**Supporting Information**

A Novel Electromagnetic Mill Promoted Mechanochemical Solid-State Suzuki–Miyaura Cross-Coupling Reactions: Ultra-Low Catalyst Loading without Molecular Dispersants

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## General Information

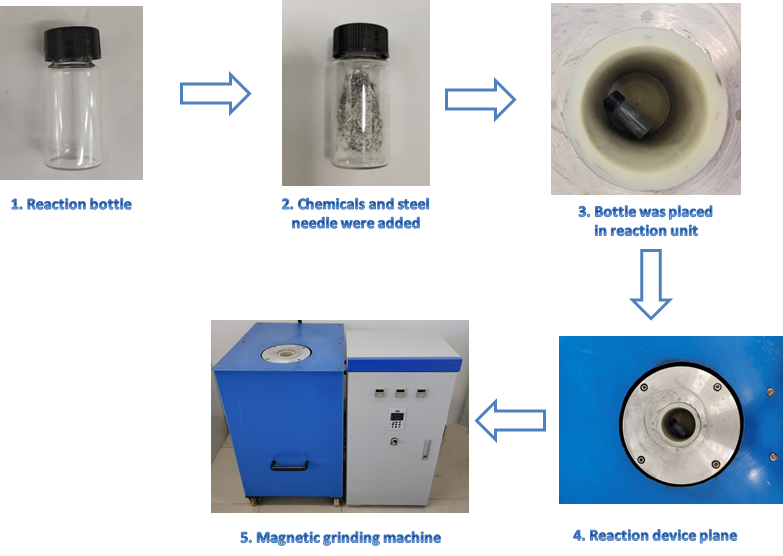
The starting materials were obtained from commercial suppliers and used as received. Solvents were purchased from commercial suppliers. Purifications of reactions products were carried out by flash chromatography using Merck silica gel (40-63 µ m). All mechanochemical reactions were carried out using grinding vessels in a Magnetic grinding machine. The reaction bottles used were commercially available 10 ml flat bottom flask or customized reaction jar（6.5◊8cm）.The grinding medium is customized ferromagnetic rods（3.5◊0.5mm）. 1H NMR (400 MHz), 13C NMR (100 MHz) were measured on a Brucker Avance 400 MHz spectrometer. Chemical shifts are reported in parts per million (ppm, δ) downfield from residual solvents peaks and coupling constants are reported as Hertz (Hz). Splitting patterns are designated as singlet (s), doublet (d), triplet (t), ….Splitting patterns that could not be interpreted or easily visualized are designated as multiplet (m).Electrospray mass spectra were obtained using an ESI/TOF Mariner Mass Spectrometer. Unless otherwise noted, all other commercially available reagents and solvents were used without further purification. XPS analyses were made Thermo ESCALAB XI+.

## General Procedure for Solid-State Cross-Coupling

1)**Procedure l:General solid-state cross –coupling reaction**



Aryl halide **1a** (199.04mg. 1mmol 1.0equiv), aryl boronic acid **2a** (181.2mg, 1.2mmol, 1.2 equiv), Pd(dppf)Cl2 (0.4mg. 0.05% mmol) and DavePhos (0.3mg, 0.075%mmol), KF (174.21mg, 3mmol 3.0 equiv) ferromagnetic rods 5g (about a quarter of the volume of the bottle)were placed in a flat bottom flask. Put it in magnetic grinder. After 1h , the mixture was dissolved in CH2Cl2, filtering out ferromagnetic rods. Then remove the inorganic salt from the mixture with water. The organic phase was combined, dried over Na2SO4, evaporated and purified by flash chromatography (PE:EA = 10:1) to give compound **3a** (224.0 mg, 99% yield).



**Figure S1.**Set-up procedure for the solid-state cross-coupling.

**2) Procedure II: Procedure for Solid-State Cross-Coupling at Gram Scale**

Aryl halide **1a** (2g. 5mmol 1.0equiv), aryl boronic acid **2a** (1.81g, 6.1mmol, 1.2 equiv), Pd(dppf)Cl2 (1.8 mg. 0.05% mmol) and DavePhos (1.5 mg, 0.075% mmol), KF (0.87g, 1.5mmol 3.0 equiv) ferromagnetic rods 20g (about a quarter of the volume of the jar) were placed in ferromagnetic rods. After 6 h, the mixture was dissolved in CH2Cl2, filtering out ferromagnetic rods .Then remove the inorganic salt from the mixture with water. The organic phase was combined, dried over Na2SO4, evaporated and purified by flash chromatography (PE:EA = 10:1) to give compound **3a** (2.08g, 92% yield).

**3) Procedure III: Coupling procedure for fluorescent products**



Aryl halide **1-6a** (287.11mg. 1mmol 1.0equiv), aryl boronic acid **2a** (181.2mg, 1.2mmol, 1.2 equiv), Pd(dppf)Cl2 (0.4 mg. 0.05% mmol) and DavePhos (0.3 mg, 0.075% mmol), KF (174.21g, 3mmol 3.0 equiv) ferromagnetic rods 5g (about a quarter of the volume of the jar) were placed in flat bottom flask. After 1 h , the mixture was dissolved in CH2Cl2, filtering out ferromagnetic rods .Then remove the inorganic salt from the mixture with water. The organic phase was combined, dried over Na2SO4, evaporated and purified by flash chromatography (PE:EA = 10:1) to give compound yellow solid **6a** (300mg, 95% yield).

**4) Procedure IV: Coupling procedure for insoluble compounds**



Aryl halide **1-7a** (168.01mg. 0.5mmol 1.0equiv), aryl boronic acid **2a** (181.2mg, 1.2mmol, 1.2 equiv), Pd(dppf)Cl2 (0.4 mg. 0.05% mmol) and DavePhos (0.3 mg, 0.075% mmol), KF (174.21g, 3mmol 3.0 equiv) ferromagnetic rods 5g (about a quarter of the volume of the jar) were placed in flat bottom flask. After 1 h , the mixture was dissolved in CH2Cl2, filtering out ferromagnetic rods .Then remove the inorganic salt from the mixture with water. The organic phase was combined, dried over Na2SO4, evaporated and purified by flash chromatography (PE:EA = 10:1) to give compound yellow solid **7a** (145mg, 74% yield).

**Solubility evaluation of compounds**

An compound (7d.7e) and toluene were add vial and stirred for 1 h at room temperature (23℃). Then the mixture was filtered using a syringe filter (diameter; 0.22 um) and concentrated under reduced pressure until the weight of a flask with the obtained solid was not changed. The solubility was evaluated by measuring the weight of the obtained solid. (1-7a,1-7b,1-7c solubility(1))



**Figure S2**.Solubility evaluation of compounds.

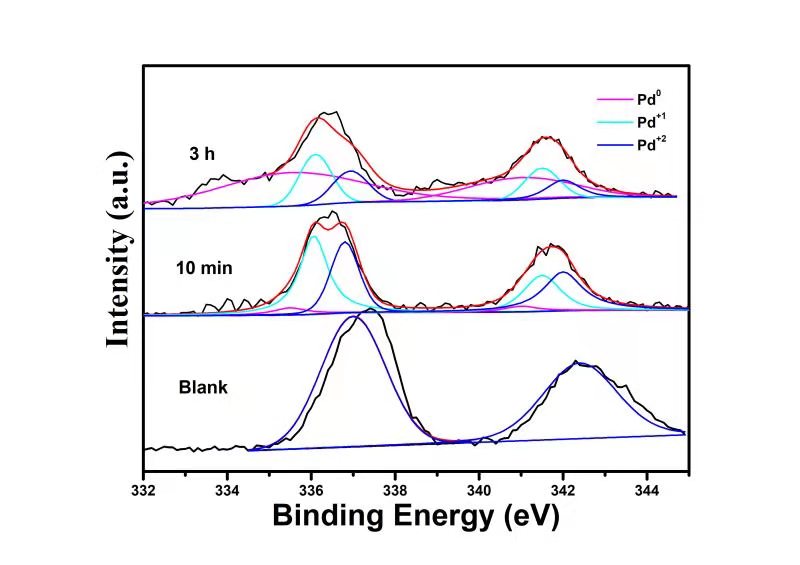
**4) Procedure V: General procedure for drug molecular coupling**



Aryl halide **1-8a** (182.02mg. 1mmol 1.0equiv), aryl boronic acid **2-8a** (163.152mg, 1.5mmol, 1.2 equiv), Pd(dppf)Cl2 (0.4 mg. 0.05% mmol) and DavePhos (0.3 mg, 0.075% mmol), KF (174.21g, 3mmol 3.0 equiv) ferromagnetic rods 5g (about a quarter of the volume of the jar) were placed flat bottom flask. After 1 h, the mixture was dissolved in CH2Cl2, filtering out ferromagnetic rods .Then remove the inorganic salt from the mixture with water. The organic phase was combined, dried over Na2SO4, evaporated and purified by flash chromatography (PE:EA = 10:1) to give compound white solid **8a** (139.14mg, 72% yield).

