A New Generation of Columns for Pesticide Analysis
The use of many pesticides has been limited or discontinued due to the health and environmental risks they pose. However, the persistence of these compounds in the environment has left many sites heavily contaminated and in need of remediation. There is also international concern for the residual level of pesticides in foods, especially those imported from countries where banned pesticides are still permitted.

Though the standard method for pesticide testing varies throughout the world, many labs use Electron Capture Detectors (ECD), Nitrogen Phosphorous Detectors (NPD) or Flame Photometric Detectors (FPD) because of the sensitivity level they offer for specific compound classes. While these detectors are extremely sensitive, they do not provide spectral confirmation of the analyte. In order to provide positive confirmation, Mass Spectrometry (MS) must be used. The GC/MS can be used in Selected Ion Monitoring (SIM) mode to provide sensitivity levels similar to the previously mentioned detectors, while also providing positive confirmation of the analyte in question.

Regardless of the technique, the new MultiResidue GC columns have been optimized for pesticides, herbicide, or insecticide analysis. Each column has a unique selectivity that allows them to be used together in dual column confirmation analysis. Both phases are MS Certified, so they can also be used on MS to confirm results. We have run a number of pesticide compounds on these phases. If you don’t find the application you are looking for in this brochure, be sure to contact your local Phenomenex representative.
Zebron™ MultiResidue™ (MR) Columns

- Optimized Selectivity
- Two Complementary Phase Chemistries
- MS Certified Bleed Levels
- High Temperature Limits (320/340 °C)
- Very Low Column Activity

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<td>DIN 51527</td>
<td>ECD</td>
<td>17</td>
</tr>
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</table>
A New Generation of Columns for Pesticide Analysis

Optimized Selectivity

Many pesticide compounds have very similar structures or have isomers that are present in the product. Zebron™ MultiResidue™ phases were developed to provide optimum selectivity for a variety of pesticide compounds. Every analysis is unique, but we can help you determine the best phase to use in order to resolve your pesticide sample.

Figure 1: Selectivity of DDT, DDD, and DDE isomers at 25 ng on column using MS

Sample:

1. o,p-DDE
2. p,p-DDE
3. o,p-DDD
4. o,p-DDT
5. p,p-DDD
6. p,p-DDT

Low Bleed

Zebron columns have developed a reputation for low column bleed and high temperature limits and Zebron MultiResidue columns are no different. The columns are made using an extremely stable siloxane-based polymer that contains absolutely no cyano functionality. Our Engineered Self Crosslinking™ (ESC) bonding process makes both columns MS certified, allowing for unmatched spectral integrity even for low-level samples.
Low Column Activity

Many pesticides are sensitive to system activity and readily breakdown. This can be a significant problem when working with low level samples and sensitive detectors such as ECD. Zebron™ MultiResidue™ columns have been completely deactivated and provide excellent peak shape for even the most active compounds.

Tunable Selectivity

Zebron MultiResidue GC columns were developed specifically for pesticide analysis. Their unique selectivity improves the separation of all classes of pesticides, herbicides, and insecticides. Since the columns are MS Certified, samples can be prescreened using analyte specific detectors, and then confirmed using MS.

Sensitive and Stable

Figure 3: Repeatability at 5 pg on-column concentration of various pesticides using Zebron MR-1 and MR-2 columns

Table 1: Relative standard deviation (RSD) for five replicate injections of pesticide at 5 pg on-column concentration
The EPA outlines strict performance guidelines that must be met for compound linearity, percent relative standard deviation (% RSD), and breakdown of DDT and endrin. Column resolution and performance are critical in meeting these requirements. The data below was calculated using EPA Method 8081A guidelines.

