

Fast and Simple Removal of Rhodium using StratoSpheres™ SPE

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Introduction

Rhodium containing catalysts are used extensively in many aspects of synthetic chemistry, from small-scale applications such as the synthesis of small molecules for medicinal chemistry to large-scale manufacturing. One of the most well-known rhodium catalysts is Wilkinson's catalyst, a homogeneous catalyst used in the hydrogenation of alkenes. Another highly useful rhodium-based reagent is rhodium acetate, which is used to initiate carbene formation from a diazo species.

Despite the utility of rhodium reagents, the removal of the metal residues post reaction can cause difficulties. Rhodium species can remain chelated to desired reaction products, particularly if those entities contain good chelation groups, ie amines, amides and urea groups. Reducing the residual rhodium content in a sample may reduce any potential high throughput screen failures. In more large-scale applications, effective sequestration is often required due to economic and environmental constraints.

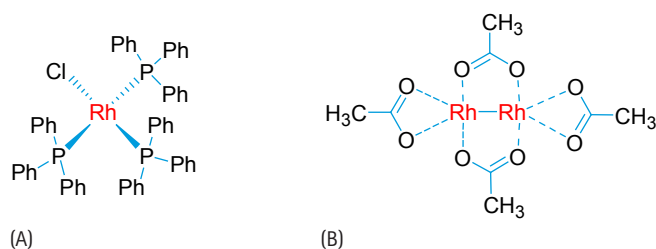


Figure 2. Wilkinson's Catalyst (A) and Rhodium Diacetate (B)

PL-Thiol MP SPE and PL-Thiourea MP SPE for Flow-Through Removal of Rhodium

Polymer Laboratories, now a part of Varian, Inc., has developed two devices that can effectively remove rhodium residues from organic solutions in a single pass under gravity. The availability of two functional polymers gives the end user more choice in matching resin with substrate. The sorbents are made from a highly cross-linked macroporous polymer that does not swell and can be used for a range of protic, non-protic, polar and apolar solvents. The polymer is also

specially engineered to be self-indicating in the presence of certain metal ions, thus informing the user when the device is nearing its capacity.



Figure 2. (left to right) PL-Thiol MP SPE and PL-Thiourea MP SPE

Typical Procedure for the Removal of Rhodium Containing Catalysts

Pre-condition the SPE tube with MeOH (1 mL). Add the catalyst containing solution to the tube (PL-Thiol MP SPE or PL-Thiourea MP SPE) and allow the solution to pass through the medium under gravity. Once all of the solution has moved through, wash the tube with 1-2 mL of a suitable solvent (MeOH, THF, DCM, etc). Take the organic solution and remove the solvent *in vacuo* to obtain the desired compound free of metal contamination (Table 1).

If MeOH is not compatible with the chemistry application then it can be substituted with a non-viscous solvent such as DCM or THF, but for most applications MeOH is recommended.

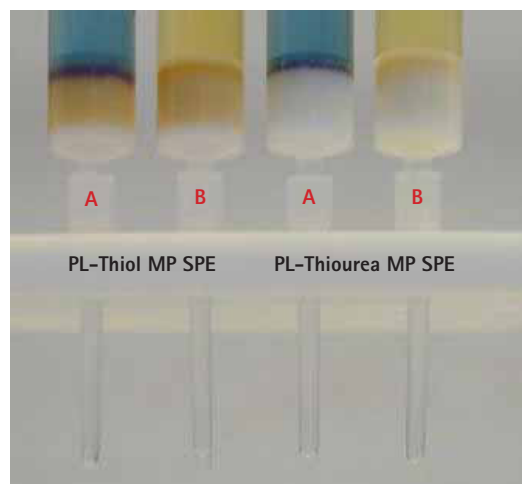


Figure 3. Removal of Rhodium based catalysts using PL-Thiol MP SPE & PL-Thiourea MP SPE

Table 1. ICP analysis of samples after Rh scavenging.

Catalyst	Final Rh Conc (ppm)	
	PL-Thiol MP SPE	PL-Thiourea MP SPE
Rh ₂ (OAc) ₄ (in 1,4 dioxane) A	<0.1	<0.1
Wilkinson's Catalyst (in MeOH) B	<0.1	<0.1

Starting concentrations of Rh catalysts were ~1000 ppm

Conclusion

PL-Thiol MP SPE and PL-Thiourea MP SPE are fast and effective tools for removing rhodium residues from organic solutions. These single use devices, with optimum flow characteristics, are compatible with a broad range of organic solvents and pH levels.

These data represent typical results.

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