QuadraPure™ & QuadraSil™ Scavengers

Functionalised beads and silicas for extraction of metal contaminants in batch or flow mode
Catalogue supplies from Aldrich & Wako
Available at commercial scale from Reaxa Products designed for GMP use

Metal Contamination Issues:

- High levels in products
- Regulatory limits in API
- Complex work up
- Wasteful yield losses
- Lost metal value
- Waste-stream poisoning

QuadraPure™ & QuadraSil™ Solutions:

- Effective purification
- GMP compliant
- Easy filtration
- Absorbs target metals only
- Efficient metal recovery
- Clean waste streams
QuadraPure™ Range

**Microporous**
- 1-4% crosslinked
- Gel-like
- Swell x 3-10
- Generally high loading
- Fast in selected organic solvents
- Ideal for batch use
- Available at R&D scale

**Macroporous**
- 5-50% crosslinked
- Rigid Structure
- Swell x 0.1-1.0
- Functionality on pore surface
- Used in all solvents, inc. aqueous
- Designed for flow and batch use
- Available R&D → manufacturing scale
# QuadraPure™ Macroporous Products

<table>
<thead>
<tr>
<th>QuadraPure™</th>
<th>functional group</th>
<th>metals removed</th>
</tr>
</thead>
<tbody>
<tr>
<td>TU</td>
<td>thiourea</td>
<td>Pd, Pt, Ru, Rh, Au, Ag, Cu, Hg, Pb, Cd, Ni, Co, Fe, V, Zn</td>
</tr>
<tr>
<td>IDA</td>
<td>imino diacetate</td>
<td>Cu, Al, Ga, In, V, Pb, Ni, Zn, Cd, Be, Mn, Sr, Ba, Co, Fe</td>
</tr>
<tr>
<td>AMPA</td>
<td>aminomethyl phosphonic acid</td>
<td>Fe, Cu, Ni, Al, Co, V</td>
</tr>
<tr>
<td>BZA</td>
<td>benzyl amine</td>
<td>Rh, Pd, Cu, Co, Ni</td>
</tr>
<tr>
<td>BDZ</td>
<td>imidazole</td>
<td>Rh, Co, Pd, Ni</td>
</tr>
<tr>
<td>EDA</td>
<td>amine</td>
<td>Pd in basic media and with phosphines, Co, Ni, Rh</td>
</tr>
<tr>
<td>DET</td>
<td>thiol</td>
<td>Pd in acidic media and with phosphines, Co, Fe, Ni, Rh</td>
</tr>
</tbody>
</table>
## QuadraPure™ Microporous Products

<table>
<thead>
<tr>
<th>QuadraPure™</th>
<th>functional group</th>
<th>metals removed</th>
</tr>
</thead>
<tbody>
<tr>
<td>IMDAZ</td>
<td>Imidazolylpropyl amino</td>
<td>Pd, Ru, Os, Co, Ni, V, Rh, Cu, Fe, Sn, V</td>
</tr>
<tr>
<td>MPA</td>
<td>mercaptophenyl amino</td>
<td>Pd, Pt, Rh, Ni, Ru, Cu, Sn, Ag, Au, Cd, Hg, Pb</td>
</tr>
<tr>
<td>AEA</td>
<td>aminoethyl amino</td>
<td>Pd, Rh, V, Cu, Fe, V</td>
</tr>
</tbody>
</table>
Macroporous Range: Typical Properties

- **polymer matrix**
- **average bead size (monodispersed)** 500 µm
- **operating pH range** 1 - 14
- **operating temperature range** RT → 60 °C

