Encapsulated Catalysts and Metal Scavengers

Sigma-Aldrich and Reaxa partner to bring you...
...easier, faster, cleaner processes.
Easier, Faster, Cleaner Processes with Encapsulated Catalysts and Metal Scavengers

Sigma-Aldrich is pleased to partner with Reaxa Ltd to offer an array of new technologies for precious metal catalysis to achieve easier, faster and cleaner processes. The technology integrates smoothly with existing methods, reduces purification steps, cuts contamination, and allows for efficient recovery of precious metals. Encapsulated catalysts (EnCat™) and metal scavengers (QuadraPure™ and QuadraSil™) are available exclusively through Sigma-Aldrich at research scale.

Dihydroxylation Catalyst

**Os EnCat™ 40**

Osmium tetroxide is known to be one of the most reliable and effective catalysts for oxidative cleavage and dihydroxylation of olefins. However, it is very toxic, volatile and expensive, limiting its use at large-scale. Os EnCat™ 40 effectively immobilizes and stabilizes osmium tetroxide to produce a safer and easier to use catalyst for use in oxidation chemistry. In the picture on the right, a sample of Os EnCat™ 40 placed in corn oil is unchanged after several months, whereas an equivalent sample of volatile osmium tetroxide quickly reacts to produce black decomposition products.

<table>
<thead>
<tr>
<th>Product No.</th>
<th>Name</th>
<th>Metal content (w/w%)</th>
<th>Loading (mmol/g)</th>
<th>Particle size range in µm (average)</th>
</tr>
</thead>
<tbody>
<tr>
<td>658685</td>
<td>Os EnCat™ 40</td>
<td>4.8 – 5.7</td>
<td>0.25 – 0.30</td>
<td>40 – 300 (165)</td>
</tr>
</tbody>
</table>

**Cleaner Products**

Typically less than 100 ppm Os in crude product

**Easier storage, transport & use**

Low residual OsO₄ vapor over Os EnCat™ 40 product

**Faster, efficient processes**

Resin beads filter easily

**High selectivity**

Can be used for asymmetric dihydroxylation

**Reduced plant contamination**

OsO₄ is trapped in the beads

**Improved cost effectiveness**

Os EnCat™ 40 may be reused in repeat reaction cycles
Coupling Catalysts

Pd(II) EnCat™

Pd(II) EnCat™ covers a versatile range of immobilized palladium acetate catalysts with or without activating ligands contained in a robust, porous polyurea matrix which allow easier, faster and cleaner C–C coupling processes to be developed.

Cleaner products
- typically less than 10 ppm Pd in crude product

Cleaner waste streams
- reduced metal losses in Pd(II) EnCat™ processes

Fast, efficient processes
- EnCat™ beads filter easily

No plant contamination
- metal & ligands are trapped within the beads

Improved processes
- high activity in many types of coupling reactions

Process intensification
- can be used in batch & flow processes

LaPCat™

An exclusive collection of palladium and copper containing perovskite heterogeneous catalysts for use in a range of organic reactions. The LaPCat™ perovskite catalysts have shown very high catalytic activity in many coupling reactions with a wide variety of substrates.

Cleaner products
- typically 10 ppm or less of metal contamination in crude product

Cleaner waste streams
- reduced metal losses in LaPCat™ processes

Fast, efficient processes
- equivalent or better than homogeneous catalysts

Improved processes
- very low catalyst loadings achieved

Improved cost effectiveness
- LaPCat™ catalysts may be reused in repeat reaction cycles

Product No. | Name | Metal content (w/w%) | Co-encapsulated ligand
---|---|---|---
644714 | Pd(II) EnCat™ 30 | 4.3 | none
644722 | Pd(II) EnCat™ 40 | 4.6 | none
644706 | Pd(II) EnCat™ TPP30 | 4.7 | 
644692 | Pd(II) EnCat™ TOTP30 | 4.7 | 
658693 | Pd(II) EnCat™ BINAP30 | 4.7 | 
666335 | Pd(II) EnCat™ Kit I (five component kit of 1 gram units of each Pd(II) EnCat™) | 4.7 | 

