y-Valerolactone as a renewable dipolar aprotic solvent deriving from biomass degradation for the Hiyama reaction

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Experimental Section

Unless otherwise stated, all solvents and reagents were used as obtained from commercial sources without further purification. GLC analyses were performed by using Hewlett-Packard HP 5890A equipped with a capillary column DB-35MS (30 m, 0.53 mm), a FID detector and hydrogen as gas carrier. GC-EIMS analyses were carried out by using a Hewlett-Packard HP 6890N Network GC system/5975 Mass Selective Detector equipped with an electron impact ionizer at 70 eV. NMR spectra were recorded on a Bruker DRX-ADVANCE 400 MHz (1H at 400 MHz and 13C at 100.6 MHz) in CDCl₃ using TMS as the internal standard. Elemental Analyses were conducted on a Fisons EA1108CHN. Melting points are not corrected and they were measured on a Büchi 510.

Compounds 3a, 3b, 3c, 3d, 3e, 3f, 3g, 3h, 3i, 3j, 3k, 3l, 3m are known compounds.

Characterization data and copies of the 1H and 13C NMR are reported below.

Reference:

**Chem. Name**: 4-nitro-1,1'-biphenyl (3a)

**Lit. Ref.**: *RSC Adv.*, 2012, 2, 590–594

![Chemical Structure](image)

**METHOD:**
In a screw capped vial equipped with a magnetic stirrer Pd/C 10% wt. (5.3 mg, 0.5 mmol%), GVL (1 mL), TBAF (488 mg, 1.5 mmol), 1-bromo-4-nitrobenzene (1a) (204 mg, 1 mmol), were consecutively added and the resulting mixture was degassed with argon. Then triethoxy(phenyl)silane (2a) (368 mg, 0.369 mL, 1.5 mmol) was added and the mixture was heated at 130 °C under an atmosphere of argon. After 24 h water was added, the formed precipitate was filtered off, washed with water and purified by column chromatography (petroleum ether : EtOAc / 95 : 5). 3a was obtained as a solid (165 mg, 83% yield).

**Mol Formula**: \( \text{C}_{12}\text{H}_9\text{NO}_2 \)

**m.p.**: 116-118 °C

**Elemental Analysis**: Calc.: C, 72.35; H, 4.55; N, 7.03. Found: C, 72.40; H, 4.65; N, 7.00

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**\(^{13}\text{C NMR} (100.6 \text{ MHz, CDCl}_3) \delta :****
\[ 124.2, 127.5, 127.9, 129.0, 129.3, 138.9, 147.2, 147.8 \]

**GC-EIMS (m/z, %):**
200 (10), 199 (M⁺, 83), 169 (44), 153 (31), 152 (100), 151 (33), 150 (11), 141 (40), 127 (11), 126 (11), 115 (19), 77 (12), 76 (14), 63 (13), 51 (12), 50 (10)
**Chem. Name**: 4-nitro-1,1'-biphenyl (3a)  
**Lit. Ref.**: *RSC Adv.*, 2012, 2, 590–594

![Chemical Reaction Diagram]

**METHOD:**  
In a screw capped vial equipped with a magnetic stirrer Pd/C 10% wt. (5.3 mg, 0.5 mmol%), GVL (1 mL), TBAF (488 mg, 1.5 mmol), 1-iodo-4-nitrobenzene (4a) (254 mg, 1 mmol), were consecutively added and the resulting mixture was degassed with argon. Then triethoxy(phenyl)silane (2a) (368 mg, 0.369 mL, 1.5 mmol) was added and the mixture was heated at 130 °C under an atmosphere of argon. After 24 h water was added, the formed precipitate was filtered off, washed with water and purified by column chromatography (petroleum ether : EtOAc / 95 : 5). 3a was obtained as a solid (155 mg, 78% yield).