Typical Working Capacity for Metal Removal 10 - 30 mg/g
QuadraSil™ Product Range

<table>
<thead>
<tr>
<th>QuadraSil™</th>
<th>functional group</th>
<th>mmol/g</th>
<th>metals removed</th>
</tr>
</thead>
<tbody>
<tr>
<td>AP</td>
<td>amino propyl</td>
<td>2.0</td>
<td>Pd, Ru, Rh, Cu, Fe, Co, Ni</td>
</tr>
<tr>
<td>MP</td>
<td>mercaptopropyl</td>
<td>1.2</td>
<td>Pd, Rh, Cu, Ru, Pt, Pb, Ag, Hg</td>
</tr>
<tr>
<td>MTU</td>
<td>methylthiourea</td>
<td>1.4</td>
<td>Pd, Rh, Cu, Ru, Pb, Fe, Co</td>
</tr>
<tr>
<td>TA</td>
<td>triamine</td>
<td>1.0</td>
<td>Pd, Rh, Co, Cu, Fe, Ru, Cd, Au, V, Zn, Pt</td>
</tr>
</tbody>
</table>

- functionalised spherical mesoporous silicas
- average particle size 54 µm
- surface area 715 m²/g
- zero swell in solvents
- extremely fast scavenging
QuadraPure™ in Process Chemistry

- stirred batch or fixed bed
- fixed bed options: loose fixed bed on plant or pre-packed cartridges
  - avoid handling loose resin in reactor
  - direct translation from batch screening

**Development Scale**

- pre-packed cartridges for Biotage purification equipment
- compatible with SP and FLASH systems
- 1 to 10 mmol metal loading
- metal recovery possible from cartridge
- rapid, convenient clean-up of process streams
- linearity through sizes
QuadraPure™ in Process Chemistry

Pilot Scale

Clear-Edge Filter:

- food grade PTFE cartridges for Miniline filter housing
- development in alliance with a major pharma company
- capacity approx. 60 mmol metal (400 g resin)

Production Scale

Clear-Edge Filter:

- developing food grade PTFE cartridges
- compatible with standard filter housing units
- capacity approx. 1 mol metal (4.5 kg resin)
QuadraPure™ Grades

GMP grade:
- High-purity reagents and extensive washing regime to eliminate potential drug substance contamination
- Intended for contact use to purify active pharmaceutical ingredients
- GMP dossier available:
  - Product Quality
  - Statement on risk materials (e.g. BSE risk)
  - Certificates of analysis
  - Analytical specification and methods of analysis
  - Chemical stability and stability testing
  - Extractable impurities
  - Toxicology information

Non-GMP grade:
- Standard reagents and minimum washing, some organic contaminants may be released during processing
- Intended for use in non-pharma processing or waste-streams
Easier, Faster, Cleaner

- Easy to handle and filter robust resin beads
- Cost effective clean-up and metal recovery, reducing purification steps
- Selective metal removal with minimal loss of product

- QuadraPures™ and QuadraSils™ are readily available in bulk in GMP and non-GMP grades
- Reaxa offer full technical support including scavenging trials

info@reaxa.com
Technical Support

- Resin screening selector guide
- QuadraPure™ and QuadraSil™ user guides
- Application notes on specific metals (Pd, Ni, Rh, Cu, Fe, etc)

- Reaxa support
  - support of customers’ QA
  - technical seminars at customers’ sites
  - scavenger trials in batch and flow
  - FTE programmes to solve customers’ problems
  - custom/experimental resins
### Macroporous Palladium Removal

<table>
<thead>
<tr>
<th>QuadraPure™</th>
<th>initial Pd (ppm)</th>
<th>% Pd removal</th>
<th>Time (h)</th>
</tr>
</thead>
<tbody>
<tr>
<td>TU</td>
<td>1000</td>
<td>&gt;99</td>
<td>0.3</td>
</tr>
<tr>
<td>DET</td>
<td>1000</td>
<td>&gt;99</td>
<td>0.3</td>
</tr>
<tr>
<td>BDZ</td>
<td>1000</td>
<td>&gt;99</td>
<td>0.3</td>
</tr>
<tr>
<td>EDA</td>
<td>1000</td>
<td>&gt;99</td>
<td>0.6</td>
</tr>
<tr>
<td>BZA</td>
<td>1000</td>
<td>&gt;99</td>
<td>&lt;10</td>
</tr>
<tr>
<td>IDA</td>
<td>1000</td>
<td>&gt;99</td>
<td>&lt;10</td>
</tr>
<tr>
<td>AMPA</td>
<td>1000</td>
<td>&gt;99</td>
<td>&lt;10</td>
</tr>
</tbody>
</table>

