**Acceptorless Alcohol Dehydrogenation with an Iron–Cobalt Double-Atom Catalyst**

**Significance:** An Fe–Co double-atom catalyst supported on N-doped carbon (FeCo-DAC), prepared according to equation 1, catalyzed the dehydrogenation of alcohols to afford the corresponding aldehydes or ketones in ≤98% yield with generation of H₂ gas (eqs. 2 and 3).

**Comment:** FeCo-DAC was characterized by means of XRD, XPS, Raman, ICP-AES, TEM, HAADF-STEM, EDX, XANES, and EXAFS analyses. In the dehydrogenation of benzyl alcohol, the catalytic activity of FeCo-DAC was superior to that of the corresponding Fe and Co single-atom catalysts or other supported metal catalysts, such as Pd/C or Ru/C.

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**Equations:**

1. \[ \text{FeCl}_2 \cdot 4\text{H}_2\text{O} + \text{CoCl}_2 \cdot 6\text{H}_2\text{O} + 1,10\text{-phenanthroline} \rightarrow \text{Mg(OH)}_2 + \text{EtOH, 60 °C, 0.5 h} + \text{FeCo-DAC (1)} \]

2. \[ \text{R}_1\text{R}_2\text{OH} + \text{FeCo-DAC (30 mg)} + \text{Et}_3\text{N (0 or 10 mol%)} \rightarrow \text{R}_1\text{R}_2\text{O} + \text{H}_2 \text{gas (eqs. 2 and 3)} \]

3. \[ \text{FeCo-DAC (60 mg)} + \text{Et}_3\text{N (0 or 10 mol%)} \rightarrow \text{FeCo-DAC (60 mg)} + \text{Et}_3\text{N (0 or 10 mol%)} \rightarrow \text{40 examples up to 98% yield} \]

**Selected Results:**

- R = Me; 96% yield
- R = F; 88% yield
- R = I; 94% yield
- R = CN; 95% yield
- R = NO₂; 89% yield

- R = Me; 97% yield
- R = OMe; 83% yield
- R = Cl; 81% yield

- 91% yield

- 92% yield

- 88% yield

- 94% yield

- 93% yield

- 98% yield

- 94% yield

- 59% yield

- R = Me; 76% yield
- R = Ph; 78% yield

- 86% yield

- 93% yield

- 99% yield

- 94% yield

- 84% yield

Selected Results:

- 98% yield
- 92% yield

- 89% yield

- 92% yield

- n-C₆H₁₃

- 86% yield

- 88% yield

- 94% yield

- 92% yield

- 88% yield

- 94% yield

- 88% yield

- 98% yield

- 92% yield