**Mol Formula**: C_{12}H_{9}NO_{2}  
**m.p.**: 116-118 °C

**Elemental Analysis**: Calc.: C, 72.35; H, 4.55; N, 7.03. Found: C, 72.42; H, 4.67; N, 7.05

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**13C NMR (100.6 MHz, CDCl₃)** δ : 124.2, 127.5, 127.9, 129.0, 129.3, 138.9, 147.2, 147.8

**GC-EIMS (m/z, %)**: 200 (10), 199 (M⁺, 83), 169 (44), 153 (31), 152 (100), 151 (33), 150 (11), 141 (40), 127 (11), 126 (11), 115 (19), 77 (12), 76 (14), 63 (13), 51 (12), 50 (10)
**METHOD:**
In a screw capped vial equipped with a magnetic stirrer Pd/C 10% wt. (5.3 mg, 0.5 mmol%), GVL (1 mL), TBAF (488 mg, 1.5 mmol), bromobenzene (1b) (158 mg, 0.107 mL, 1 mmol), were consecutively added and the resulting mixture was degassed with argon. Then triethoxy(phenyl)silane (2a) (368 mg, 0.369 mL, 1.5 mmol) was added and the mixture was heated at 130 °C under an atmosphere of argon. After 24 h water was added, the formed precipitate was filtered off, washed with water and purified by column chromatography (petroleum ether). 3b was obtained as a solid (145 mg, 94% yield).

**Mol Formula**  
\[ \text{C}_{12}\text{H}_{10} \]  
**m.p.** 68-70 °C

**Elemental Analysis:** Calc.: C, 93.46; H, 6.54. Found: C, 93.50; H, 6.60

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| \( ^{13}\text{C NMR} (100.6 \text{ MHz, CDCl}_3) \ \delta \) | 127.3, 127.4, 128.9, 141.4 |

| \( \text{GC-EIMS (m/z, \%)} \) | 155 (13), 154 (M\(^+\), 100), 153 (40), 152 (29), 76 (11) |
In a screw capped vial equipped with a magnetic stirrer Pd/C 10% wt. (5.3 mg, 0.5 mmol%), GVL (1 mL), TBAF (488 mg, 1.5 mmol), iodobenzene (4b) (208 mg, 0.114 mL, 1 mmol), were consecutively added and the resulting mixture was degassed with argon. Then triethoxy(phenyl)silane (2a) (368 mg, 0.369 mL, 1.5 mmol) was added and the mixture was heated at 130 °C under an atmosphere of argon. After 24 h water was added, the formed precipitate was filtered off, washed with water and purified by column chromatography (petroleum ether). 3b was obtained as a solid (142 mg, 92% yield).
**Chem. Name**: 4-methyl-1,1'-biphenyl (3c)

**Lit. Ref.**: RSC Adv., 2012, 2, 590–594

**METHOD:**
In a screw capped vial equipped with a magnetic stirrer Pd/C 10% wt. (5.3 mg, 0.5 mmol%), GVL (1 mL), TBAF (488 mg, 1.5 mmol), 1-bromo-4-methylbenzene (**1c**) (174 mg, 1 mmol), were consecutively added and the resulting mixture was degassed with argon. Then triethoxy(phenyl)silane (**2a**) (368 mg, 0.369 mL, 1.5 mmol) was added and the mixture was heated at 130 °C under an atmosphere of argon. After 24 h water was added, the formed precipitate was filtered off, washed with water and purified by column chromatography (petroleum ether). **3c** was obtained as a solid (145 mg, 86% yield).

**Mol Formula**: C_{13}H_{12}  
**m.p.**: 45-47 °C

**Elemental Analysis**: Calc.: C, 92.81; H, 7.19. Found: C, 92.75; H, 7.27

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| **13C NMR (100.6 MHz, CDCl₃)** δ: 21.2, 127.09, 127.11, 128.8, 129.6, 137.1, 138.4, 141.3 |