- Pd(OAc)$_2$ 1000 ppm in THF
- 0.5 g QuadraPure™ added to 10 ml Pd solution
- TU, DET and BDZ clear in 20 minutes
## Microporous Palladium Removal

- Pd(OAc)$_2$ 1000 ppm in THF
- 0.5 g QuadraPure™ added to 10 ml Pd solution
- all clear in under 1 hour

<table>
<thead>
<tr>
<th>QuadraPure™</th>
<th>initial Pd (ppm)</th>
<th>% Pd removal</th>
<th>Time (min)</th>
</tr>
</thead>
<tbody>
<tr>
<td>MPA</td>
<td>1000</td>
<td>&gt;99</td>
<td>10</td>
</tr>
<tr>
<td>IMDAZ</td>
<td>1000</td>
<td>&gt;99</td>
<td>10</td>
</tr>
<tr>
<td>AEA</td>
<td>1000</td>
<td>&gt;99</td>
<td>35</td>
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</tbody>
</table>

**return to technical support**
QuadraSil™ Palladium Removal

<table>
<thead>
<tr>
<th>QuadraSil™</th>
<th>initial Pd (ppm)</th>
<th>% Pd removal</th>
<th>Time (min)</th>
</tr>
</thead>
<tbody>
<tr>
<td>TA</td>
<td>1000</td>
<td>&gt;99</td>
<td>5</td>
</tr>
<tr>
<td>MTU</td>
<td>1000</td>
<td>&gt;99</td>
<td>5</td>
</tr>
<tr>
<td>AP</td>
<td>1000</td>
<td>&gt;99</td>
<td>5</td>
</tr>
<tr>
<td>MP</td>
<td>1000</td>
<td>&gt;95</td>
<td>18</td>
</tr>
</tbody>
</table>

- Pd(OAc)$_2$ 1000 ppm in THF
- 0.5 g QuadraSil™ added to 10 ml Pd solution
- all clear in under 20 minutes
Macroporous Iron Removal

- **FeCl$_3$** 1000 ppm in THF
- 0.25 g QuadraPure™ added to 5 ml Fe solution
- three shown clear in 1 hour
- FeCl$_3$ scavenge also successful in cartridge

<table>
<thead>
<tr>
<th>QuadraPure™</th>
<th>initial Fe (ppm)</th>
<th>% Fe removal</th>
<th>Time (h)</th>
</tr>
</thead>
<tbody>
<tr>
<td>BZA</td>
<td>1000</td>
<td>&gt;99</td>
<td>0.6</td>
</tr>
<tr>
<td>EDA</td>
<td>1000</td>
<td>&gt;99</td>
<td>0.6</td>
</tr>
<tr>
<td>AMPA</td>
<td>1000</td>
<td>&gt;99</td>
<td>1</td>
</tr>
</tbody>
</table>

[![Image of QuadraPure™ bottles](image-url)](image-url)
Microporous Iron Removal

- FeCl₃ 1000 ppm in THF
- 0.25 g QuadraPure™ added to 5 ml Fe solution
- of microporous range only IMDAZ cleared solution

<table>
<thead>
<tr>
<th>QuadraPure™</th>
<th>initial Fe (ppm)</th>
<th>% Fe removal</th>
<th>Time (h)</th>
</tr>
</thead>
<tbody>
<tr>
<td>IMDAZ</td>
<td>1000</td>
<td>&gt;99</td>
<td>0.5</td>
</tr>
</tbody>
</table>

return to technical support
QuadraSil™ Iron Removal

<table>
<thead>
<tr>
<th>QuadraSil™</th>
<th>initial Fe (ppm)</th>
<th>% Fe removal</th>
<th>Time (h)</th>
</tr>
</thead>
<tbody>
<tr>
<td>TA</td>
<td>1000</td>
<td>&gt;99</td>
<td>0.3</td>
</tr>
<tr>
<td>AP</td>
<td>1000</td>
<td>&gt;99</td>
<td>0.3</td>
</tr>
</tbody>
</table>