## Details of X-ray Photoelectron Spectroscopy

The crude mixtures were prepared by the following conditions: Aryl halide **1** (199.04mg. 1mmol 1.0equiv), aryl boronic acid **2** (181.2mg, 1.2mmol, 1.2 equiv), Pd(OAc)2 (15.7mg. 0.07 mmol) and DavePhos (39.3mg, 10 mmol), KF (174.21mg, 3mmol 3.0 equiv) ferromagnetic rods 5g (about a quarter of the volume of the bottle)were placed in flat bottom flask; 50 Hz. According to the above conditions, two reactions are opened in parallel, one is taken out after 10min and the other is taken out after 3h. The solid in the bottle is put into a mortar and ground into a powder to take 20-30mg sample preparation .Then we made the blank sample only added Pd(OAc)2 (15.7mg. 0.07 mmol) and DavePhos (39.3mg, 10 mmol) take 20-30mg sample preparation . In order to study the valence change of Pd during grinding process, we used blank sample 10 min sample and 3 h sample to carry out XPS analysis.



**Figure S3**.Peak splitting display diaram of XPS.

## **Characterization of Obtained Coupling Products**.

**1-(4'-methoxy-[1,1'-biphenyl]-4-yl)ethan-1-one**



**C15H14O2 Melting Point**:150.0℃-153.7℃

**MW**: 226.28 g·mol-1 White Solid

**Isolated amounts**:224.0 mg **Yield**: 99%

**1H NMR (400 MHz, CDCl3, δ ppm):** 8.01 (d, *J* = 7.6 Hz, 2H), 7.64 (d, *J* =7.6 Hz, 2H), 7.58 (d, *J* = 8.0 Hz, 2H), 7.00 (d, *J* =8.0 Hz, 2H), 3.86(s, 3H). 2.63 (s, 3H),

**13C NMR (100 MHz, CDCl3, δ ppm):** 197.75, 159.84, 145.31, 135.18, 132.16, 128.90, 128.31, 128.55, 114.34, 55.33, 26.60.

**HRMS (ESI)** Calcd for C15H14O2+H 227.1080., Found 227.1072

**1-(2'-methoxy-[1,1'-biphenyl]-4-yl)ethan-1-one**



**C15H14O2 Melting Point**:106.5℃-106.8℃

**MW**: 226.28 g·mol-1 White Solid

**Isolated amounts**:208.18 mg **Yield**: 92%

**1H NMR (400 MHz, CDCl3, δ ppm):** 8.00 ( d, *J* =7.6 Hz, 2H), 7.64 (d, *J* =7.6Hz, 2H ) 7.36( q , *J* =9.6 Hz,2 H) , 7.07-7.00(m, 2H) , 3.83( s , 3H), 2.64 ( s , 3H)

**13C NMR (100 MHz, CDCl3, δ ppm):** 197.90, 156.41, 143.56, 135.45, 130.69, 129.70, 129.46, 129.40, 128.06, 120.92, 111.28, 55.54, 26.65.

**1-([1,1'-biphenyl]-4-yl)ethan-1-one**



**C14H12O Melting Point**:122.0℃-122.2℃

**MW**: 196.25 g·mol-1 White Solid

**Isolated amounts**:184.48 mg **Yield**: 94%

**1H NMR (400 MHz, CDCl3, δ ppm):** 8.04 ( d, *J* =7.6 Hz, 2H), 7.69 (d, *J* =7.6Hz, 2H ) 7.63( d, *J* =7.2 Hz, 2 H) , 7.48 ( t , *J* =7.2Hz, 2 H), 7.41( t , *J* =7.2 Hz, 1 H), 2.64 ( s , 3H)

**13C NMR (100 MHz, CDCl3, δ ppm):** 197.75, 145.72, 139.79, 135.76, 128.90, 128.87, 128.18, 127.21, 127.17, 26.64.

**1-(2'-methyl-[1,1'-biphenyl]-4-yl)ethan-1-one**



**C15H14O**

**MW**: 210.28 g·mol-1 Yellow Oil

**Isolated amounts**:203.97 mg **Yield**: 97%

**1H NMR (400 MHz, CDCl3, δ ppm):** 7.99 ( d, *J* =7.6 Hz, 2H), 7.40 (d, *J* =7.6 Hz, 2H ) 7.26-7.19( m, 4 H) , 2.61( s , 3H), 2.25 ( s , 3H).

**13C NMR (100 MHz, CDCl3, δ ppm):** 197.52, 146.70, 140.50, 135.35, 134.89, 130.34, 129.28,129.24, 128.02, 127.71, 125.75, 26.42, 20.20

**1-(4'-(tert-butyl)-[1,1'-biphenyl]-4-yl)ethan-1-one**



**C18H20O Melting Point**:131.8℃-133.0℃

**MW**: 252.38 g·mol-1 White Solid

**Isolated amounts**:239.76 mg **Yield**: 95%

**1H NMR (400 MHz, CDCl3, δ ppm):** 7.98 ( d, *J* = 7.6 Hz, 2H), 7.64 (d, *J* =8.0 Hz, 2H), 7.55 (d, *J* =7.6 Hz, 2H) , 7.47 (d, *J* =7.6 Hz, 2H) ,2.58 ( s, 3H), 1.35 ( s , 9 H)

**13C NMR (100 MHz, CDCl3, δ ppm):** 197.76, 151.41, 145.57 136.86, 135.55, 128.87, 126.96, 126.89, 125.91, 34.61, 31.28, 26.65,.

**1-(2',4',6'-trimethyl-[1,1'-biphenyl]-4-yl)ethan-1-one**



**C17H18O Melting Point**:93.8℃-94.5℃

**MW**: 238.33 g·mol-1 White Solid

**Isolated amounts**:197.81 mg **Yield**: 83%

**1H NMR (400 MHz, CDCl3, δ ppm):** 7.94 ( d, *J* =8 Hz, 2H), 7.17 (d, *J* =7.6 Hz, 2H ) 6.87( s, 2H) , 2..57( s , 3H) , 2.25( s , 3H), 1.90 ( s , 6H)

**13C NMR (100 MHz, CDCl3, δ ppm):** 197.89, 146.49, 137.83, 137.08, 135.47 135.41, 129.63, 128.49, 128.15, 26.59, 20.59,20.98, 20.60.

**1-(4'-(trifluoromethoxy)-[1,1'-biphenyl]-4-yl)ethan-1-one**



**C15H11F3O2 Melting Point**:85.9℃-86.5℃

**MW**: 280.25 g·mol-1 White Solid

**Isolated amounts**:238.21 mg **Yield**: 85%

**1H NMR (400 MHz, CDCl3, δ ppm):** 8.02 ( d, *J* = 7.6 Hz, 2H), 7.62 (d, *J* =9.2 Hz, 4H), 7.30 (d, *J* =8 .0Hz, 2H) , ,2.62 ( s, 3H).

**13C NMR (100 MHz, CDCl3, δ ppm):** 197.43, 149.15(d, *J* =2 Hz ), 144.04, 138.39 , 136.00, 128.87, 128.52, 127.02, 121.21, 26.45.