**GC-EIMS (m/z, %)**: 169 (14), 168 (M⁺, 100), 167 (66), 165 (28), 153 (15), 152 (21), 115 (11)
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<th>Chem. Name</th>
<th>4-methyl-1,1'-biphenyl (3c)</th>
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**METHOD:**
In a screw capped vial equipped with a magnetic stirrer Pd/C 10% wt. (5.3 mg, 0.5 mmol%), GVL (1 mL), TBAF (488 mg, 1.5 mmol), 1-iodo-4-methylbenzene (4c) (220 mg, 1 mmol), were consecutively added and the resulting mixture was degassed with argon. Then triethoxy(phenyl)silane (2a) (368 mg, 0.369 mL, 1.5 mmol) was added and the mixture was heated at 130 °C under an atmosphere of argon. After 24 h water was added, the formed precipitate was filtered off, washed with water and purified by column chromatography (petroleum ether). 3c was obtained as a solid (135 mg, 80% yield).

<table>
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<th>Mol Formula</th>
<th>C_{13}H_{12}</th>
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**Elemental Analysis:** Calc.: C, 92.81; H, 7.19. Found: C, 92.74; H, 7.28

**\[^1\text{H}\text{ NMR 400 MHz CDCl}_3\]**

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**\[^{13}\text{C NMR (100.6 MHz, CDCl}_3\]** δ : 21.2, 127.09, 127.11, 128.8, 129.6, 137.1, 138.4, 141.3

**GC-EIMS (m/z, %):** 169 (14), 168 (M^+, 100), 167 (66), 165 (28), 153 (15), 152 (21), 115 (11)
**Chem. Name**: 1-phenylnaphthalene (3d)


**METHOD**: In a screw capped vial equipped with a magnetic stirrer Pd/C 10% wt. (5.3 mg, 0.5 mmol%), GVL (1 mL), TBAF (488 mg, 1.5 mmol), 1-bromonaphthalene (1d) (213 mg, 0.144 mL, 1 mmol), were consecutively added and the resulting mixture was degassed with argon. Then triethoxy(phenyl)silane (2a) (368 mg, 0.369 mL, 1.5 mmol) was added and the mixture was heated at 130 °C under an atmosphere of argon. After 24 h water was added, the formed precipitate was filtered off, washed with water and purified by column chromatography (petroleum ether). 3d was obtained as a solid (167 mg, 82% yield).

**Molecular Weight**: 204

**Mol Formula**: C₁₆H₁₂

**m.p.**: 44-46 °C

**Elemental Analysis**: Calc.: C, 94.08; H, 5.92. Found: C, 94.15; H, 5.85

**¹H NMR 400 MHz CDCl₃**

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**¹³C NMR (100.6 MHz, CDCl₃)** δ: 125.5, 125.9, 126.13, 126.14, 127.0, 127.3, 127.7, 128.37, 128.38, 130.2, 131.7, 133.9, 140.4, 140.9

**GC-EIMS (m/z, %)**: 204 (M⁺, 100), 203 (98), 202 (61), 201 (13), 200 (13), 101 (16)
Chem. Name  1-([1,1’-biphenyl]-4-yl)ethanone (3e)


METHOD:
In a screw capped vial equipped with a magnetic stirrer Pd/C 10% wt. (5.3 mg, 0.5 mmol%), GVL (1 mL), TBAF (488 mg, 1.5 mmol), 1-(4-bromophenyl)ethanone (1e) (203 mg, 1 mmol), were consecutively added and the resulting mixture was degassed with argon. Then triethoxy(phenyl)silane (2a) (368 mg, 0.369 mL, 1.5 mmol) was added and the mixture was heated at 130 °C under an atmosphere of argon. After 24 h water was added, the formed precipitate was filtered off, washed with water and purified by column chromatography (petroleum ether : EtOAc / 95 : 5). 3e was obtained as a solid (153 mg, 78% yield).

