Benzylic C–H Borylation on Ceria-Supported Nickel Hydroxides

**Significance:** Nickel hydroxides supported on CeO₂ (Ni(OH)ₓ/CeO₂), prepared by mixing NiCl₂·6H₂O and CeO₂ in H₂O (pH = 10) (eq. 1), catalyzed the benzylic C–H borylation of alkylarenes or diphenylmethanes with pinacolborane to give the corresponding benzylic boronates (eq. 2). In the reaction of methylarenes, gem-diborylated products were obtained as the main products (eq. 3). The monoborylation of methylarenes was also achieved by using the catalyst pretreated with HBpin and with the methylarene as the solvent (eq. 4).

**Comment:** Ni(OH)ₓ/CeO₂ was characterized by means of XANES XPS, EXAFS, HAADF-STEM, STEM-EDS, XRD, and ICP-AES analyses. A hot-filtration experiment suggested that the reaction occurred heterogeneously.

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**Equations:**

1. \[ \text{NiCl}_2\cdot6\text{H}_2\text{O} + \text{CeO}_2 \quad \text{pH} = 10 \quad \text{H}_2\text{O, r.t., 24 h} \quad \text{Ni(OH)}_x/\text{CeO}_2 \]

2. \[ \text{Ni(OH)}_x/\text{CeO}_2 \quad (3.6–11 \text{ mol% Ni}) \quad \text{HBpin (4 equiv)} \quad \text{methylcyclohexane or CPME} \quad \text{argon, 120 °C, 4 h} \]

3. \[ \text{Ni(OH)}_x/\text{CeO}_2 \quad (3.6–11 \text{ mol% Ni}) \quad \text{HBpin (4 equiv)} \quad \text{methylcyclohexane or CPME} \quad \text{argon, 120 °C, 4 h} \]

4. \[ \text{Ni(OH)}_x/\text{CeO}_2 - \text{HBpin (1.8 mol% Ni)} \quad \text{HBpin (0.4 equiv)} \quad \text{neat or CPME, argon, 120 °C, 4 h} \]