**19F NMR (376 MHz, CDCl3, δ ppm):** δ-57.82

**1-(2'-fluoro-[1,1'-biphenyl]-4-yl)ethan-1-one**



**C14H11FO Melting Point**:84.9℃-85.8℃

**MW**: 214.24 g·mol-1 White Solid

**Isolated amounts**:162.82 mg **Yield**: 76%

**1H NMR (400 MHz, CDCl3, δ ppm):** 8.04 (d, *J* = 7.6 Hz, 2H), 7..66 (d, *J* =8.0 Hz, 2H), 7.46 (t, *J* = 7.6 Hz, 1H), 7.37 (q , *J* = 6.0Hz, 1 H), 7.24 (d , *J* = 7.4Hz, 1H), 7.18 (t , *J* = 9.6 Hz, 1H) ,2.65 (s, 3H),

**13C NMR (100 MHz, CDCl3, δ ppm):** 197.74, 160.92, 158.45, 140.53, 136.04, 130.58 (d*,J*=3 Hz) 129.87(d*,J*=8 Hz), 129.18 d*,J*=3 Hz), 128.45, 127.84(d*,J*=13 Hz), 124.53(d*,J*=3 Hz), 116.26(d*,J*=23 Hz), 26.68.

**19F NMR (376 MHz, CDCl3, δ ppm):** δ-117.48

**1-(2'-chloro-[1,1'-biphenyl]-4-yl)ethan-1-one**



**C14H11ClO Melting Point**:52.9℃-53.9℃

**MW**: 230.69 g·mol-1 White Solid

**Isolated amounts**:177.63 mg **Yield**: 77%

**1H NMR (400 MHz, CDCl3, δ ppm):** 8.03 (d, *J* = 7.6 Hz, 2H), 7.55 (d, *J* =8.0 Hz, 2H), 7.49 (d, *J* = 6.8 Hz, 1H) ,7.34-7.31(m, 3H), 2.65 (s, 3H),

**13C NMR (100 MHz, CDCl3, δ ppm):** 197.73, 144.08, 139.34, 136.08, 132.25, 131.06, 130.08, 129.71, 129.17, 128.09, 126.96, 26.68

**HRMS (ESI)** Calcd forC14H11ClO+H 231.0572, Found 231.0577

**1-(4'-fluoro-[1,1'-biphenyl]-4-yl)ethan-1-one**



**C14H11FO Melting Point**:102.3℃-103.7℃

**MW**: 214.24 g·mol-1 White Solid

**Isolated amounts**:194.96 mg **Yield**: 91%

**1H NMR (400 MHz, CDCl3, δ ppm):** 8.02 ( d, *J* =8.0 Hz, 2H), 7.64-7.57(m, 4H ) 7.15 ( t , *J* =8.4 Hz, 2 H) , 2.63 ( s , 3H)

**13C NMR (100 MHz, CDCl3, δ ppm):** 197.62, 162.90((d, *J* =246 Hz), 144.63, 135.90 (d, *J* =3 Hz), 135.73, 128.91, 128.82, 126.98,115.85, (d, *J* =22 Hz) 26.62

**19F NMR (376 MHz, CDCl3, δ ppm):** δ-113.967.

**1-(2',4'-difluoro-[1,1'-biphenyl]-4-yl)ethan-1-one**



**C14H10F2O Melting Point**:77.2℃-79.8℃

**MW**: 230.07 g·mol-1 White Solid

**Isolated amounts**:204.76 mg **Yield**: 89%

**1H NMR (400 MHz, CDCl3, δ ppm):** 8.02 ( d, *J* =8.0 Hz, 2H) , 7.59 (d, *J* =7.6 Hz, 2H), 7.42 (q, *J* =8 Hz, 1H) , 6.99-6.90 (m, 2H) , 2.63 ( s , 3H)

**13C NMR (100 MHz, CDCl3, δ ppm):** 197.58, 162.47(dd, *J* =309,285 Hz)159.97(dd, *J* =310,262Hz)139.59(d,*J* =1Hz) 136.07, 131.29 (dd, *J* = 15, 5Hz) 128.99(d, *J* = 2Hz), 124.10(dd,*J* = 18, 10Hz)111.81(dd, *J* = 25, 17Hz)104.60(t, *J* = 17Hz), 26.61

**19F NMR (376 MHz, CDCl3, δ ppm):** δ-109.79, δ-112.94

**1-(3',4'-dichloro-[1,1'-biphenyl]-4-yl)ethan-1-one**



**C14H10Cl2O Melting Point**:131.7℃-134.5℃

**MW**:265.13g·mol-1 White Solid

**Isolated amounts**:188.24 mg **Yield**: 71%

**1H NMR (400 MHz, CDCl3, δ ppm):** 8.02 ( d, *J* =8.0 Hz, 2H), 7.68 (s ,1H ) 7.61( d, *J* =8.0 Hz, 2 H) , 7.51 (d, *J* =8.4 Hz, 1 H), 7.43 (d, *J* =8.0 Hz, 1 H), 2.64 ( s , 3H).

**13C NMR (100 MHz, CDCl3, δ ppm):** 197.41, 142.98, 139.69, 136.37, 133.02, 132.39, 130.80, 128.98, 128.94, 126.97, 126.34, 26.62.

**1-(3',5'-difluoro-[1,1'-biphenyl]-4-yl)ethan-1-one**



**C14H10F2O Melting Point**:72.5℃-72.9℃

**MW**: 230.07 g·mol-1 White Solid

**Isolated amounts**:179.45 mg **Yield**: 78%

**1H NMR (400 MHz, CDCl3, δ ppm):** 8.02 ( d, *J* =7.6 Hz, 2H), 7.61 (d, *J* =7.6Hz, 2H ) 7.11 (d, *J* =7.2 Hz, 2H ) 6.81(t, *J* =8.4 Hz, 1H 2.63 ( s , 3H).

**13C NMR (100 MHz, CDCl3, δ ppm):** 197.40, 163.24(dd, *J*=260,235 Hz),143.06, 143.04, 142.92, 136.67, 128.97, 127.08,110.07(dd, *J=*26,12Hz), 103.34(t, *J*=25Hz) 26.60.

**19F NMR (376 MHz, CDCl3, δ ppm):** δ-57.82

**1-(3',5'-dichloro-[1,1'-biphenyl]-4-yl)ethan-1-one**



**C14H10Cl2O Melting Point**:95.8℃-97.0℃

**MW**: 265.13 g·mol-1 White Solid

**Isolated amounts**:217.69 mg **Yield**: 85%

**1H NMR (400 MHz, CDCl3, δ ppm):** 8.04 ( d, *J* =8.0 Hz, 2H), 7.63 (d, *J* =8.0 Hz, 2H ) 7.48( s, 2 H) , 7.39 ( s , 1H), 2.65 ( s , 3H).

**13C NMR (100 MHz, CDCl3, δ ppm):** 197.48, 142.84, 142.78, 136.71, 135.48, 129.04, 128.00, 127.22, 125.73, 26.70.

**1,1'-([1,1'-biphenyl]-3,4'-diyl)bis(ethan-1-one)**



**C16H14O2 Melting Point**:101.4℃-101.8℃

**MW**: 238.10 g·mol-1 White Solid

**Isolated amounts**:188.10 mg **Yield**: 79%

**1H NMR (400 MHz, CDCl3, δ ppm):** 8.16 ( s, 1H), 7.99 (d, *J* =7.6 Hz, 2H), 7.92 (d, *J* =7.6 Hz, 1H) , 7.6 (d, *J* =7.6 Hz, 1H) , 7.65 (d, *J* =7.6 Hz, 2H) ,7.51 ( t, *J* =7.6 Hz 1H), 2.61( s , 3H) , 2.59( s , 3H)

**13C NMR (100 MHz, CDCl3, δ ppm):** 197.83, 197.64, 144.59, 140.35, 137.67, 136.20, 131.74, 129.23, 128.98, 128.09, 127.28, 126.91, 26.76, 26.69.

**4'-acetyl-[1,1'-biphenyl]-3-carbonitrile**



**C15H11NO Melting Point**:133.3℃-134.3℃

**MW**: 221.08 g·mol-1 White Solid

**Isolated amounts**:174.65 mg **Yield**: 79%

**1H NMR (400 MHz, CDCl3, δ ppm):** 8.06 ( d, *J* =8 Hz, 2H) 1H), 7.88 (s, 1H), 7.84 (d, *J* =8Hz, 1H) , 7.66 (t, *J* =8.6Hz, 3H) , 7.58 (t, *J* =7.8 Hz, 1H)) , 2.65( s , 3H)

**13C NMR (100 MHz, CDCl3, δ ppm):** 197.47, 143.18, 141.11, 136.67, 131.55, 130.78, 129.81, 129.13, 127.25, 118.53, 113.19, 26.72.