Elemental Analysis: Calc.: C, 85.68; H, 6.16. Found: C, 85.78; H, 6.10

\[ ^1H \text{ NMR } 400 \text{ MHz CDCl}_3 \]

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\[ ^{13}C \text{ NMR } (100.6 \text{ MHz, CDCl}_3) \ δ : 26.8, 127.3, 127.4, 128.4, 129.0, 129.1, 136.0, 140.0, 145.9, 197.9 \]

GC-EIMS (m/z, %): 196 (M⁺, 48), 182 (14), 181 (100), 153 (42), 152 (69), 151 (21), 76 (11), 43 (12)
Chem. Name: 1-([1,1'-biphenyl]-3-yl)ethanone (3f)

Lit. Ref.: *Synthesis*, 2013, 40-44

**METHOD:**
In a screw capped vial equipped with a magnetic stirrer Pd/C 10% wt. (5.3 mg, 0.5 mmol%), GVL (1 mL), TBAF (488 mg, 1.5 mmol), 1-(3-bromophenyl)ethanone (1f) (204 mg, 0.136 mL, 1 mmol), were consecutively added and the resulting mixture was degassed with argon. Then triethoxy(phenyl)silane (2a) (368 mg, 0.369 mL, 1.5 mmol) was added and the mixture was heated at 130 °C under an atmosphere of argon. After 24 h water was added, the formed precipitate was filtered off, washed with water and purified by column chromatography (petroleum ether : EtOAc / 95 : 5). 3f was obtained as a solid (145 mg, 74% yield).

**Mol Formula:** C_{14}H_{12}O

**m.p.:** 38-41 °C

**Elemental Analysis:** Calc.: C, 85.68; H, 6.16. Found: C, 85.80; H, 6.12

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**13C NMR (100.6 MHz, CDCl3)** δ : 26.9, 127.1, 127.31, 127.32, 127.9, 129.0, 129.2, 131.9, 137.8, 140.3, 141.9, 198.2

**GC-EIMS (m/z, %):** 196 (M⁺, 66), 182 (14), 181 (100), 153 (68), 152 (82), 151 (25), 76 (13), 43 (19)
Chem. Name| 4-methoxy-1,1'-biphenyl (3g)  

METHOD:  
In a screw capped vial equipped with a magnetic stirrer Pd/C 10% wt. (5.3 mg, 0.5 mmol%), GVL (1 mL), TBAF (488 mg, 1.5 mmol), 1-bromo-4-methoxybenzene (1g) (188 mg, 0.126 mL, 1 mmol), were consecutively added and the resulting mixture was degassed with argon. Then triethoxy(phenyl)silane (2a) (368 mg, 0.369 mL, 1.5 mmol) was added and the mixture was heated at 130 °C under an atmosphere of argon. After 24 h water was added, the formed precipitate was filtered off, washed with water and purified by column chromatography (petroleum ether). 3g was obtained as a solid (144 mg, 78% yield).  

Mol Formula| C\textsubscript{13}H\textsubscript{12}O  
m.p.| 87-89 °C  
Elemental Analysis:| Calc.: C, 84.75; H, 6.57. Found: C, 84.87; H, 6.55  

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\textsuperscript{13}C NMR (100.6 MHz, CDCl\textsubscript{3}) δ : 55.5, 114.3, 126.8, 126.9, 128.3, 128.8, 133.9, 141.0, 159.3  

GC-EIMS (m/z, %): 185 (14), 184 (M\textsuperscript{+}, 100), 169 (51), 141 (61), 139 (17), 115 (53)
### Chem. Name
4-methoxy-1,1'-biphenyl (3g)

### Lit. Ref.

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![Chemical Structure](image)

#### Method:
In a screw capped vial equipped with a magnetic stirrer Pd/C 10% wt. (5.3 mg, 0.5 mmol%), GVL (1 mL), TBAF (488 mg, 1.5 mmol), bromobenzene (1b) (158 mg, 0.107 mL, 1 mmol), were consecutively added and the resulting mixture was degassed with argon. Then triethoxy(4-methoxyphenyl)silane (2b) (418 mg, 0.406 mL, 1.5 mmol) was added and the mixture was heated at 130 °C under an atmosphere of argon. After 24 h water was added, the formed precipitate was filtered off, washed with water and purified by column chromatography (petroleum ether). 3g was obtained as a solid (149 mg, 81% yield).

