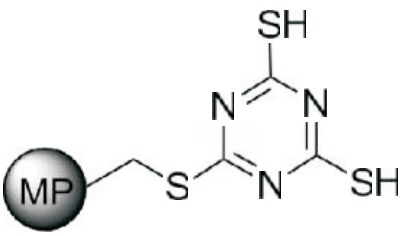
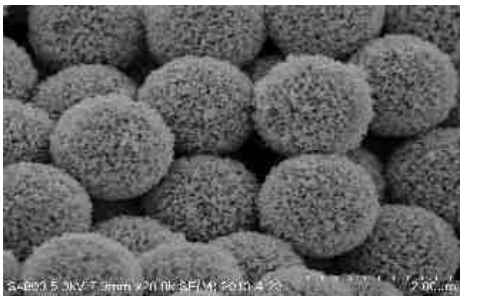
MP-TMT Highlight - Metal scavenging with resins / silicas in DMF





	Loading (mmol g)	$Pd(OAc)_2$ 3 eq (ppm)	$CuCl_2 - 2H_2O$ 3 eq (ppm)	$ZnCl_2$ 3 eq (ppm)	$CoCl_2 - 6H_2O$ 3 eq (ppm)	$Ni(OAc)_2 - 4H_2O$ 3 eq (ppm)	$FeCl_3 - 6H_2O$ 3 eq (ppm)
MP Polyamine (Nexgen)	1.71	0	0	0	0	15	0
MP-TMT (Nexgen)	1.51	0	-	-	-	-	-
Polyamine (Purolite S985)	3.1	-	-	-	-	-	-
Polyamine (Purolite S992)	1.52	-	-	-	-	-	-
Polyamine (Purolite A149)	1.7	-	-	-	-	-	-
Polyamine (Purolite A170)	0.8	-	-	-	-	-	-
MP-Thiol (Purolite S924)	7.7	-	-	-	-	-	-
Thiourea (Purolite S914)	2.5	196	-	-	-	-	-
Thiourea (Purolite TP214)	1.68	209	-	-	-	-	-
MP-Iminodiacetic (Purolite S930)	3.21	-	-	-	-	-	-
Aldoxime (Purolite S910)	6.95	-	-	-	-	-	-
QuadraPure BZA	1.3	562	0	1	0	231	27
QuadraPure BDZ	1.3	466	697	718	735	353	696
QuadraPure TU	1.3	646	0	235	307	482	505
QuadraPure DET	1	1157	1174	1176	1048	849	1033
QuadraPure IDA	1.3	1429	832	1116	982	441	886
QuadraPure AMPA	1.3	1279	1220	1044	998	374	836
SiliaMet DMT	0.62	50	22	310	214	5	442
SiliaMet Triamine	1.28	53	2	7	0	13	0
SiliaMet TAAcOH	0.44	68	61	490	244	3	286
SiliaMet TAAONa	0.45	58	0	1	0	1	0
SiliaMet Thiol	1.28	52	1226	1151	1129	392	1088
SiliaMet Thiourea	1.08	44	208	957	946	3	1079
SiliaMet Imidazole	1.16	60	11	49	53	904	127
SiliaMet Cysteine	0.35	44	278	197	84	6	20

Scavenging experimental procedure:

Resins were added to 10mL stock solutions (2000 ppm) of catalyst in DMF at room temperature and stirred for 2 hours.

Rinsed with DMF (3x2mL).

The DMF solutions were analyzed by Atomic Absorption (detection limit: 0.5 ppm)

"0 ppm" means less than the limit of detection (e.g.: 0.5 ppm).

"- ppm" means that the colored solution was still visible and wasn't analyzed.

Metal Scavenger Sample Calculations

Pd	106.42 g/mol
$Pd(OAc)_2$	224.5 g/mol

Based on known metal concentration (ppm)

For instance, if you know the palladium concentration in 950G of material is 1000ppm

Known values:
- Loading of the Scavenger (Silica Thiopropyl) = 1.6 mmol/g
- Molecular Weight of metal: Pd = 106.42 g/mol
- Residual metal concentration: 800ppm of Pd
- Amount of Pd to be scavenged = 950G

1. Determine the amount of palladium to be scavenged

$$\text{Amount of Pd (mg)} = \frac{\text{Residual metal concentration x Amount of Pd to be scavenged}}{1000}$$

$$\text{Amount of Pd (mg)} = \frac{800\text{ppm} \times 950\text{G}}{1000} = 760\text{ mg of Pd in 950 of product}$$

$$\text{Conversion in mmol of Pd} = \frac{\text{Amount of Pd in mg}}{\text{Molecular Wt of Metal}}$$

$$\text{Conversion in mmol of Pd} = \frac{760\text{ mg}}{106.42\text{ g/mol}} = 7.14\text{ mmol of Pd}$$

2. Calculate the amount of scavenger (Silica Thiopropyl) to use (4 equivalents)

$$\text{Amount of Silica Thiopropyl to use} = \frac{\text{Number of mmol of metal concentration}}{\text{Silica Thiopropyl Loading}}$$

$$\text{Conversion in mmol of Pd} = \frac{7.14\text{ mmol of Pd}}{1.6\text{ mmol/g}} = 4.46\text{ g of Silica Thiopropyl for 1 eq.}$$

To scavenge 800ppm of Pd, 4.46 g of Silica Thiopropyl is needed if using only one equivalent. However, it is recommended to start with a minimum of 4 equivalents at first meaning amount of Silica Thiopropyl will be 4 times higher (17.85 g). Moreover, many chemists start with very high equivalents (20 or more) to identify best scavenger to use and then optimize, thereafter. Sometimes, the residual metal concentration is unknown. In these cases, the amount (g) of Pd to be scavenged can be replaced by the amount of metal catalyst used for the reaction:

Based on amount of metal catalyst used

Known values:
- Amount of metal catalyst used: Ex. 25g of Pd(OAc)2
- Metal catalyst molecular weight: Pd(OAc)2 = 224.5 g/mol

1. Determine the amount of palladium to be scavenged

$$\text{Amount of Pd (mg)} = \frac{\text{Qty of catalyst used for the reaction x 1000}}{\text{Metal catalyst molecular weight}}$$

$$\text{Amount of Pd (mg)} = \frac{25\text{g of Pd(OAc)2} \times 1000}{224.5\text{ g/mol}} = 111.36\text{ mmol of Pd (maximum to be scavenged)}$$

The amount of Silica Thiopropyl to be used can then be determined as stated above (see #2). In this particular example, one equivalent of Silica Thiopropyl corresponds to 69.6 g.

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