Formal Hydroacylation of Alkynes on Mg₃Al–CO₃-Layered Double Hydroxide

**Significance:** Mg₃Al–CO₃-layered double hydroxide (Mg₃Al–CO₃ LDH) catalyzed the formal hydroacylation of terminal alkynes with aromatic aldehydes under argon to give the corresponding diaryl α,β-unsaturated ketones in up to 85% yield (24 examples).

**Comment:** In the reaction of phenylacetylene with p-anisaldehyde, Mg₃Al–CO₃ LDH was recovered and reused three times with a slight loss of its catalytic activity (fresh: 91% yield; third reuse: 73% yield).

**Selected examples:**

- R = OMe, 76% yield
- R = H, 71% yield
- R = Me, 68% yield
- R = CF₃, 53% yield
- R = Cl, 78% yield
- R = NMe₂, 64% yield
- R = CN, 54% yield

Other examples:

- R = F, 79% yield
- R = Br, 65% yield
- R = OMe, 74% yield
- R = n-Bu, 69% yield

**Equation:**

\[
R^1\text{H} + R^2\equiv \xrightarrow{\text{Mg₃Al–CO₃ LDH (130–200 mg)}} R^1\equiv R^2
\]

(0.5 mmol) (1.2 equiv)

PhMe, 120 °C, 15–24 h argon atmosphere

24 examples up to 85% yield