**HRMS (ESI)** Calcd for C15H11NO+H 222.0922, Found 222.0919



**1-(4-(benzofuran-2-yl)phenyl)ethan-1-one**

**C16H12O2 Melting Point**:165.9℃-168.0℃

**MW**: 236.27 g·mol-1 White Solid

**Isolated amounts**:186.65 mg **Yield**: 79%

**1H NMR (400 MHz, CDCl3, δ ppm):** 8.04 ( d, *J* =8.0 Hz, 2H), 7.95 (d, *J* =8.0 Hz, 2H ) 7.62( d , *J* =7.6 Hz, 1H) , 7.55( d , *J* =8.0 Hz, 1H) 7.34 ( t , *J* =7.6 Hz, 1H) , 7.28-7.24( m , 1H) 7.17 ( s , 1H), 2.64( s , 3H)

**13C NMR (100 MHz, CDCl3, δ ppm):** 197.36, 155.17, 154.50, 136.48, 134.57, 128.92, 128.85, 125.13, 124.76, 123.23, 121.31, 111.34, 103.64, 26.65.



**1-(4'-phenoxy-[1,1'-biphenyl]-4-yl)ethan-1-one**

**C20H12O2 Melting Point**:128.1℃-128.5℃

**MW**: 288.35 g·mol-1 White Solid

**Isolated amounts**:210.50 mg **Yield**: 73%

**1H NMR (400 MHz, CDCl3, δ ppm):** 8.03 ( d, *J* =7.6 Hz, 2H), 7.66 ( d, *J* =7.2Hz, 2H) 7.60 ( d, *J* =7.2 Hz, 2H) 7.38 (t, *J* =7.4 Hz, 2H), 7.15 (t, *J* =7.4 Hz, 1H) , 7.09 (t, *J* =8.0 Hz, 4H), 2.64 ( s , 3H).

**13C NMR (100 MHz, CDCl3, δ ppm):** 197.72, 157.74, 156.66, 145.01, 135.51, 134.58, 129.83, 128.93, 128.57, 126.81, 123.66, 119.22, 118.91, 26.63.

**HRMS (ESI)** Calcd for C20H12O2+H 289.1230, Found 289.1229

**1-(4-(naphthalen-1-yl)phenyl)ethan-1-one**



**C18H14O Melting Point**:105.3℃-105.8℃

**MW**: 246.31 g·mol-1 White Solid

**Isolated amounts**:224.14 mg **Yield**: 91%

**1H NMR (400 MHz, CDCl3, δ ppm):** 8.10 ( d, *J* =7.6 Hz, 2H) , 7.92 (t, *J* =9.2 Hz, 2H), 7.85 (d, *J* =8.4 Hz, 1H) , 7.61 (d, *J* =7.6 Hz, 2H) , 7.57-7.50 (m, 2H) ,7.45 ( q , *J* =5.2 Hz 2H), 2.69( s , 3H)

**13C NMR (100 MHz, CDCl3, δ ppm):** 197.90, 145.77, 138.97, 135.93, 133.74, , 131.15, 130.30, 128.40, 128.35, 128.33, 126.91, 126.36, 125.98, 125.54, 125.32, 26.74.

**1-(4-(naphthalen-2-yl)phenyl)ethan-1-one**



**C18H14O Melting Point**:137.8℃-138.5℃

**MW**:246.31 g·mol-1 White Solid

**Isolated amounts**:221.70 mg **Yield**: 90%

**1H NMR (400 MHz, CDCl3, δ ppm):** 8.08 ( d, *J* =9.2 Hz, 2H), 7.96-7.88 (m, 3H ) 7.82 ( d, *J* =7.6Hz, 2H) 7.76 ( d, *J* =8.4Hz, 1H) 7.56-7.50(m, 2 H), 2.66 ( s , 3H).

**13C NMR (100 MHz, CDCl3, δ ppm):** 197.80, 145.64, 137.08, 135.78, 133.49, 132.96, 128.67, 128.31, 127.65, 127.42, 126.53, 126.46, 126.34, 125.13, 26.69.

**HRMS (ESI)** Calcd for C18H14O+H 247.1128, Found 247.1123

**4-(4-phenylnaphthalen-1-yl)benzonitrile**



**C23H15N Melting Point**:

**MW**: 305.38 g·mol-1 Yellow Solid

**Isolated amounts**:171.01 mg **Yield**: 56%

**1H NMR (400 MHz, CDCl3, δ ppm):** 8.01-7.99 (m, 1H),7.856-7.81(m, 3H),7.67 (d, *J* =8.0 Hz, 2H),7.53-7.44(m, 9H),

**13C NMR (100 MHz, CDCl3, δ ppm):** 145.69, 140.97, 140.31, 137.65, 133.37, 132.60, 132.14, 131.91, 131.22, 130.84, 130.00, 128.35, 127.49, 126.67, 126.52, 126.42, 126.33, 126.20, 125.50, 118.92, 111.09



**4'-morpholino-[1,1'-biphenyl]-4-carbonitrile**

**C17H16N2O Melting Point**:183.6℃-187.0℃

**MW**: 264.33 g·mol-1 White Solid

**Isolated amounts**: 162.24mg **Yield**: 61%

**1H NMR (400 MHz, CDCl3, δ ppm):** 7.65 (q ,*J*=6.8 Hz, 4H),7.53, (q, *J* =7.6 Hz, 2H), 6.99 (d, *J* =7.6 Hz, 2H),3.88 (s, 4H), 3.24(s,4H).

**13C NMR (100 MHz, CDCl3, δ ppm):** 151.40, 145.07, 132.49, 129.80, 127.90, 126.68, 119.16, 115.44,109.64, 66.69, 48.54.

**4'-(4-propylcyclohexyl)-[1,1'-biphenyl]-4-carbonitrile**



**C22H25N Melting Point**:129.8℃-131.2℃

**MW**:303.45g·mol-1 White Solid

**Isolated amounts**:267.03 mg **Yield**: 88%

**1H NMR (400 MHz, CDCl3, δ ppm):** 7.69 (q,*J*=6.8 Hz, 4H), 7.53(d, *J* =7.2 Hz, 2H), 7.33(d, *J* =7.2 Hz, 2H), 2.54 (t, *J* =12 Hz, 1H), 1.92 (t, *J* =13.2 Hz, 4H), 1.49 (q, *J* =12.4 Hz, 2H),1.40-1.33(m,3H), 1.24 (q, *J* =7.6 Hz, 2H), 1.08 (q, *J* =12 Hz, 2H), 0.92(t, *J* =7.0Hz, 3H).

**13C NMR (100 MHz, CDCl3, δ ppm):** 148.67, 145.54, 136.54, 132.49, 127.58, 127.42, 127.06, 119.02, 110.42, 44.29, 39.64, 36.93, 34.21, 33.44, 20.00, 14.39.

**1-(5'-phenyl-[1,1':3',1''-terphenyl]-4-yl)ethan-1-one**



**C26H20O Melting Point**:140.8℃-142.7℃

**MW**: 348.45 g·mol-1 White Solid

**Isolated amounts**:233.46 mg **Yield**: 67%

**1H NMR (400 MHz, CDCl3, δ ppm):** 8.08 ( d, *J* =7.2 Hz, 2H),7.84(s, 1H) 7.80 (d , *J* =8.0 Hz, 4H) 7.71( d, *J* =7.6 Hz, 4 H) , 7.51 (t, *J* =7.4 Hz, 4 H), 7.42 (d, *J* =7.2 Hz, 2H), 2.67( s , 3H).

**13C NMR (100 MHz, CDCl3, δ ppm):** 197.73, 145.64, 142.57, 140.96, 140.80, 136.02, 128.96, 128.89, 127.70, 127.42, 127.31, 136.06, 125.16, 26.70.

