8\text{Hz}$, $J^3=7.8\text{Hz}$), 7.44 (1H, dd, $J^3=7.8\text{Hz}$, $J^4=1.4\text{Hz}$), 7.56 (1H, d, $J^3=7.8\text{Hz}$); **¹³C NMR (100 MHz, CDCl₃, 25°C)** δ : 33.4, 124.4, 127.9, 130.1, 131.2, 133.3, 136.9 ; **MS (EI, 70eV) m/z (%)** : 250 (20) [M^{+}], 171 & 169 (96 & 100) [M^{+} -Br], 90 (35) [M^{+} -2Br], 63 (14).

1,2-bis(2-bromophenyl)ethane²⁰(14): white solid; **m.p.** 84-85°C (Lit. 84-86°C); **IR (KBr)** ν = 2932, 1566, 1472, 1453, 1435, 1293, 1156, 1096, 1040, 1021, 951, 870, 809, 753, 721, 655, 546 cm^{-1} ; **¹H NMR (400 MHz, CDCl₃, 25°C)** δ : 3.03 (2H, s), 7.01-7.09 (1H, m), 7.14-7.23 (2H, m),

7.53 (1H, d, $J^3=8.2\text{Hz}$); ^{13}C NMR (100 MHz, CDCl_3 , 25°C) δ : 36.4, 124.5, 127.4, 127.8, 130.6, 132.8, 140.5; MS (EI, 70eV) m/z (%) : 340 (26) [M^{++}], 259 (09) [$\text{M}^{++}\text{-Br}$], 179 (24) [$\text{M}^{++}\text{-2Br}$], 169 (100) [$\text{M}^{++}\text{-BrC}_6\text{H}_4\text{CH}_3$], 90 (23), 63 (06).

6-iodo-3,4-methylenedioxybenzoic acid²¹(15): white solid, m.p. $209\text{-}211^\circ\text{C}$ (Lit. $216\text{-}217^\circ\text{C}$); IR (KBr) ν = 3758, 2913, 1702, 1621, 1505, 1448, 1420, 1376, 1359, 1284, 1256, 1152, 1120, 1035, 922, 885, 857, 786, 697, 617 cm^{-1} ; ^1H NMR (400 MHz, $\text{DMSO-}d_6$, 25°C) δ : 6.09 (2H, s), 7.28 (1H, s), 7.47 (1H, s); ^{13}C NMR (100 MHz, $\text{DMSO-}d_6$, 25°C) δ : 85.5, 103.1, 110.7, 120.3, 129.8, 148.3, 151.0, 167.6; MS (EI, 70eV) m/z (%) : 292 (100) [M^{++}], 275 (32) [$\text{M}^{++}\text{-OH}$], 247 (04) [$\text{M}^{++}\text{-COOH}$], 165 (05), 107 (05), 62 (08).

2,2'-diiodo-4,5-methylenedioxybenzanilide (16): white solid, m.p. $182\text{-}183^\circ\text{C}$; IR (KBr) ν = 3152, 1645, 1583, 1533, 1495, 1476, 1434, 1401, 1383, 1345, 1302, 1242, 1143, 1035, 932, 744, 702, 678, 556 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3 , 25°C) δ : 6.06 (2H, s), 6.90 (1H, ddd, $J^3=7.3\text{Hz}$, $J^3=7.3\text{Hz}$, $J^4=1.4\text{Hz}$), 7.08 (1H, s), 7.33 (1H, s), 7.40 (1H, dd, $J^3=7.8\text{Hz}$, $J^3=7.8\text{Hz}$), 7.73 (1H, s), 7.82 (1H, dd, $J^3=8.2\text{Hz}$, $J^4=1.4\text{Hz}$), 8.36 (1H, d, $J^3=7.3\text{Hz}$); ^{13}C NMR (100 MHz, CDCl_3 , 25°C) δ : 81.7, 90.3, 102.3, 108.7, 119.7, 122.2, 126.5, 129.3, 134.9, 137.9, 138.9, 148.4, 150.0, 166.7; MS (EI, 70eV) m/z (%) : 493 (13) [M^{++}], 366 (59) [$\text{M}^{++}\text{-I}$], 275 (100) [$\text{M}^{++}\text{-IC}_6\text{H}_4\text{NH}$], 247 (12) [$\text{M}^{++}\text{-IC}_6\text{H}_4\text{NH-CO}$], 239 (12) [$\text{M}^{++}\text{-2I}$], 120 (13), 91 (05), 62 (05). Elemental Analysis calculated : C 34.11 %, H 1.84 %, N 2.84 % ; found C 33.93 %, H 1.78 %, N 2.74 %.

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X : parts per Million : 1H

2q - ¹³C NMR

CDCl₃, 100.5 MHz

X : parts per Million : ¹³C

3q - ¹³C NMR

CDCl₃, 100.5 MHz

9 - ¹H NMR

CDCl₃, 400 MHz

X : parts per Million : 1H