CATAPOWER

# APPLICATION NOTE

## Catalyst quench & removal with MetalShark Mako

In collaboration with Prof. Steven Diver at the University of Buffalo, Catapower is proud to offer silica-supported MetalShark Mako (**1**) for convenient removal of spent metal catalysts from completed reaction mixtures. Scavenger **1** is easily removed from quenched reaction mixtures by simple filtration.

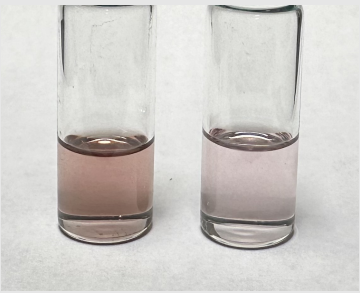
With Ru catalysts originally present at 2.5 mol % loading for ring-closing metathesis, a single 30-minute treatment with MetalShark Mako affords >99.9% removal of spent catalyst to ppm levels.

MetalShark Mako is similarly effective in sequestering spent Pd from cross-coupling reactions, typically affecting >99.9% removal to ppm levels with a variety of precatalysts and product classes. Difficult product mixtures, such as those containing chelating 1,2-diamines from Buchwald-Hartwig aminations, affect > 90% Pd removal in a single pass under unoptimized conditions.

Optimization of the equivalents of the scavenger and the reaction time is recommended for new applications and those requiring very low (trace) quantities of residual metals. Generally, 8 - 15 equivalents scavenger relative to catalyst is adequate.

Beyond Ru and Pd, MetalShark Mako may also be effective in removing a wide variety of other spent metal catalysts. The product degrades slowly with exposure to atmospheric moisture; storage at ambient temperature under dry conditions is recommended.

**Before quench:**
1st. gen. Grubbs catalyst #02-01-1001 at 0.05 mM in a reaction mixture



**After quench:**
The same mixture 1 h after addition of 8 equiv **1** (relative to catalyst) and filtration.

*Representative procedure:*
On completion of the catalytic reaction, the process temperature and inert atmosphere are maintained as appropriate. Silica-supported scavenger **1** (8 - 15 equiv relative to the catalyst) is added, and the resulting slurry is stirred 1 h. A change in color may be observed depending on the specific catalyst used, but chromatic shift is not necessarily diagnostic. The metal-loaded scavenger may be removed by simple filtration, and the desired product recovered by appropriate means.

### MetalShark Mako

| | |
|---|---|
| Scavenging functional group: | Isonitrile |
| Solid support: | Silica gel |
| Loading: | 0.8 - 1.2 mmol / g |
| Catalog: | 01-09-2001 |