**4'-methoxy-[1,1'-biphenyl]-4-carbonitrile**



**C14H11NO Melting Point**:102.4℃-104.8℃

**MW**: 209.25 g·mol-1 White Solid

**Isolated amounts**:192.51 mg **Yield**: 92%

**1H NMR (400 MHz, CDCl3, δ ppm):**7.66 (d, *J* = 7.6 Hz, 2H), 7.61 (d, *J* = 7.6 Hz, 2H), 7.53 (d, *J* = 8.0 Hz, 2H), 7.00 (d, *J* = 7.6 Hz, 2H), 3.85 (s, 3H).

**13C NMR (100 MHz, CDCl3, δ ppm):**160.02, 145.95, 132.37, 131.20, 128.17, 126.88, 118.96, 114.36, 109.84, 55.21.

**4-methoxy-4'-nitro-1,1'-biphenyl**



**C13H11NO3 Melting Point**:109.2℃-109.6℃

**MW**: 229.24 g·mol-1 Yellow Solid

**Isolated amounts**:164.71 mg **Yield**: 64%

**1H NMR (400 MHzCDCl3, δ ppm):**8.27 (d, *J*=8.0 Hz, 2H), 7.69 (d, *J*=8.0 Hz, 2H), 7.58 (d, J=8.0 Hz, 2H ), 7.02 (d, J, =7.6 Hz, 2H), 3.88 (s , 3H),

**13C NMR (100 MHz, CDCl3, δ ppm):**160.38, 147.15, 146.46, 131.00, 128.53, 127.02, 124.11, 114.55, 55.39.

**HRMS (ESI)** Calcd for C13H11NO3+H230.0817, Found 230.0817

**4'-methoxy-[1,1'-biphenyl]-4-carbaldehyde**



**C14H12O2 Melting Point**:86.3℃-86.9℃

**MW**: 212.25 g·mol-1 White Solid

**Isolated amounts**:199.52 mg **Yield**: 94%

**1H NMR (400 MHz, CDCl3, δ ppm):**10.02, (s, 1H), 7.91 (d, *J*=7.6Hz, 2H), 7.70(d, *J*=7.6, 2H), 7.58 (d, *J*=7.6Hz, 2H), 7.00 (d, *J* = 7.6 Hz, 2H), 3.85 (s, 3H).

**13C NMR (100 MHz, CDCl3, δ ppm):**191.84, 160.00, 146.66,134.54, 131.90, 130.23, 128.41, 126.94, 114.38, 55.30.

**4-chloro-4'-methoxy-1,1'-biphenyl**



**C13H11ClO Melting Point**:113.8℃-114.4℃

**MW**: 218.68 g·mol-1 White Solid

**Isolated amounts**:194.63 mg **Yield**: 89%

**1H NMR (400 MHz, CDCl3, δ ppm):**7.46 (d, *J*=7.0 Hz, 4H), 7.36(d, *J*=7.6 Hz, 2H), 6.96 (d , *J*=7.6 Hz, 2H), 3.83 (s, 3H).

**13C NMR (100 MHz, CDCl3, δ ppm):**159.29, 139.19, 132.60, 132.40, 128.79, 127.96, 127.88, 114.25, 55.31.

**1-(4'-methoxy-[1,1'-biphenyl]-4-yl)pentan-1-one**



**C18H20O2 Melting Point**:119.4℃-119.9℃

**MW**: 268.36 g·mol-1 White Solid

**Isolated amounts**:214.52 mg **Yield**: 90%

**1H NMR (400 MHz, CDCl3, δ ppm):** 7.99 (d, *J*=7.6Hz, 2H), 7.61 (d, *J*=8.0, 2H), 7.55 (d, *J* = 7.6 Hz, 2H), 6.98 (d, *J*=7.6 Hz, 2H), 3.83 (s, 3H), 2.96( t, *J*=7.6, 2H), 1.77-1.69(m, 2H), 1.46-1.37(m 2H), 0.96, ( t ,J=7.4, 3H),

**13C NMR (100 MHz, CDCl3, δ ppm):**199.98, 159.74, 144.94, 135.04, 132.11, 128.57, 128.20, 126.43, 55.22, 38.22, 26.48, 22.43, 13.90.

**4'-methoxy-3,5-dimethyl-[1,1'-biphenyl]-2-amine**



**C15H17NO Melting Point**:70.3℃-70.8℃

**MW**: 227.31 g·mol-1 White Solid

**Isolated amounts**:170.48 mg **Yield**: 75%

**1H NMR (400 MHz, CDCl3, δ ppm):**7.34 (d, *J*=7.6 Hz, 2H), 6.95(d, *J*=7.6 Hz, 2H), 6.86 (s,1 H) ,6.08(s,1H) 3.81 (s, 3H), 3.51 (s, 2H), 2.24, (s, 3H), 2.17 (s, 3H).

**13C NMR (100 MHz, CDCl3, δ ppm):**158.53, 139.14, 132.08, 130.18, 128.66, 127.04, 122.48, 114.02, 55.15, 20.30, 17.79 .

**4'-methoxy-N,N-dimethyl-[1,1'-biphenyl]-4-amine**



**C15H17NO Melting Point**:151.5℃-151.9℃

**MW**: 227.31 g·mol-1 White Solid

**Isolated amounts**:163.66 mg **Yield**: 72%

**1H NMR (400 MHz, CDCl3, δ ppm)** 7.48(t, *J*=9.2, 4H), 6.94 (d, *J*=8.0Hz, 2H), 6.79(d, *J*=8.0, 2H), 3.83 (s, 3H), 2.97 (s, 6H)

**13C NMR (100 MHz, CDCl3, δ ppm):**158.20, 149.56, 133.92, 129.11, 127.28, 114.07, 112.88, 55.31, 40.66.

**4-ethyl-4''-methoxy-1,1':4',1''-terphenyl**



**C21H20O Melting Point**:234.6℃-235.6℃

**MW**: 288.39 g·mol-1 White Solid

**Isolated amounts**:236.48 mg **Yield**: 82%

**1H NMR (400 MHz, CDCl3, δ ppm):**7.64 (t, *J* = 9.6 Hz, 4H), 7.58 (t, *J* = 6.4 Hz, 4H), 7.30 (d, *J* = 7.6 Hz, 2H), 7.00 (d, *J*=8.0 Hz, 2H), 3.87 (s, 3H), 2.72 (q, *J* = 7.6Hz, 2H), 1.30 (t, *J* = 7.8 Hz, 3H).

**13C NMR (100 MHz, CDCl3, δ ppm):**159.13, 143.37, 139.43, 139.40, 138.10, 133.26, 128.30, 128.00, 127.27, 126.97, 126.88, 114.21, 55.34, 28.52, 15.59 .

**2-(4-methoxyphenyl)naphthalene**



**C17H14O Melting Point**:135.9℃-136.2℃

**MW**: 234.30 g·mol-1 White Solid

**Isolated amounts**:217.90 mg **Yield**: 93%

**1H NMR (400 MHz, CDCl3, δ ppm):**8.02, (s, 1H), , 7.90 (q, *J*=6.4 Hz, 2H), 7.75(d, *J*=8.8HZ, 1H), 7.69 (d, *J*=7.6HZ, 2H), 7.54-7.47 (m, H),7.05 (d, *J*=7.6 Hz, 2H) 3.89 (s, 3H).

**13C NMR (100 MHz, CDCl3, δ ppm):**159.17, 138.07, 13.69, 133.54, 132.24, 128.37, 128.30, 128.01, 127.58, 126.19, 125.60, 125.38, 124.97, 114.25, 55.32.

**(4'-methoxy-[1,1'-biphenyl]-4-yl)(phenyl)methanone**



**C20H16O2 Melting Point**:165.7℃-166.6℃

**MW**: 288.35 g·mol-1 White Solid

**Isolated amounts**:242.21 mg **Yield**: 84%

**1H NMR (400 MHz, CDCl3, δ ppm):**7.88 (d, *J* = 7.6 Hz, 2H), 7.84 (d, *J* = 7.6 Hz, 2H), 7.67 (d, *J* = 7.6 Hz, 2H), 7.61 (d, *J* = 7.6 Hz, 2H) 7.50 (t, *J* = 7.2 Hz, 2H), 7.02 (d, *J* = 8.0 Hz, 2H), 3.87 (s, 3H).

**13C NMR (100 MHz, CDCl3, δ ppm):**183.32, 159.82, 144.80, 137.80, 135.51, 132.26, 130.77, 129.93, 128.35, 128.24, 126.32, 114.37, 55.35.

