Efficient Removal of Metal Based Catalysts Using SiliaBond® DMT
François Béland, Geneviève Gingras, Nathalie Huther*, Steeves Potvin, Lynda Tremblay
SiliCycle® Inc. (www.silicycle.com) 2500 Parc-Technologique Blvd, Quebec City, Quebec, G1P 4S6, Canada
*(nathaliehuther@silicycle.com)

Heck and Buchwald

Other type of reactions, such as Heck and Buchwald, imply the use of palladium and as for the Suzuki coupling, palladium removal may be problematic. Treatment with Si-DMT on these reactions gave the following results:

<table>
<thead>
<tr>
<th>Conditions</th>
<th>Before treatment</th>
<th>1eq, 4h, 80 ºC</th>
<th>2eq, 4h, 80 ºC</th>
<th>3eq, 1h, 22 ºC</th>
<th>4eq, 4h, 22 ºC</th>
</tr>
</thead>
<tbody>
<tr>
<td>Residual Pd (ppm)</td>
<td>407.0</td>
<td>56.8 (867%)</td>
<td>34.4 (92%)</td>
<td>46.5 (89%)</td>
<td>40.9 (90%)</td>
</tr>
</tbody>
</table>

Once again, Si-DMT allowed a significant reduction in the amount of residual palladium.

Removal of Other Metal Residues

Here is a selection of palladium mediated reaction that also involve other metals and the scavenging results obtained by using Si-DMT:

1. Negishi:

2. Sonogashira:

3. Cyanation:

Stille

Stille coupling is another type of palladium mediated reaction that also involve the use of organotin compounds and hence, contamination by tin residues is usually an issue. To probe the potential scavenging ability of Si-DMT for a mixture of metal contaminants, a traditional Stille coupling reaction was performed as follow:

In this case, we not only attempted to reduce the amount of palladium in solution but we were also hoping to remove tin residues in a single treatment. The results are summarized in the table below:

<table>
<thead>
<tr>
<th>Conditions</th>
<th>Before treatment</th>
<th>1eq, 4h, 80 ºC</th>
<th>2eq, 4h, 80 ºC</th>
<th>3eq, 1h, 22 ºC</th>
<th>4eq, 4h, 22 ºC</th>
</tr>
</thead>
<tbody>
<tr>
<td>Residual Pd (ppm)</td>
<td>145.1</td>
<td>24.0 (84%)</td>
<td>3.1 (98%)</td>
<td>6.7 (95%)</td>
<td>4.9 (97%)</td>
</tr>
<tr>
<td>Residual Sn (ppm)</td>
<td>2306</td>
<td>No scavenging</td>
<td>No scavenging</td>
<td>1880 (99%)</td>
<td>1830 (92%)</td>
</tr>
</tbody>
</table>

Unfortunately, although palladium concentrations were significantly reduced, the tin seems untouched by Si-DMT.*

Conclusion

This study has demonstrated that not only SiliaBond DMT can be used as an efficient scavenger for palladium residues after reaction workups but it can also remove other metals. Si-DMT is therefore a very versatile metal scavenger for common metal mediated synthetic transformations.

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* Tin residues can be successfully removed using SiliaBond TAAcONa.