LaPCat™ Perovskite Catalyst Kit I (100 mg units of each LaPCat™)

<table>
<thead>
<tr>
<th>Catalyst</th>
<th>Formulation</th>
<th>Surface area (m²/g)</th>
<th>Particle size (µm)</th>
<th>Reactions</th>
</tr>
</thead>
<tbody>
<tr>
<td>FP2</td>
<td>LaFe₀.₃₅Pd₀.₆₅O₃</td>
<td>5.5</td>
<td>41.1</td>
<td>Suzuki</td>
</tr>
<tr>
<td>FP8</td>
<td>LaFe₀.₂₅Pd₀.₇₅O₃</td>
<td>5.7</td>
<td>3.3</td>
<td>Suzuki</td>
</tr>
<tr>
<td>CoP</td>
<td>LaFe₀.₁₇Co₀.₈₃Pd₀.₅O₃</td>
<td>3.4</td>
<td>4.0</td>
<td>Suzuki, Heck, Sonogashira</td>
</tr>
<tr>
<td>CuP</td>
<td>LaFe₀.₁₇Cu₀.₈₃Pd₀.₅O₃</td>
<td>12</td>
<td>3.7</td>
<td>Suzuki, Sonogashira, Ullmann</td>
</tr>
<tr>
<td>CoCu</td>
<td>La₃.₃₂Ce₅.₇₂Co₀.₄₆Cu₀.₄₆O₃</td>
<td>7.9</td>
<td>5.4</td>
<td>Ullmann</td>
</tr>
<tr>
<td>YBCu</td>
<td>YBa₂Cu₃O₇</td>
<td>0.3</td>
<td>10.6</td>
<td>Ullmann</td>
</tr>
</tbody>
</table>
Hydrogenation Catalysts

**Pd(0) EnCat™ 30NP**

Pd(0) EnCat™ 30NP is a versatile polymer encapsulated nanoparticulate Pd(0) hydrogenation and transfer hydrogenation catalyst with high chemoselectivity and improved safety profile allowing easier, faster and cleaner processes to be developed. Reaxa’s controlled manufacturing process produces regular palladium(0) nano-particles stabilized by the polymer matrix within the EnCat™ beads, ensuring that the catalyst performance is extremely reproducible from batch to batch (c.f. palladium on carbon).

**Cleaner products** typically less than 1 ppm Pd in crude product

**Cleaner waste streams** reduced metal losses in EnCat™ processes

**Fast, efficient processes** EnCat™ beads filter easily and can be recycled

**No plant contamination** palladium metal is trapped within the beads

**High selectivity** in hydrogenation & transfer hydrogenation reactions

**Improved safety profile** non-pyrophoric compared with Pd/C catalysts

**Process intensification** can be used in batch & flow processes

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![Chemical reaction](image)

**Pt(0) EnCat™ 40**

Pt(0) EnCat™ 40 is a versatile hydrogenation catalyst demonstrating high chemoselectivity and improved safety profile over platinum on carbon allowing easier, faster and cleaner processes to be developed. Isolating the active platinum within the polymer matrix has produced an easily handled and safer alternative to Pt/C.

**Cleaner products** very low levels of Pt in crude product

**Cleaner waste streams** reduced metal losses in EnCat™ process (typically <0.005% Pt loss)

**Fast, efficient processes** EnCat™ beads filter easily

**No plant contamination** platinum metal remains entrapped within the beads

**High selectivity** in hydrogenation reactions

**Process intensification** can be used in batch & flow processes

**Improved safety profile** initial tests show no tendency for activated catalyst to ignite upon exposure to air

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For detailed technical information on these encapsulated catalysts or metal scavengers, including application guides and references, please visit sigma-aldrich.com/reaxa.
**Metal Scavengers**

QuadraPure™ and QuadraSil™ metal scavengers offer a variety of complementary solutions for efficient removal and economic recovery of precious metal contamination from products and process streams (aqueous or organic). The picture shows a typical ‘before-and-after’ scavenger treatment of a metal catalyst solution (initial metal content 1000 ppm) at room temperature.