**5-(4-methoxyphenyl)-2,3-dihydro-1H-inden-1-one**



**C16H14O2 Melting Point**: 154.6℃-155.1℃

**MW**: 238.39 g·mol-1 White Solid

**Isolated amounts**:202.63 mg **Yield**: 85%

**1H NMR (400 MHz, CDCl3, δ ppm):**7.78 (d, *J* =8.0 Hz, 1H),7.61 (s,1H), 7.56 (t, *J* =7.6 Hz, 3H), 6.99 (d, *J* =7.6 Hz, 2H), 3.85 (s,3H) 3.16 (t, *J* =5 Hz, 2H), 2.71 (t, *J* = 5 Hz, 2H).

**13C NMR (100 MHz, CDCl3, δ ppm):**206.52, 159.88, 155.88, 147.14, 135.32, 132.40, 128.49, 128.13, 124.35, 123.94, 114.30, 55.30, 36.44, 25.78

**HRMS (ESI)** Calcd for C16H14O2+H 239.1076, Found 239.1072

**2-(4-methoxyphenyl)dibenzo[b,d]thiophene**



**C19H14OS Melting Point**:163.3℃-168.5℃

**MW**: 290.38 g·mol-1 White Solid

**Isolated amounts**:209.07 mg **Yield**: 72%

**1H NMR (400 MHz, CDCl3, δ ppm):**8.35, (s, 1H), 8.25-8.23 (m, 1H), 7.90 (d, *J*=8.4Hz, 2H), 7.68(d, *J*=8.4Hz, 3H), 7.52-7.50 (m, 2H), 7.07 (d, *J* = 7.6 Hz, 2H), 3.90 (s, 3H).

**13C NMR (100 MHz, CDCl3, δ ppm):**159.02, 139.79, 137.71, 137.34, 125.95, 135.44, 133.49, 128.22, 126.67, 125.74, 124.25, 122.81, 122.77, 121.49, 119.38, 114.18, 55.20

**2-(4-methoxyphenyl)benzo[b]thiophene**



**C15H12OS Melting Point**:191.0℃-193.1℃

**MW**: 240.32 g·mol-1 White Solid

**Isolated amounts**:180.24 mg **Yield**: 75%

**1H NMR (400 MHz, CDCl3, δ ppm):** 7.80 (d, *J*=7.6, 1H), 7.74 (d, *J* = 8.0 Hz, 1H), 7.64 (d, *J*=7.6 Hz, 2H), 7.43(s, 1H), 7.35-7.25(m,2H), 6.96(d, *J*=8, 2H), 3.85(s,3H).

**13C NMR (100 MHz, CDCl3, δ ppm):**159.74, 144.10, 140.85, 139.13, 127.71, 127.00, 124.42, 123.22, 122.15, 118.16, 114.31, 55.38.

**6-(4-methoxyphenyl)benzo[b]thiophene**



**C15H12OS Melting Point**:138.2℃-138.9℃

**MW**: 240.32 g·mol-1 White Solid

**Isolated amounts**:213.78 mg **Yield**: 89%

**1H NMR (400 MHz, CDCl3, δ ppm):** 7.99 (s ,1H), 7.92 (d, *J*=8.4, 1H), 7.55 (d, *J* = 7.6 Hz, 2H), 7.61 (d, *J*=8.0 Hz, 2H), 7.57(d, *J*=8.4 Hz, 1H), 7.48(d, *J*=5.2, 1H), 7.38(d, *J*=5.2, 1H), 7.02(d, *J*=8.0, 2H), 3.88(s,3H).

**13C NMR (100 MHz, CDCl3, δ ppm):**159.00, 140.18, 138.15, 137.28, 133.82, 128.36, 126.92, 123.99, 123.65, 122.61, 121.45, 114.21, 55.34.

**1-(6-(4-methoxyphenyl)pyridin-3-yl)ethan-1-one**



**C14H13NO2 Melting Point**:102.6℃-103.6℃

**MW**: 227.26 g·mol-1 White Solid

**Isolated amounts**: 106.81 mg **Yield**: 47%

**1H NMR (400 MHz, CDCl3, δ ppm):**9.18 (s, 1H), 8.24 (d, *J*=8.4 Hz, 1H), 8.04(d, *J*=8.0Hz, 2H), 7.77 (d, *J*=8.4Hz,2H),7.01(d, *J*=8.0HZ, 2H), 3.88 (s, 3H).2.64 (s, 3H).

**13C NMR (100 MHz, CDCl3, δ ppm):**196.47, 161.33, 160.50, 150.10, 136.25, 130.62, 129.88, 128.78, 119.18, 114.27, 55.38, 26.66

**5-(4-methoxyphenyl)-1-methyl-1H-indole**



**C16H15NO Melting Point**:127.9℃-128.3℃

**MW**: 237.30 g·mol-1 YellowSolid

**Isolated amounts**:132.89 mg **Yield**: 56%

**1H NMR (400 MHz, CDCl3, δ ppm):**7.88, (s, 1H), 7.66 (d, *J*=7.6Hz, 2H), 7.52 (d, *J*=8.4, 1H), 7.42 (d, *J* = 8.8 Hz, 1H), 7.12 (s, 1 H),7.06 (d, *J*=7.6 Hz, 2H), 6.60 (s, 1 H), 3.91 (s, 3 H), 3.83 (s, 3 H).

**13C NMR (100 MHz, CDCl3, δ ppm):**158.33, 135.88, 135.16, 132.39, 129.33, 128.87, 128.22, 121.08. 118.78, 114.01, 109.32, 101.08, 55.25, 32.80.

**4-methoxy-5'-phenyl-1,1':3',1''-terphenyl**



**C25H20O Melting Point**:137.0℃-137.2℃

**MW**: 336.43 g·mol-1 White Solid

**Isolated amounts**:272.51 mg **Yield**: 81%

**1H NMR (400 MHz, CDCl3, δ ppm):**7.81 (s, 3H) 7.75 (d, *J*=7.6, 4H), 7.69 (d, *J* = 7.6 Hz, 2H), 7.53 (t, *J*=7.4 Hz, 4H), 7..44 (t, *J*=7.2 Hz, 2H), 7.07(d, *J*=7.6, 2H), 3.90 (s,3H).

**13C NMR (100 MHz, CDCl3, δ ppm):**159.29, 142.25, 141.85, 141.17, 133.54, 128.78, 127.48, 127.31, 124.71, 124.58, 114.22, 55.30.

**4'-methoxy-N,N-diphenyl-[1,1'-biphenyl]-4-amine**



**C25H21NO Melting Point**:130.7℃-131.8℃

**MW**: 351.45 g·mol-1 White Solid

**Isolated amounts**:224.93 mg **Yield**: 64%

**1H NMR (400 MHz, CDCl3, δ ppm):**7.49 (d, *J* = 7.6 Hz, 2H ), 7.42 (d, *J* = 7.6 Hz, 2H ), 7.24(t, *J* = 6.6 Hz 4H), 7.11(d, *J* = 8.0 Hz,, 6H), 7.00 (t , *J* = 7.4 Hz 2H), 6.94 (d, *J* = 8.0 Hz, 2H), 3.82 (s, 3H).

**13C NMR (100 MHz, CDCl3, δ ppm):**158.74, 147.69, 146.52, 134.92, 133.18, 129.20, 127.63, 127.29, 124.17, 122.70, 114.12, 55.28.

**9-(4'-methoxy-[1,1'-biphenyl]-4-yl)-9H-carbazole**



**C25H19NO Melting Point**:269.1℃-269.5℃

**MW**: 349.43 g·mol-1 White Solid

**Isolated amounts**:251.59mg **Yield**: 72%

**1H NMR (400 MHz, CDCl3, δ ppm):** 8.17 (d, *J*=8.0, 2H), 7.78 (d, *J* = 7.2 Hz, 2H), 7.63 (t, *J*=8.0 Hz, 4H), 7.49-7.41(m, 4H), 7.31 (t, *J*=7.2 Hz, 2H), 7.05(d, *J*=7.6, 2H), 3.90 (s,3H).