Table 1: Five-point calibration curve at 5, 10, 25, 100, and 250 ppb

<table>
<thead>
<tr>
<th>Analyte</th>
<th>Zebron™ MR-1 % RSD</th>
<th>Zebron™ MR-2 % RSD</th>
<th>US EPA Specifications</th>
</tr>
</thead>
<tbody>
<tr>
<td>α-BHC</td>
<td>6.75</td>
<td>7.91</td>
<td>&lt; 20</td>
</tr>
<tr>
<td>γ-BHC (Lindane)</td>
<td>8.52</td>
<td>5.70</td>
<td>&lt; 20</td>
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<tr>
<td>β-BHC</td>
<td>3.57</td>
<td>9.21</td>
<td>&lt; 20</td>
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<td>Heptachlor</td>
<td>5.90</td>
<td>7.58</td>
<td>&lt; 20</td>
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<tr>
<td>Aldrin</td>
<td>4.21</td>
<td>5.37</td>
<td>&lt; 20</td>
</tr>
<tr>
<td>Heptachlor epoxide</td>
<td>4.34</td>
<td>5.25</td>
<td>&lt; 20</td>
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<tr>
<td>γ-Chlordane</td>
<td>3.70</td>
<td>4.48</td>
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<tr>
<td>α-Chlordane</td>
<td>2.91</td>
<td>3.39</td>
<td>&lt; 20</td>
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<tr>
<td>Endosulfan I</td>
<td>2.93</td>
<td>3.91</td>
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<tr>
<td>DDE</td>
<td>4.56</td>
<td>6.77</td>
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<tr>
<td>Dieldrin</td>
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<tr>
<td>Endrin</td>
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<td>DDD</td>
<td>4.79</td>
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<td>Endosulfan II</td>
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<td>DDT</td>
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<td>Endosulfan sulfate</td>
<td>3.31</td>
<td>3.20</td>
<td>&lt; 20</td>
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<tr>
<td>Methoxychlor</td>
<td>7.39</td>
<td>4.21</td>
<td>&lt; 20</td>
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<tr>
<td>Endrin ketone</td>
<td>3.48</td>
<td>3.95</td>
<td>&lt; 20</td>
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<tr>
<td><strong>Average</strong></td>
<td><strong>4.28</strong></td>
<td><strong>5.21</strong></td>
<td><strong>&lt; 20</strong></td>
</tr>
</tbody>
</table>

*Calculated using response factors as per EPA guidelines

Table 2: Percent breakdown of endrin & DDT as per EPA guidelines

<table>
<thead>
<tr>
<th>Analyte</th>
<th>Zebron MR-1 % Breakdown</th>
<th>Zebron MR-2 % Breakdown</th>
<th>US EPA Requirements</th>
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<tbody>
<tr>
<td>Endrin</td>
<td>5.3</td>
<td>7.0</td>
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<tr>
<td>DDT</td>
<td>2.3</td>
<td>2.9</td>
<td>&lt; 15</td>
</tr>
</tbody>
</table>
Baseline Resolution of all 20 EPA 8081A’s Chlorinated Pesticides!

The US EPA regulates the testing of 20 specific chlorinated pesticides under the official Method 8081A. The method specifies an Electron Capture Detector (ECD), which is extremely sensitive for chlorinated compounds. However, it does not provide any confirmatory information about the peak.

To reduce the occurrence of misidentifications, the method requires the use of two GC columns of dissimilar selectivity in a parallel configuration. The EPA considers an analyte’s presence confirmed if it has a peak at the pre-determined retention time on both columns. The unique selectivity of Zebron™ MultiResidue™ columns allows for baseline resolution ($R_s > 1.5$) of all compounds with two elution order changes between the columns.