#### Molecular Weight
184

#### Elemental Analysis:
- **Calc.:** C, 84.75; H, 6.57.
- **Found:** C, 84.88; H, 6.53

#### NMR Spectra

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<table>
<thead>
<tr>
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<tr>
<td><strong>13C NMR (100.6 MHz, CDCl₃)</strong></td>
<td>55.5, 114.3, 126.8, 126.9, 128.3, 128.8, 133.9, 141.0, 159.3</td>
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**GC-EIMS (m/z, %):** 185 (14), 184 (M⁺, 100), 169 (51), 141 (61), 139 (17), 115 (53)
**Chem. Name** 4-methoxy-1,1'-biphenyl (3g)  
**Lit. Ref.** *RSC Adv.*, 2012, 2, 590–594

\[
\begin{align*}
\text{4b} & \quad \text{Si(OEt)}_3 \\
\text{1.5 eq.} & \quad \text{TBAF (1.5 eq.), Pd/C (0.5 mol%), GVL (1M), 130 °C, 24 h} \\
& \quad \text{3g} \\
\text{Molecular Weight: 184}
\end{align*}
\]

**METHOD:**  
In a screw capped vial equipped with a magnetic stirrer Pd/C 10% wt. (5.3 mg, 0.5 mmol%), GVL (1 mL), TBAF (488 mg, 1.5 mmol), iodobenzene (4b) (208 mg, 0.114 mL, 1 mmol), were consecutively added and the resulting mixture was degassed with argon. Then triethoxy(4-methoxyphenyl)silane (2b) (418 mg, 0.406 mL, 1.5 mmol) was added and the mixture was heated at 130 °C under an atmosphere of argon. After 24 h water was added, the formed precipitate was filtered off, washed with water and purified by column chromatography (petroleum ether). 3g was obtained as a solid (148 mg, 80% yield).

**Mol Formula** C\textsubscript{13}H\textsubscript{12}O  
**m.p.** 87-89 °C

**Elemental Analysis:** Calc.: C, 84.75; H, 6.57. Found: C, 84.86; H, 6.60

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<th>( \delta ) value</th>
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<th>Mult.</th>
<th>j value/Hz</th>
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\( ^{13}C \text{ NMR} \) (100.6 MHz, CDCl\textsubscript{3}) \( \delta \) : 55.5, 114.3, 126.8, 126.9, 128.3, 128.8, 133.9, 141.0, 159.3

**GC-EIMS (m/z, %):** 185 (14), 184 (M\textsuperscript{+}, 100), 169 (51), 141 (61), 139 (17), 115 (53)
Chem. Name 4-methoxy-4'-methyl-1,1'-biphenyl (3h)

\[
\begin{align*}
\text{Br} & \quad + \quad \text{Si(OEt)}_3 \\
1c & \quad 1.5 \text{ eq.} \\
\text{O} & \quad 3h \\
\end{align*}
\]

Molecular Weight: 198

METHOD:
In a screw capped vial equipped with a magnetic stirrer Pd/C 10% wt. (5.3 mg, 0.5 mmol%), GVL (1 mL), TBAF (488 mg, 1.5 mmol), 1-bromo-4-methylbenzene (1c) (174 mg, 1 mmol), were consecutively added and the resulting mixture was degassed with argon. Then triethoxy(4-methoxyphenyl)silane (2b) (418 mg, 0.406 mL, 1.5 mmol) was added and the mixture was heated at 130 °C under an atmosphere of argon. After 24 h water was added, the formed precipitate was filtered off, washed with water and purified by column chromatography (petroleum ether). 3h was obtained as a solid (161 mg, 81% yield).