**13C NMR (100 MHz, CDCl3, δ ppm):**159.37, 140.84, 139.89, 136.19, 132.74, 128.15, 127.98, 127.29, 125.90, 123.32, 120.28, 119.87, 114.35,

**HRMS (ESI)** Calcd for C25H19NO+H 350.1548, Found 350.1545

**1-(4'-methoxy-[1,1'-biphenyl]-4-yl)naphthalene**



**C23H18O Melting Point**:122.0℃-123.0℃

**MW**:310.40 g·mol-1 White Solid

**Isolated amounts**:192.45 mg **Yield**: 62%

**1H NMR (400 MHz, CDCl3, δ ppm):**8.01, (d,*J*=8.4 Hz, 1H), 7.94 (q, *J*=8.0 Hz, 1H), 7.89(d, *J*=8.0Hz, 1H), 7.70 (d, *J*=7.6HZ, 2H),7.65(d, *J*=7.6HZ, 2H), 7.59-7.45 (m,6 H),7.04 (d, *J*=7.6 Hz, 2H) 3.89 (s, 3H).

**13C NMR (100 MHz, CDCl3, δ ppm):**159.18, 139.88, 139.66, 139.06, 133.79, 133.29, 131.57, 130.44, 128.27, 128.11, 127.61, 126.90, 126.51, 126.02, 125.77, 125.40, 114.25, 55.34.

**4'-methoxy-[1,1'-biphenyl]-2-ol**



**C13H12O2**

**MW**: 200.34 g·mol-1 Yellow Oil

**Isolated amounts**:144.24 mg **Yield**: 72%

**1H NMR (400 MHz, CDCl3, δ ppm):**7.37 (d, *J*=7.6 Hz, 2H), 7.21(d, *J*=10.8 Hz, 2H), 6.96 (q , *J*=6.4 Hz, 2H), 5.36 (s, 1H). 3.80 (s, 3H).

**13C NMR (100 MHz, CDCl3, δ ppm):**159.07, 152.40, 130.20, 130.17, 129.19, 128.68, 127.76, 120.70, 115.63, 114.51, 55.23

**4'-methoxy-[1,1'-biphenyl]-4-ol**



**C13H12O2 Melting Point**:169.6℃-170.4℃

**MW**: 200.34 g·mol-1 White Solid

**Isolated amounts**:152.26 mg **Yield**: 76%

**1H NMR (400 MHz, DMSO d-6, δ ppm):**9.47 (s,1H), 7.49(d, *J*=7.6 Hz, 2H),7.42 (d , *J*=7.6 Hz, 2H), 6.97(d, *J*=7.6 Hz, 2H),6.83 (d , *J*=7.6 Hz, 2H), 3.77 (s, 3H).

**13C NMR (100 MHz, DMSO d-6 δ ppm):**158.54, 156.97, 133.20, 131.18, 127.68 127.46, 116.09, 114.67, 55.54

**4-methoxy-4'-methyl-1,1'-biphenyl**



**C14H14O Melting Point**:109.0℃-109.4℃

**MW**: 198.27 g·mol-1 White Solid

**Isolated amounts**:109.05 mg **Yield**: 55%

**1H NMR (400 MHz, CDCl3, δ ppm):**7.51 (d, *J*=7.2 Hz, 2H), 7.45(d, *J*=7.6 Hz, 2H), 7.22(d, *J*=7.2Hz, 2H), 6.96 (d, *J*=7.6 HZ, 2H), 3.84 (s, 3H), 2.38 (s, 3H).

**13C NMR (100 MHz, CDCl3, δ ppm):**158.86, 137.91, 136.32, 133.69, 129.40, 127.92,126.55, 114.10, 55.31, 21.04.

**2-(4-methoxyphenyl)-4a,9a-dihydroanthracene-9,10-dione**



**C21H15O3 Melting Point**:175.8℃-176.7℃

**MW**: 314.34 g·mol-1 YellowSolid

**Isolated amounts**:298.62 mg **Yield**:95%

**1H NMR (400 MHz, CDCl3, δ ppm):**8.48 (s, 1H), 8.33 (d, *J* = 7.2 Hz, 3H), 7.97 (d, *J* = 8.0 Hz, 1H), 7.81-7.80 (m, 2H), 7.69 (d, *J* = 8.0 Hz, 2H), 7.03 (d, *J* = 8.4 Hz, 2H), 3.88 (s, 3H).

**13C NMR (100 MHz, CDCl3, δ ppm):**183.32, 182.83, 160.36, 146.37, 134.12, 133.95, 133.84, 133.64, 133.57, 131.66, 131.51, 131.21, 128.48. 128.03, 127.22, 127.15, 124.79, 114.54, 55.40

**HRMS (ESI)** Calcd for C21H15O3+H 315.1025, Found 315.1021

**1-(4'-(diphenylamino)-[1,1'-biphenyl]-4-yl)ethan-1-one**



**C26H21NO Melting Point**:105.8℃-106.3℃

**MW**: 363.46 g·mol-1 Green Solid

**Isolated amounts**:210.80 mg **Yield**: 58%

**1H NMR (400 MHz, DMSO, δ ppm):** 7.91 ( d, *J* =8.0 Hz, 2H), 7.56, (d, *J* =7.6 Hz, 2H),7.41 (d, *J* =8.0 Hz, 2H), 7.19 (t ,*J*=7.4 Hz, 4H), 7.05(d, *J* =8.4 Hz, 6H) 6.97 (t ,*J*=7.2 Hz, 2H), 2.53( s , 3H).

**13C NMR (100 MHz, DMSO, δ ppm):** 197.65, 148.09, 147.31, 145.11, 135.20, 133.01, 129.32, 128.91, 127.85, 126.41, 124.70, 123.29, 123.20, 26.60.

**HRMS (ESI)** Calcd for C26H21NO+H 364.700, Found 364.1701

**9-(4-methoxyphenyl)-3,6a-dihydroperylene**



**C27H20O Melting Point**:255.3℃-257.0℃

**MW**: 360.46 g·mol-1 YellowSolid

**Isolated amounts**:273.95 mg **Yield**:76%

**1H NMR (400 MHz, CDCl3, δ ppm):**8.19 (t, *J*=5.8 Hz, 4H), 7.79 (d, *J*=8.4 Hz, 1H), 7.68(d, *J*=8.0Hz, 2H), 7.50-7.39(m, 6H),7.05(d, *J*=8.0HZ, 2H), 3.90 (s, 3H).

**13C NMR (100 MHz, CDCl3, δ ppm):**158.96, 139.59, 134.65, 133.06, 131.41, 131.33, 131.26, 131.00, 130.26, 129.05, 128.60 ,127.70, 127.67, 127.59, 126.57, 126.55, 126.37, 126.10, 120.28, 120.23, 119.96, 119.82, 113.78, 55.34.

**9,10-bis(4-methoxyphenyl)anthracene**



**C28H22O2  Melting Point**:277.9℃-279.1℃

**MW**:390.48 g·mol-1 White Solid

**Isolated amounts**:144 mg **Yield**: 74%

**1H NMR (400 MHz, CDCl3, δ ppm):**7.75-7.73(m,4H), 7.39 (d , *J*=7.6 Hz, 4H), 7.33 (d , *J*=7.2 Hz, 4H), 7.14 (d , *J*=7.6 Hz, 4H), 3.96 (s, 6H).

**13C NMR (100 MHz, CDCl3, δ ppm):**159.26, 158.97, 140.46, 136.72, 132.37, 131.12, 130.23, 128.66, 127.56, 127.02, 124.87, 123.38, 113.83, 113.73, 55.38

**4,7-bis(4-methoxyphenyl)benzo[c][1,2,5]thiadiazole**



**C20H18N2O5  Melting Point**:196.7℃-197.0℃

**MW**: 348.42 g·mol-1 Yellow Solid

**Isolated amounts**:151.56 mg **Yield**: 87%

**1H NMR (400 MHz, CDCl3, δ ppm):**7.92(d, *J*=7.6 Hz, 4H),7.71(s, 2H) 7.08 (d , *J*=7.6 Hz, 4H),3.89 (s, 6H).

**13C NMR (100 MHz, CDCl3, δ ppm):**159.71, 154.16, 132.28, 130.34, 129.96, 127.37, 114.06, 55.38.

**HRMS (ESI)** Calcd for C20H18N2O5+H 4349.1009, Found 349.1011

**A name could not be generated for this structure.**



**C54H38  Melting Point**:293.6℃-297.2℃

**MW**: 686.90 g·mol-1 White Solid

**Isolated amounts**: 216.37 mg **Yield**:63%

**1H NMR (400 MHz, CDCl3, δ ppm):** 7.85(s 4H), 7.83-7.78 (m, 14H) 7.73(d , *J*=7.6 Hz,2H), 7.50 (t , *J*=7.4 Hz, 8H), 7.41 (t , *J*=7.4 Hz, 4H),

**13C NMR (100 MHz, CDCl3, δ ppm):** 142.43, 141.77, 141.11, 140.12, 139.82, 139.64, 139.86, 127.77, 127.58, 127.48, 127.45, 127.36, 125.31, 125.03.