- FeCl₃ 1000 ppm in THF
- 0.2 g QuadraSil™ added to 5 ml Fe solution
- all clear in 20 minutes
Other Scavengers: Iron Removal

10 ml FeCl$_3$ 1000 ppm in THF with 0.5 g of scavenger after RT overnight
Macroporous Copper Removal

<table>
<thead>
<tr>
<th>QuadraPure™</th>
<th>initial Cu (ppm)</th>
<th>% Cu removal</th>
<th>Time (h)</th>
</tr>
</thead>
<tbody>
<tr>
<td>TU</td>
<td>1000</td>
<td>&gt;99</td>
<td>0.5</td>
</tr>
<tr>
<td>IDA</td>
<td>1000</td>
<td>&gt;99</td>
<td>1</td>
</tr>
<tr>
<td>AMPA</td>
<td>1000</td>
<td>&gt;99</td>
<td>1</td>
</tr>
<tr>
<td>BDZ</td>
<td>1000</td>
<td>&gt;99</td>
<td>1.5</td>
</tr>
</tbody>
</table>

- Cu(OAc)$_2$ 1000 ppm in THF
- 0.5 g QuadraPure™ added to 10 ml Cu solution
- Cu scavenge also successful in cartridge
Microporous Copper Removal

- Cu(OAc)$_2$ 1000 ppm in THF
- 0.5 g QuadraPure™ added to 10 ml Cu solution
- all cleared in under 1 hour

<table>
<thead>
<tr>
<th>QuadraPure™</th>
<th>initial Cu (ppm)</th>
<th>% Cu removal</th>
<th>Time (min)</th>
</tr>
</thead>
<tbody>
<tr>
<td>MPA</td>
<td>1000</td>
<td>&gt;99</td>
<td>12</td>
</tr>
<tr>
<td>IMDAZ</td>
<td>1000</td>
<td>&gt;99</td>
<td>40</td>
</tr>
<tr>
<td>AEA</td>
<td>1000</td>
<td>&gt;99</td>
<td>60</td>
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</tbody>
</table>

return to technical support
QuadraSil™ Copper Removal

<table>
<thead>
<tr>
<th>QuadraSil™</th>
<th>initial Cu (ppm)</th>
<th>% Cu removal</th>
<th>Time (min)</th>
</tr>
</thead>
<tbody>
<tr>
<td>TA</td>
<td>1000</td>
<td>&gt;99</td>
<td>5</td>
</tr>
<tr>
<td>AP</td>
<td>1000</td>
<td>&gt;99</td>
<td>5</td>
</tr>
<tr>
<td>MP</td>
<td>1000</td>
<td>&gt;99</td>
<td>7</td>
</tr>
<tr>
<td>MTU</td>
<td>1000</td>
<td>&gt;99</td>
<td>30</td>
</tr>
</tbody>
</table>

- Cu(OAc)$_2$ 1000 ppm in THF
- 0.5 g QuadraSil™ added to 10 ml Cu solution
- all cleared in under 30 minutes
Other Scavengers: Copper Removal

10 ml Cu(OAc)$_2$ 1000 ppm in THF with 0.5 g of scavenger after RT overnight
Nickel Salt Removal

- Ni(acac)$_2$ 370 ppm in dichloromethane
- 0.5 g QuadraPure™ added to 10 ml Ni solution
- Macroporous and microporous resins successful in under 2 h