Molar Formula \( \text{C}_{14}\text{H}_{14}\text{O} \)
m.p. 110-112 °C

Elemental Analysis: Calc.: C, 84.81; H, 7.12. Found: C, 84.75; H, 7.08

\( ^1\text{H} \text{NMR 400 MHz CDCl}_3 \)

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</table>

\( ^{13}\text{C} \text{NMR (100.6 MHz, CDCl}_3) \) \( \delta \): 21.0, 55.2, 114.1, 126.5, 127.9, 129.4, 133.7, 136.3, 137.9, 158.8

GC-EIMS (m/z, %): 199 (14), 198 (M\(^+\), 100), 183 (59), 155 (44), 154 (11), 153 (17), 152 (17), 128 (16), 127 (10), 115 (12)
**Chem. Name**: 4-methoxy-4'-methyl-1,1'-biphenyl (3h)


![Reaction Scheme](image)

**Molecular Weight**: 198

**METHOD:**
In a screw capped vial equipped with a magnetic stirrer Pd/C 10% wt. (5.3 mg, 0.5 mmol%), GVL (1 mL), TBAF (488 mg, 1.5 mmol), 1-iodo-4-methylbenzene (4c) (220 mg, 1 mmol), were consecutively added and the resulting mixture was degassed with argon. Then triethoxy(4-methoxyphenyl)silane (2b) (418 mg, 0.406 mL, 1.5 mmol) was added and the mixture was heated at 130 °C under an atmosphere of argon. After 24 h water was added, the formed precipitate was filtered off, washed with water and purified by column chromatography (petroleum ether). 3h was obtained as a solid (158 mg, 80% yield).

**Mol Formula**: C14H14O

**m.p.**: 110-112 °C

**Elemental Analysis**: Calc.: C, 84.81; H, 7.12. Found: C, 84.77; H, 7.11

<table>
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<th>j (Hz)</th>
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</table>

**1H NMR (400 MHz, CDCl3)**

**13C NMR (100.6 MHz, CDCl3)** δ : 21.0, 55.2, 114.1, 126.5, 127.9, 129.4, 133.7, 136.3, 137.9, 158.8

**GC-EIMS (m/z, %):** 199 (14), 198 (M+, 100), 183 (59), 155 (44), 154 (11), 153 (17), 152 (17), 128 (16), 127 (10), 115 (12)
**Chem. Name**: 4,4’-dimethoxy-1,1’-biphenyl (3i)

**Lit. Ref.**: *Organometallics*, 2008, 27, 2833–2839

![Chemical Structure]

**METHOD:**

In a screw capped vial equipped with a magnetic stirrer Pd/C 10% wt. (5.3 mg, 0.5 mmol%), GVL (1 mL), TBAF (488 mg, 1.5 mmol), 1-bromo-4-methoxybenzene (1g) (188 mg, 0.126 mL, 1 mmol), were consecutively added and the resulting mixture was degassed with argon. Then triethoxy(4-methoxyphenyl)silane (2b) (418 mg, 0.406 mL, 1.5 mmol) was added and the mixture was heated at 130 °C under an atmosphere of argon. After 24 h water was added, the formed precipitate was filtered off, washed with water and purified by column chromatography (petroleum ether). 3i was obtained as a solid (195 mg, 91% yield).

**Mol Formula**: C_{14}H_{14}O_{2}

**m.p.**: 177-180 °C

**Elemental Analysis**: C, 78.48; H, 6.59. Found: C, 78.44; H, 6.64

<table>
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</table>

| 13C NMR (100.6 MHz, CDCl₃) | δ: 55.5, 114.3, 127.9, 133.6, 158.8 |

**GC-EIMS (m/z, %):** 215 (16), 214 (M⁺, 100), 200 (14), 199 (95), 171 (45), 156 (17), 139 (14), 128 (34), 127 (11), 102 (10)
Chem. Name 4-methoxy-4'-nitro-1,1'-biphenyl (3j)  

METHOD:  
In a screw capped vial equipped with a magnetic stirrer Pd/C 10% wt. (5.3 mg, 0.5 mmol%), GVL (1 mL), TBAF (488 mg, 1.5 mmol), 1-bromo-4-nitrobenzene (1a) (204 mg, 1 mmol), were consecutively added and the resulting mixture was degassed with argon. Then triethoxy(4-methoxyphenyl)silane (2b) (418 mg, 0.406 mL, 1.5 mmol) was added and the mixture was heated at 130 °C under an atmosphere of argon. After 24 h water was added, the formed precipitate was filtered off, washed with water and purified by column chromatography (petroleum ether : EtOAc / 95 : 5). 3j was obtained as a solid (186 mg, 81% yield).