**1-(4'-(naphthalen-2-yl)-[1,1'-biphenyl]-4-yl)ethan-1-one**



**C24H18O Melting Point**:228.7℃-229.2℃

**MW**: 322.41 g·mol-1 White Solid

**Isolated amounts**:109.70 mg **Yield**: 66%

**1H NMR (400 MHz, CDCl3, δ ppm):** 8.11( s , 1H). 8.07 ( d, *J* =7.6 Hz, 2H), 7.94, (t, *J* =9.2 Hz, 2 H),7.89 (d , *J* =7.2 Hz, 1H), 7.85 (d , *J* =7.6 Hz, 2H), 7.78 (t, *J* =10.6 Hz, 5 H),7.55-7.49(m,2H) 2.66( s , 3H).

**13C NMR (100 MHz, CDCl3, δ ppm):** 197.75, 145.21, 140.99, 138.73, 137.65, 135.86, 133.63, 132.72, 128.99, 128.57, 128.22, 127.91, 127.72, 127.66, 127.08, 126.41, 126.12, 125.80, 125.30, 26.71.

**2,3,6,7,10,11-hexakis(4-methoxyphenyl)triphenylene**



**C60H48O6  Melting Point**:259.0℃-260.1℃

**MW**: 865.04 g·mol-1 White Solid

**Isolated amounts**:367.29 mg **Yield**: 85%

**1H NMR (400 MHz, DMSO d-6, δ ppm):**8.71 (s,5H), 7.27(d, *J*=8.0 Hz, 12H), 6.89 (d , *J*=8.0 Hz, 12H), 3.77 (s, 18H).

**13C NMR (100 MHz, DMSO d-6, δ ppm):**158.64, 139.69, 133.74, 131.45, 128.45, 125.82, 114.06, 55.52

**4'-methyl-[1,1'-biphenyl]-2-carbonitrile**



**C14H11N Melting Point**:48.9℃-49.1℃

**MW**: 193.25 g·mol-1 White Solid

**Isolated amounts**:139.14 mg **Yield**: 72%

**1H NMR (400 MHz, CDCl3, δ ppm):**7.70(d, *J*=7.6 Hz, 1H), 7.58 (t , *J*=7.6 Hz, 1H),7.45(t, *J*=8.2Hz, 3H)，7.37(t, *J*=7.6Hz, 1H)，7.27(d, *J*=7.6 Hz, 2H), 2.38（s,3H）

**13C NMR (100 MHz, CDCl3, δ ppm):**145.22, 138.44, 135.04, 133.48, 132.61, 129.76, 129.23, 128.40, 127.10, 118.71, 110.87, 21.05.

**1-(5-(tetrahydrofuran-3-yl)pyridin-2-yl)ethan-1-one**



**C11H13NO2  Melting Point**:117.8℃-118.9℃

**MW**:191.23 g·mol-1 Yellow Solid

**Isolated amounts**:120.47 mg **Yield**: 63%

**1H NMR (400 MHz, CDCl3, δ ppm):** 8.81（s,1H）8.05 (d, *J*=10.4 Hz, 1H), 7.87, (d , *J*=8.0 Hz, 2H),7.56(s, 1H), 6.77(s, 1H), 2.73（s,3H）

**13C NMR (100 MHz, CDCl3, δ ppm):**199.42, 1551.80, 145.98, 144.44, 139.86, 133.19, 131.62, 122.61, 121.78, 108.20, 25.64.

**HRMS (ESI)** Calcd for C11H13NO2+H 192.1025, Found 192.1025

**5'-chloro-2',4-difluoro-[1,1'-biphenyl]-2-carbonitrile**



**C13H6ClF2N Melting Point**:117.8℃-118.9℃

**MW**: 249.64 g·mol-1 Yellow Solid

**Isolated amounts**:199.71 mg **Yield**: 80%

**1H NMR (400 MHz, CDCl3, δ ppm):**7.48(t, *J*=9.2 Hz, 2H), 7.39 (q , *J*=9.6 Hz, 3H),7.17(t, *J*=9 Hz, 1H)

**13C NMR (100 MHz, CDCl3, δ ppm):**159.84(dd, *J=*634,137Hz)，134.40,(d, *J*=4Hz，132.79(dd, *J=*10,8 Hz)，130.90，130.85, 130.82,129.54(d, *J=*3 Hz)126.28,(d, *J*=16Hz)，120.27(dd, *J=*63,18 Hz)，117.58(d *J=* 24Hz),116.41(d, *J=*3 Hz) ,114.28(d, *J=*9 Hz)

**4-chloro-2',4'-difluoro-[1,1'-biphenyl]-3-carbonitrile**



**C13H6CIF2N**

**MW**: 249.64 g·mol-1 Colorless Oil

**Isolated amounts**:217.18 mg **Yield**: 87%

**1H NMR (400 MHz, CDCl3, δ ppm):** 7.80（s,1H）7.68(d, *J*=8.4 Hz, 1H), 7.59, (d , *J*=8.4 Hz, 1H),7.39(q, *J*=8.0 Hz, 1H),7.03-6.93 (m, 2H),

**13C NMR (100 MHz, CDCl3, δ ppm):**162.54(dd,*J*=356,332Hz),160.04(dd, *J*=356,338Hz) 136.07, 134.36, 134.05(dd,*J*=16,10Hz), 131.03(dd, *J*=14,5Hz),130.18, 121.95(dd,*J=*17,9Hz)115.68, 113.66, 112.22(dd,*J=*25,17Hz), 104.81(t, *J=* 26 Hz)

**4'-chloro-[1,1'-biphenyl]-2-amine**



**C12H10ClN**

**MW**: 230.67 g·mol-1 Yellow Oil

**Isolated amounts**:186.84 mg **Yield**: 81%

**1H NMR (400 MHz, CDCl3, δ ppm):** 7.40-7.36（m,4H）7.14(t, *J*=7.6 Hz, 1H), 7.07, (d , *J*=7.2 Hz, 1H), 6.81(t, *J*=7.6 Hz, 1H)6.73, (d , *J*=8.0 Hz, 1H) 3.66(s,2H).

**13C NMR (100 MHz, CDCl3, δ ppm):**143.25, 137.73, 132.82, 130.26, 130.15, 128.78, 128.65, 126.01, 118.55, 115.55,

**2-chloro-N-(4'-chloro-[1,1'-biphenyl]-2-yl)nicotinamide**



**C18H12Cl2N2O Melting Point**:140.4℃-141.6℃

**MW**: 343.21 g·mol-1 White Solid

**Isolated amounts**:302.02 mg **Yield**: 88%

**1H NMR (400 MHz, CDCl3, δ ppm):** 8.35-8.29（m,2H）8.04(s,1H), 8.03, (d , *J*=7.6 Hz, 1H), 7.38-7.34(m,3H)7.25 (d , *J*=7.2 Hz, 3H) 7.19-7.18(m,2H).

**13C NMR (100 MHz, CDCl3, δ ppm):**162.50, 151.17, 146.58, 139.96, 136.19, 134.30, 134.17, 132.29, 131.03, 130.71, 130.17, 129.19, 128.79, 125.33, 122.82, 122.18.

## **Optimization of the Reaction Conditions.**



## References

（1）Seo, T.; Toyoshima, N.; Kubota, K., Ito, H. Tackling Solubility Issues in Organic Synthesis: Solid-State Cross-Coupling of Insoluble Aryl Halides. *J. Am. Chem. Soc*. **2021**, *143*, 6165-6175.

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## Copies of the 1H NMR, 13C NMR