### QuadraPure™

The QuadraPure™ collection consists of two ranges of functionalized polystyrene beads:

- Macroporous, low-swell, robust beads for use in manufacturing either in batch or flow processes.
- Microporous, solvent swellable beads with high capacity intended largely for R&D application.

<table>
<thead>
<tr>
<th>Product No.</th>
<th>Name</th>
<th>Functional group</th>
<th>Examples of metals removed</th>
</tr>
</thead>
<tbody>
<tr>
<td>655422</td>
<td>QuadraPure™ 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>657026</td>
<td>QuadraPure™ IDA</td>
<td>iminodiacetate</td>
<td>Cu, Al, Ga, In, V, Pb, Ni, Zn, Cd, Be, Mn, Sr, Ba, Co, Fe</td>
</tr>
<tr>
<td>657611</td>
<td>QuadraPure™ AMPA</td>
<td>aminomethyl-phosphonic acid</td>
<td>Fe, Cu, Ni, Al, Co, V</td>
</tr>
<tr>
<td>668591</td>
<td>QuadraPure™ BZA</td>
<td>benzylamine</td>
<td>Rh, Pd, Cu, Co, V</td>
</tr>
<tr>
<td>668605</td>
<td>QuadraPure™ BDZ</td>
<td>imidazole</td>
<td>Rh, Co, Pd, Ni</td>
</tr>
<tr>
<td>668583</td>
<td>QuadraPure™ EDA</td>
<td>amine</td>
<td>Pd in basic media and with phosphines, Co, Ni, Rh</td>
</tr>
<tr>
<td>668575</td>
<td>QuadraPure™ DET</td>
<td>thiol</td>
<td>Pd in acidic media and with phosphines, Co, Fe, Ni</td>
</tr>
<tr>
<td>657654</td>
<td>QuadraPure™ IMDAZ</td>
<td>imidazolylpropyl-amino</td>
<td>Pd, Ru, Os, Co, Ni, V, Rh, Cu, Fe, Sn</td>
</tr>
<tr>
<td>657662</td>
<td>QuadraPure™ MPA</td>
<td>mercaptophenyl-amino</td>
<td>Pd, Pt, Rh, Ni, Ru, Cu, Sn, Ag, Au, Cd, Hg, Pb</td>
</tr>
<tr>
<td>657646</td>
<td>QuadraPure™ AEA</td>
<td>aminoethylamino</td>
<td>Pd, Rh, V, Cu, Fe</td>
</tr>
</tbody>
</table>

### QuadraSil™

The QuadraSil™ scavengers offer extremely rapid removal of metal catalyst residues. QuadraSil™ products are based on high-quality spherical silica beads with well defined porosity that can be used in aqueous or organic solution and in batch or flow processes.

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<tr>
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<th>Name</th>
<th>Functional group</th>
<th>Examples of metals removed</th>
</tr>
</thead>
<tbody>
<tr>
<td>679534</td>
<td>QuadraSil™ AP</td>
<td>aminopropyl</td>
<td>Pd, Ru, Rh, Cu, Fe, Co, Ni</td>
</tr>
<tr>
<td>679526</td>
<td>QuadraSil™ MP</td>
<td>mercaptopropyl</td>
<td>Pd, Rh, Cu, Ru, Pt, Pb, Ag, Hg</td>
</tr>
<tr>
<td>679518</td>
<td>QuadraSil™ MTU</td>
<td>methythiourea</td>
<td>Pd, Rh, Cu, Ru, Pb, Fe, Co</td>
</tr>
<tr>
<td>679496</td>
<td>QuadraSil™ TA</td>
<td>triamine</td>
<td>Pd, Rh, Co, Cu, Fe, Ru, Cd, Au, V, Zn, Pt</td>
</tr>
</tbody>
</table>

680427 QuadraSil™ Metal Scavengers Kit I (four component kit of 5 g units of each QuadraSil™)