<table>
<thead>
<tr>
<th>QuadraPure™</th>
<th>initial Ni (ppm)</th>
<th>% Ni removal</th>
<th>Time (h)</th>
</tr>
</thead>
<tbody>
<tr>
<td>TU</td>
<td>370</td>
<td>&gt;99</td>
<td>&lt;2</td>
</tr>
<tr>
<td>IDA</td>
<td>370</td>
<td>&gt;99</td>
<td>&lt;2</td>
</tr>
<tr>
<td>AMPA</td>
<td>370</td>
<td>&gt;99</td>
<td>&lt;2</td>
</tr>
<tr>
<td>IMDAZ</td>
<td>370</td>
<td>&gt;99</td>
<td>&lt;2</td>
</tr>
</tbody>
</table>
Rhodium Salt Removal

- Rh(acac)(CO)₂ 1000 ppm in THF
- 0.5 g QuadraPure™ added to 10 ml Rh solution
- macroporous and microporous resins successful
QuadraPure™ vs Other Scavengers: Rh

- Rh(OAc)₂ 1000 ppm in DMF
- 0.5 g of each scavenger added to 10 ml Rh solution and shaken at RT overnight
- all QuadraPure™ cleared

Images showing comparison of different scavengers.
Wilkinson’s Catalyst Reaction Scavenge

- Hydrogenation of carvone performed in toluene using Wilkinson’s catalyst (>99%)
- Approx. 200 ppm Rh in reaction mixture
- 0.5 g QuadraPure™ added to 10 ml reaction solution

<table>
<thead>
<tr>
<th>QuadraPure™</th>
<th>initial Rh (ppm)</th>
<th>% Rh removal</th>
<th>Time (h)</th>
</tr>
</thead>
<tbody>
<tr>
<td>TU</td>
<td>200</td>
<td>&gt;99</td>
<td>1</td>
</tr>
<tr>
<td>AMPA</td>
<td>200</td>
<td>&gt;99</td>
<td>1.3</td>
</tr>
<tr>
<td>BZA</td>
<td>200</td>
<td>&gt;99</td>
<td>1.5</td>
</tr>
</tbody>
</table>

return to technical support
Grubb’s Catalyst Reaction Scavenge

- Grubbs catalyst in toluene approx. 595 ppm Ru in reaction mixture
- 0.25 g QuadraSil™ added to 5 ml Ru solution
- all cleared overnight (<5 ppm)

<table>
<thead>
<tr>
<th>QuadraSil™</th>
<th>initial Ru (ppm)</th>
<th>% Ru removal</th>
<th>Time (h)</th>
</tr>
</thead>
<tbody>
<tr>
<td>AP</td>
<td>595</td>
<td>&gt;99</td>
<td>1.5</td>
</tr>
<tr>
<td>MP</td>
<td>595</td>
<td>&gt;99</td>
<td>&lt;16</td>
</tr>
<tr>
<td>MTU</td>
<td>595</td>
<td>&gt;99</td>
<td>&lt;16</td>
</tr>
<tr>
<td>TA</td>
<td>595</td>
<td>&gt;99</td>
<td>&lt;16</td>
</tr>
</tbody>
</table>
Batch Scavenging Kinetics: T Effect

- QuadraPure™ TU added to 1200 ppm Pd(OAc)$_2$ solution in THF (batch)
- Scavenge time is significantly reduced by heating:
  - RT 180 min
  - 60 °C 30 min
QuadraPure™ Biotage Cartridges

<table>
<thead>
<tr>
<th>Cartridge Size</th>
<th>Approx. Mass of QuadraPure</th>
<th>Metal Loading</th>
<th>Flow Rate (~5 CV/h)</th>
</tr>
</thead>
<tbody>
<tr>
<td>12M</td>
<td>6 g</td>
<td>1 mmol</td>
<td>1 ml/min</td>
</tr>
<tr>
<td>25S</td>
<td>13 g</td>
<td>2 mmol</td>
<td>2 ml/min</td>
</tr>
<tr>
<td>40M</td>
<td>68 g</td>
<td>10 mmol</td>
<td>8 ml/min</td>
</tr>
</tbody>
</table>