**Column:**
Zebron MultiResidue-1
Zebron MultiResidue-2

**Dimensions:**
30 meter x 0.32 mm x 0.50 µm
30 meter x 0.32 mm x 0.25 µm

**Part No.:**
7HM-G016-17; 7HM-G017-11

**Injection:**
Splitless @ 250 °C, 1 µL

**Carrier Gas:**
Helium @ 3.4 mL/min (constant flow)

**Oven Program:**
100 °C for 0.5 min to 220 °C @ 35 °C/min to 340 °C at 20 °C/min for 2 min

**Detector:**
ECD @ 350 °C

**Notes:** Columns connected using a 5 m Z-Guard and a Y-splitter

**Sample:**
1. Tetrachloro-m-xylene (TCMX) (surr)
2. 1-Bromo-2-nitrobenzene
3. α-BHC
4. γ-BHC (Lindane)
5. β-BHC
6. δ-BHC
7. Heptachlor
8. Aldrin
9. Heptachlor epoxide
10. γ-Chlordane (trans)
11. α-Chlordane (cis)
12. Endosulfan I
13. 4,4'-DDE
14. Dieldrin
15. Endrin
16. 4,4'-DDD
17. Endosulfan II
18. Endrin aldehyde
19. 4,4'-DDT
20. Endosulfan sulfate
21. Methoxychlor
22. Endrin ketone
23. Decachlorobiphenyl (DCB) (surr)
Zebron™ MultiResidue™ columns were designed to provide optimized resolution of all classes of pesticides. This unique selectivity allows your lab to maintain separation of critical compounds even when analysis times are shortened by more than 40%. The increase in lab productivity makes sure you get results to your customers on time, even for rush samples.

7 Instruments Required to Run 500 Samples Per Day

<table>
<thead>
<tr>
<th>Instruments</th>
<th>Samples</th>
</tr>
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<tr>
<td>1</td>
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<tr>
<td>2</td>
<td>80</td>
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<td>80</td>
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<td>4</td>
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<td>5</td>
<td>80</td>
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<tr>
<td>6</td>
<td>80</td>
</tr>
<tr>
<td>7</td>
<td>80</td>
</tr>
</tbody>
</table>

**Standard Conditions**

- **Column:** Zebron MultiResidue-1, Zebron MultiResidue-2
- **Dimensions:** 30 meter x 0.53 mm x 0.50 μm, 30 meter x 0.53 mm x 0.50 μm
- **Part No.:** THK-G016-17, THK-G017-17
- **Injection:** Splitless @ 250 °C, 1 µL
- **Carrier Gas:** Helium @ 5.2 mL/min (constant flow)
- **Oven Program:** 90 °C for 0.5 min to 320 °C @ 15 °C/min for 5 min
- **Detector:** ECD @ 350 °C
Same Columns + Optimized Conditions = Increased Productivity

3.5 Instruments Required to Run 500 Samples Per Day

144 Samples
144 Samples
144 Samples
68 Samples with time to spare!
These instruments available for other analysis

Optimized Conditions

Sample for all columns on p. 8-9:
1. Tetrachloro-m-xylene (TCMX) (surr)
2. 1-Bromo-2-nitrobenzene (IS)
3. α-BHC
4. γ-BHC (Lindane)
5. β-BHC
6. δ-BHC
7. Heptachlor
8. Aldrin
9. Heptachlor epoxide
10. trans-Chlordane (gamma)
11. cis-Chlordane (alpha)
12. Endosulfan I
13. 4,4'-DDE
14. Dieldrin
15. Endrin
16. DDD
17. Endosulfan II
18. Endrin aldehyde
19. DDT
20. Endosulfan sulfate
21. Methoxychlor
22. Endrin ketone
23. Decachlorobiphenyl (DCB) (surr)

44 % Shorter Analysis
Almost no change in resolution!

Tech Tip:
High flow rates reduce a compound’s residence time in the inlet. This can significantly reduce injection port breakdown for sensitive compounds such as Endrin & DDT.
Pesticides are widely used by farmers to control pests, weeds and molds that would otherwise decrease crop production. While this has significantly increased worldwide food productions, these same pesticides pose significant health and environmental risks.

The restrictions for specific pesticides differ from one country to the next. As world trade increases, the potential threat to other countries’ populations increases. This is especially true in the European Union, where produce can be transported from one country to another quite easily.

For this reason, pesticides are the subject of increasing regulation. Since many different types of pesticides can be used on the same food product, multiple residue screening approaches are used to test for more than 300 compounds. Gas Chromatography (GC) is still the most common test method for the majority of pesticide classes. While ECD or NPD may be used for screening, MS detection must be employed to provide positive confirmation.

