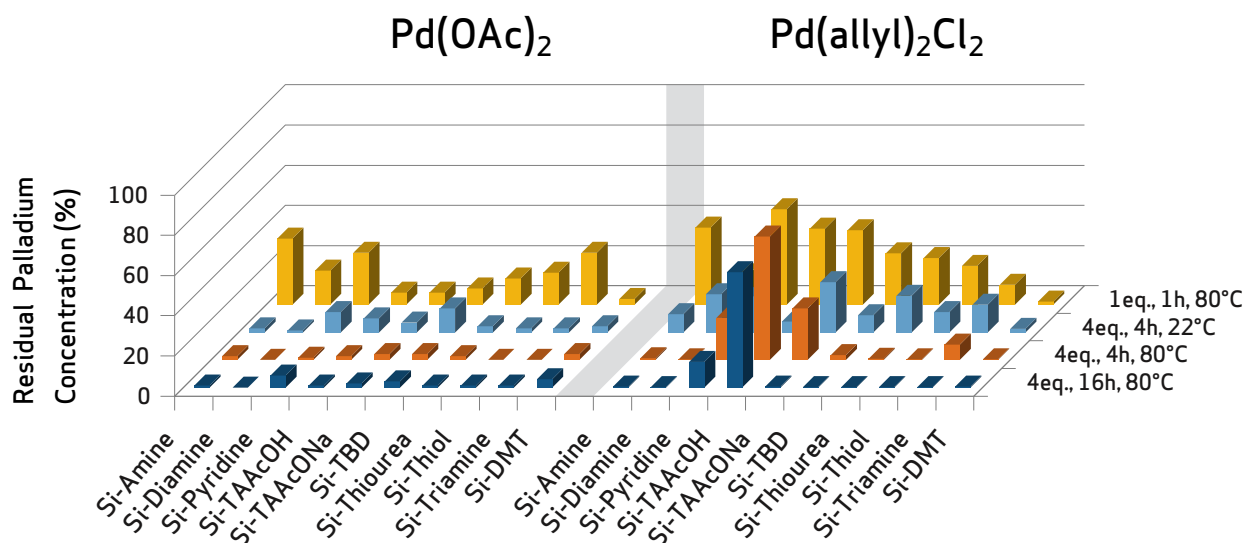


Application Note M3.0

Scavenging Palladium Catalysts

A metal scavenging study using **SiliaBond Metal Scavengers** products was conducted on various palladium catalysts commonly used in organic synthesis. Various parameters were studied for their influence on the scavengers' robustness as well as to establish the best conditions to bring the level of metal down to an acceptable level for the pharmaceutical industry (i.e.: solvent, number of equivalents, temperature, and reaction time).

SCAVENGING PALLADIUM CATALYSTS RESULTS



Pd(OAc)₂: Residual Palladium Concentration

Scavengers	Conditions	Pd (ppm)
SiliaBond Amine	4eq., 4h, 80°C	13.5
	4eq., 16h, 80°C	4.1
SiliaBond Diamine	4eq., 4h, 22°C	10.8
	4eq., 4h, 80°C	2.9
	4eq., 16h, 80°C	3.8
SiliaBond TAAcOH	4eq., 4h, 80°C	12.5
	4eq., 16h, 80°C	10.5
SiliaBond Thiol	4eq., 4h, 80°C	12.7
	4eq., 16h, 80°C	6.27
SiliaBond Thiourea	4eq., 4h, 80°C	< 1
SiliaBond Triamine	4eq., 4h, 80°C	< 1