QuadraPure™ TU

QuadraPure™ IDA

QuadraPure™ AMPA

QuadraPure™ BzA
QuadraPure™ Cartridges – Cu Salts

carried out in microwave at 250 °C, 30 min (>99% conversion)
slurry in DCM, filter (Celite) then QuadraPure™ IDA column (40M)

QuadraPure™ IDA
flow cartridge

Cu in crude 345 ppm

Cu <1 ppm

based on: Org. Synth. cv3, p361; vol21, p89

Rosemund von-Braun

Br

CuCN
pyridine

CN
QuadraPure™ Cartridges – Fe Salts

carried out neat at room temperature (>99% conversion)
taken up in THF and passed through QuadraPure AMPA cartridge and silica (literature method)

QuadraPure™ AMPA flow cartridge

Fe in crude 61 ppm ➔ Fe <1 ppm

silica ➔ Fe 23 ppm

*Based on: Org. Synth. cv10, p588; vol78, p249*
QuadraPure™ Cartridges – Rh Salts

Wilkinson’s catalyst

\[
\text{RhCl}(P\text{Ph}_3)_3 
\]

carried out at room temperature (>99% conversion)
immediately passed through QuadraPure AMPA cartridge

QuadraPure™ AMPA
flow cartridge

Rh in reaction mixture 309 ppm → Rh 4 ppm

based on: Org. Synth. cv6, p459; vol53, p63
QuadraPure™ Cartridges – Pd & Cu

Sonogashira coupling

QuadraPure TU multi-metal scavenging in the presence of ligand and triethylamine

Cu 54 ppm
Pd 58 ppm
PPh₃ and Et₃N in crude solution

QuadraPure™ TU flow cartridge

Cu and Pd <1 ppm no product absorption
Supplementary Slides
QuadraPure™ Screening Guide

## QuadraPure™ Metal Scavenger Resins

<table>
<thead>
<tr>
<th>Metal contamination level in solution (ppm)</th>
<th>10</th>
<th>25</th>
<th>50</th>
<th>100</th>
<th>200</th>
<th>300</th>
<th>400</th>
<th>500</th>
<th>600</th>
<th>700</th>
<th>800</th>
<th>900</th>
<th>1000</th>
<th>1500</th>
<th>2000</th>
<th>5000</th>
<th>10000</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Volume of solution/ml</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>10</td>
<td>0.01</td>
<td>0.03</td>
<td>0.05</td>
<td>0.1</td>
<td>0.2</td>
<td>0.3</td>
<td>0.4</td>
<td>0.5</td>
<td>0.6</td>
<td>0.7</td>
<td>0.8</td>
<td>1.0</td>
<td>1.5</td>
<td>2.0</td>
<td>5.0</td>
<td>10.0</td>
<td>20.0</td>
</tr>
<tr>
<td>25</td>
<td>0.03</td>
<td>0.06</td>
<td>0.13</td>
<td>0.25</td>
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<td>1.25</td>
<td>1.5</td>
<td>1.75</td>
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<td>2.25</td>
<td>2.5</td>
<td>3.75</td>
<td>5.0</td>
<td>12.5</td>
<td>25.0</td>
</tr>
<tr>
<td>50</td>
<td>0.05</td>
<td>0.13</td>
<td>0.25</td>
<td>0.5</td>
<td>1.0</td>
<td>1.5</td>
<td>2.0</td>
<td>2.5</td>
<td>3.0</td>
<td>3.5</td>
<td>4.0</td>
<td>4.5</td>
<td>5.0</td>
<td>7.5</td>
<td>10.0</td>
<td>25.0</td>
<td>50.0</td>
</tr>
<tr>
<td>75</td>
<td>0.08</td>
<td>0.19</td>
<td>0.38</td>
<td>0.75</td>
<td>1.5</td>
<td>2.25</td>
<td>3.0</td>
<td>3.75</td>
<td>4.5</td>
<td>5.25</td>
<td>6.0</td>
<td>6.75</td>
<td>7.5</td>
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Grammes of QuadraPure™ Required
QuadraSil™ vs Competitors’ Silicas

QuadraSil™

ScavNets™

Silicycles
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