Zebron™ MultiResidue™ columns are well suited for use on all types of detectors. They provide very low bleed levels even at elevated temperatures required to remove matrix contamination during high temperature bake outs. When used in conjunction with a screening method that uses an analyte specific detector such as ECD, Zebron MR-1 and MR-2 columns can be a powerful tool in identifying positive samples.
Confirmation of Multi-Pesticide Mixture by GC/MS

The low bleed performance of both Zebron™ MultiResidue™ MR-1 and MR-2 columns allow them to be used on GC/MS.

Retention Time Data

Retention time data is available for over 300 pesticides on a Zebron MultiResidue-1 column. Please contact your local Phenomenex representative for more details.
Aroclor 1242

MR-1

MR-2

Aroclor 1260

MR-1

MR-2

Columns: Zebron MultiResidue-1
Zebron MultiResidue-2

Dimensions: 30 meter x 0.32 mm x 0.50 µm
30 meter x 0.32 mm x 0.25 µm

Part No.: 7HM-G016-17; 7HM-G017-11

Injection: Split 50:1 @ 210 °C, 1 µL

Carrier Gas: Hydrogen @ 2.3 mL/min (constant pressure)

Oven Program: 120 °C (hold 1 min) to 300 °C @ 9 °C/min (hold 10 min)

Detector: ECD @ 310 °C

Sample: 1. Tetrachloro-m-xylene (TCMX) (surr)
2. Decachlorobiphenyl (DCB) (surr)

Notes: Columns connected using a 5 meter Z-Guard and a Y-splitter

How long will columns last?

The performance of a GC system will degrade over time requiring inlet maintenance, column trimming, and ultimately the replacement of the GC column. In order to accurately determine what your column lifetime will be it’s better to ask yourself the question: What makes your columns fail? Is it bleed, activity, or poor resolution?

The better the column performance for that criteria initially will usually lead to better overall performance in the long term. Zebron MultiResidue columns provide the lowest activity, best resolution, and overall most stable performance of any pair of columns designed specifically for pesticide testing.
RCP/MCP States of Connecticut and Massachusetts Pesticide List

Column: Zebron MultiResidue-1
Zebron MultiResidue-2
Dimensions: 30 meter x 0.53 mm x 0.50 µm
Part No.: 78K-G016-17, 78K-G017-17
Injection: Splitless @ 250 °C, 2 µL
Carrier Gas: Helium @ 6.7 psi (constant pressure)
Oven Program: 130 °C for 1 min to 325 °C @ 15 °C/min (hold 5 min)
Detector: ECD @ 380 °C

Sample:
1. Hexachlorocyclopentadiene
2. Propachlor
3. Tetrachloro-m-xylene (TCMX) (surr)
4. α-BHC
5. Hexachlorobenzene
6. γ-BHC (Lindane)
7. β-BHC
8. Alachlor
9. δ-BHC
10. Heptachlor
11. Aldrin
12. Heptachlor epoxide
13. γ-Chlordane
14. α-Chlordane
15. Endosulfan I
16. 4,4’-DDD
17. Dieldrin
18. Endrin
19. 4,4’-DDT
20. Endosulfan II
21. Endrin aldehyde
22. 4,4’-DDT
23. Endosulfan sulfate
24. Methoxychlor
25. Endrin ketone
26. Decachlorobiphenyl (DCB) (surr)

Do you have a noteworthy application using Zebron GC columns that you would like published?

For consideration, please send your chromatogram and a short abstract to sky@Phenomenex.com or fax it to (310) 328-7768 attn: Sky Countryman. Any type of application is welcome.
Column: Zebron MultiResidue™-1
Zebron MultiResidue™-2
Dimensions: 30 meter × 0.32 mm × 0.50 µm
30 meter × 0.32 mm × 0.25 µm
Part No.: 7HM-G016-17; 7HM-G017-11
Injection: On-column @ 103 °C, 1 µL
Carrier Gas: Helium @ 2.8 mL/min (constant flow)
Oven Program: 100 °C for 0.5 min to 180 °C @ 20 °C/min
to 240 °C @ 6 °C/min to 320 °C @ 15 °C/min for 2 min
Detector: FID @ 340 °C
Notes: Analytes at 2 ppm in dichloromethane. Columns connected using a 5 m Z-Guard and a Y-splitter