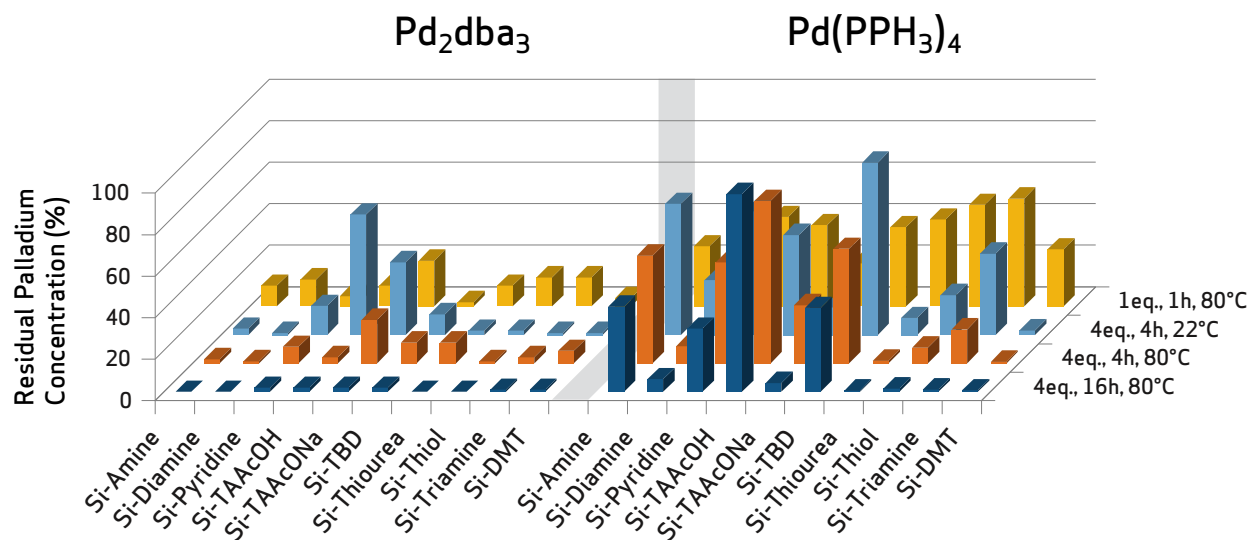
Initial concentration: 1 000 ppm in DMF

Pd(allyl)₂Cl₂: Residual Palladium Concentration

Scavengers	Conditions	Pd (ppm)
SiliaBond Amine	4eq., 4h, 80°C	8.6
	4eq., 16h, 80°C	3.9
SiliaBond Diamine	4eq., 4h, 80°C	3.6
	4eq., 16h, 80°C	3.1
SiliaBond TAAcONa	4eq., 16h, 80°C	4.0
SiliaBond TBD	4eq., 16h, 80°C	3.0
SiliaBond Thiol	4eq., 4h, 80°C	5.1
	4eq., 16h, 80°C	2.7
SiliaBond Thiourea	4eq., 4h, 80°C	3.6
	4eq., 16h, 80°C	2.6
SiliaBond Triamine	4eq., 16h, 80°C	2.5
SiliaBond DMT	4eq., 4h, 80°C	2.5

Initial concentration: 1 000 ppm in DMF

SCAVENGING PALLADIUM CATALYSTS RESULTS (CONTINUED)

 $\text{Pd}_2(\text{dba})_3$: Residual Palladium Concentration

Scavengers	Conditions	Pd (ppm)
SiliaBond Amine	4eq., 16h, 80°C	3.4
SiliaBond Diamine	4eq., 4h, 22°C	11.6
	4eq., 4h, 80°C	6.3
	4eq., 16h, 80°C	1.9
SiliaBond Thiol	4eq., 16h, 80°C	2.5
SiliaBond Thiourea	4eq., 4h, 80°C	5.3
	4eq., 16h, 80°C	1.8
SiliaBond Triamine	4eq., 16h, 80°C	4.6
SiliaBond DMT	4eq., 4h, 22°C	8.6

Initial concentration: 1 000 ppm in DMF

 $\text{Pd}(\text{PPh}_3)_4$: Residual Palladium Concentration

Scavengers	Conditions	Pd (ppm)
SiliaBond Thiol	4eq., 4h, 80°C	12.8
	4eq., 16h, 80°C	5.9
SiliaBond Thiourea	4eq., 16h, 80°C	16.7
SiliaBond Triamine	4eq., 16h, 80°C	11.6
SiliaBond DMT	4eq., 4h, 80°C	7.6

Initial concentration: 1 000 ppm in DMF