**Mol Formula** C_{13}H_{11}NO_{3}  
**m.p.** 107-110 °C

**Elemental Analysis:** Calc.: C, 68.11; H, 4.84; N, 6.11. Found: C, 68.21; H, 4.90; N, 6.05

<table>
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<th>Mult.</th>
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</table>

| $^{13}$C NMR (100.6 MHz, CDCl$_3$) δ | 55.5, 114.7, 124.2, 127.1, 128.7, 131.1, 146.6, 147.3, 160.5 |

**GC-EIMS (m/z, %):** 230 (14), 229 (M$^+$, 100), 199 (25), 183 (11), 171 (18), 168 (24), 152 (18), 140 (31), 139 (57), 128 (13)
Chem. Name 4-methoxy-4'-nitro-1,1'-biphenyl (3j)

METHOD:
In a screw capped vial equipped with a magnetic stirrer Pd/C 10% wt. (5.3 mg, 0.5 mmol%), GVL (1 mL), TBAF (488 mg, 1.5 mmol), 1-iodo-4-nitrobenzene (4a) (254 mg, 1 mmol), were consecutively added and the resulting mixture was degassed with argon. Then triethoxy(4-methoxyphenyl)silane (2b) (418 mg, 0.406 mL, 1.5 mmol) was added and the mixture was heated at 130 °C under an atmosphere of argon. After 24 h water was added, the formed precipitate was filtered off, washed with water and purified by column chromatography (petroleum ether : EtOAc / 95 : 5). 3j was obtained as a solid (179 mg, 78% yield).

Mol Formula C_{13}H_{11}NO_{3} m.p. 107-110 °C

Elemental Analysis: Calc.: C, 68.11; H, 4.84; N, 6.11. Found: C, 68.19; H, 4.80; N, 6.04

\[ \text{\textsuperscript{1}H NMR 400 MHz CDCl}_3 \]

<table>
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<th>Mult.</th>
<th>j value/Hz</th>
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</table>

\[ \text{\textsuperscript{13}C NMR (100.6 MHz, CDCl}_3 \] δ : 55.5, 114.7, 124.2, 127.1, 128.7, 131.1, 146.6, 147.3, 160.5

GC-EIMS (m/z, %): 230 (14), 229 (M⁺, 100), 199 (25), 183 (11), 171 (18), 168 (24), 152 (18), 140 (31), 139 (57), 128 (13)
Chem. Name: 1-(4-methoxyphenyl)naphthalene (3k)


![Chemical structure](image)

**METHOD:**
In a screw capped vial equipped with a magnetic stirrer Pd/C 10% wt. (5.3 mg, 0.5 mmol%), GVL (1 mL), TBAF (488 mg, 1.5 mmol), 1-bromonaphthalene (1d) (213 mg, 0.144 mL, 1 mmol), were consecutively added and the resulting mixture was degassed with argon. Then triethoxy(4-methoxyphenyl)silane (2b) (418 mg, 0.406 mL, 1.5 mmol) was added and the mixture was heated at 130 °C under an atmosphere of argon. After 24 h water was added, the formed precipitate was filtered off, washed with water and purified by column chromatography (petroleum ether). 3k was obtained as a solid (185 mg, 79% yield).

**Mol Formula**: C_{17}H_{14}O

**m.p.**: 114-116 °C

**Elemental Analysis**: Calc.: C, 87.15; H, 6.02. Found: C, 87.45; H, 6.05

<table>
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<th>No. H</th>
<th>Mult.</th>
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<td>7.89</td>
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| 13C NMR (100.6 MHz, CDCl₃) δ | 55.5, 113.8, 125.5, 125.8, 126.0, 126.2, 127.0, 127.4, 128.5, 131.2, 132.0, 133.2, 134.0, 140.0, 159.1 |

**GC-EIMS (m/z, %):** 235 (31), 234 (M⁺, 100), 219 (50), 203 (15), 202 (11), 191 (33), 190 (39), 189 (56), 101 (10), 94 (11)
METHOD:

In a screw capped vial equipped with a magnetic stirrer Pd/C 10% wt. (5.3 mg, 0.5 mmol%), GVL (1 mL), TBAF (488 mg, 1.5 mmol), 1-(4-bromophenyl)ethanone (1e) (204 mg, 1 mmol), were consecutively added and the resulting mixture was degassed with argon. Then triethoxy(4-methoxyphenyl)silane (2b) (418 mg, 0.406 mL, 1.5 mmol) was added and the mixture was heated at 130 °C under an atmosphere of argon. After 24 h water was added, the formed precipitate was filtered off, washed with water and purified by column chromatography (petroleum ether : EtOAc / 95 : 5). 3l was obtained as a solid (170 mg, 75% yield).


<table>
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GC-EIMS (m/z, %): 227 (10), 226 (M⁺, 55), 212 (12), 211 (100), 183 (13), 168 (20), 152 (12), 140 (29), 139 (49), 63 (11), 43 (18)
METHOD:
In a screw capped vial equipped with a magnetic stirrer Pd/C 10% wt. (5.3 mg, 0.5 mmol%), GVL (1 mL), TBAF (488 mg, 1.5 mmol), 1-(3-bromophenyl)ethanone (1f) (204 mg, 0.136 mL, 1 mmol), were consecutively added and the resulting mixture was degassed with argon. Then triethoxy(4-methoxyphenyl)silane (2b) (418 mg, 0.406 mL, 1.5 mmol) was added and the mixture was heated at 130 °C under an atmosphere of argon. After 24 h water was added, the formed precipitate was filtered off, washed with water and purified by column chromatography (petroleum ether : EtOAc / 95 : 5). 3m was obtained as a solid (181 mg, 80% yield).

Mol Formula | C₁₅H₁₄O₂ | m.p. | 55-57 °C


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| C NMR (100.6 MHz, CDCl₃) | δ : | 26.8, 55.4, 114.4, 126.5, 126.7, 128.3, 129.1, 131.3, 132.6, 137.6, 141.3, 159.6, 198.3 |

GC-EIMS (m/z, %): 227 (16), 226 (M⁺, 100), 212 (11), 211 (88), 183 (44), 168 (35), 153 (11), 152 (28), 140 (39), 139 (76), 63 (10)
4-nitro-1,1′-biphenyl (3a)
4-nitro-1,1′-biphenyl (3a)
1,1'-biphenyl (3b)
1,1′-biphenyl (3b)
4-methyl-1,1’-biphenyl (3c)
4-methyl-1,1'-biphenyl (3c)
1-phenyl-naphthalene (3d)
1-phenynaphthalene (3d)
1-[[1,1'-biphenyl]-4-yl]ethanone (3e)
1−((1,1′−biphenyl)−4−yl)ethanone (3e)
1-([1,1′-biphenyl]-3-yl)ethanone (3f)
1-([1,1'-biphenyl]-3-yl)ethanone (3f)
4-methoxy-1,1'-biphenyl (3g)
4-methoxy-1,1′-biphenyl (3g)
4-methoxy-4'-methyl-1,1'-biphenyl (3h)
4-methoxy-4'-methyl-1,1'-biphenyl (3h)
$4,4'$-dimethoxy-$1,1'$-biphenyl (3i)
4,4′-dimethoxy-1,1′-biphenyl (3i)
4-methoxy-4'-nitro-1,1'-biphenyl (3j)
4-methoxy-4′-nitro-1,1′-biphenyl (3j)
1-(4-methoxyphenyl)naphthalene (3k)
1-(4-methoxyphenyl)naphthalene (3k)
1-(4’-methoxy-[1,1’-biphenyl]-4-yl)ethanone (3l)
1-(4’-methoxy-[1,1’-biphenyl]-4-yl)ethanone (3l)
1-(4'-methoxy-[1,1'-biphenyl]-3-yl)ethanone (3m)
1-(4′-methoxy-[1,1′-biphenyl]-3-yl)ethanone (3m)