Sample:
1. Dichlorvos
2. Mevinphos
3. Trichlorfon
4. TEPP (Tetraethyl Pyrophosphate)
5. Demeton isomer
6. Thionazin
7. Ethoprop
8. Sulfotep
9. Naled
10. Dicrotophos
11. Phorate
12. Monocrotophos
13. Demeton
14. Terbutphos
15. Diazinon
16. Dimethoate
17. Fonofox
18. Phosphamidon isomer
19. Disulfoton
20. Dichlofenthion
21. Phosphamidon
22. Chlorpyrifos methyl
23. Ronnel
24. Aspon
25. Methyl parathion
26. Malathion
27. Fenitrothion
28. Chlorpyrifos
29. Fenthion
30. Trichloronate
31. Parathion
32. Merphos
33. Chlorfenvinphos
34. Crotamiton
35. Stirofos
36. Tokuthion
37. Merphos oxide (tribufos)
38. Ethion
39. Fensulfothion
40. Contaminant
41. Carbathion
42. Famfur
43. EPN
44. Phosmet
45. Leptophos
46. Azinphos methyl
47. Azinphos ethyl
48. Couphomos
**EPA Method 615/815**

**Chlorophenoxy Acid Herbicides**

**US EPA 615/815**

**MR-1**
- **Column:** Zebron MultiResidue-1
- **Dimensions:** 30 meter x 0.32 mm x 0.50 μm
- **Part No.:** 7HM-G016-17, 7HM-G017-11
- **Injection:** Splitless @ 250 °C, 1 µL
- **Carrier Gas:** Helium @ 2.5 mL/min (constant flow)
- **Oven Program:** 50 °C for 1 min to 180 °C @ 35 °C/min for 2 min to 205 °C @ 5 °C/min to 320 °C @ 20 °C/min
- **Detector:** ECD @ 350 °C

**MR-2**
- **Column:** Zebron MultiResidue-2
- **Dimensions:** 30 meter x 0.32 mm x 0.50 μm
- **Part No.:** 7HM-G016-17, 7HM-G017-11
- **Injection:** Splitless @ 250 °C, 1 µL
- **Carrier Gas:** Helium @ 2.5 mL/min (constant flow)
- **Oven Program:** 50 °C for 1 min to 180 °C @ 35 °C/min for 2 min to 205 °C @ 5 °C/min to 320 °C @ 20 °C/min
- **Detector:** ECD @ 350 °C

**Sample:**
1. Dalapon
2. 3,5-Dichlorobenzoic acid
3. 4-Nitrophenol
4. DCAA (surr)
5. Dicamba
6. MCPP
7. MCPA
8. Dichlorprop
9. Contaminant
10. 2,4-D
11. Pentachlorophenol
12. DBOB (IS)
13. Silvex
14. Chloramben
15. 2,4,5-T
16. Dinoseb
17. 2,4-DB
18. Bentazon
19. Picloram
20. DCPA
21. Acifluorfen

**Request Additional EPA Methods**
- **8082** Aroclors
- **507** Nitrogen- and Phosphorus-Containing Pesticides
- **8081A** Chlorinated Pesticides
**US EPA 610/8100**

**Polyaromatic Hydrocarbons**

**MR-1**

**Column:** Zebron MultiResidue-1  
**Dimensions:** 30 meter x 0.25 mm x 0.25 µm  
**Part No.:** 7HG-G016-11  
**Injection:** Splitless @ 300 °C, 1 µL  
**Carrier Gas:** Helium @ 1.4 mL/min (constant flow)  
**Oven Program:** 70 °C for 0.5 min to 275 °C @ 25 °C/min to 340 °C @ 8 °C/min for 7 min  
**Detector:** MSD @ 320 °C; 45-400

**Sample:**
1. Naphthalene  
2. 2-Methylnaphthalene  
3. Acenaphthylene  
4. Acenaphthene  
5. Fluorene  
6. Phenanthrene  
7. Anthracene  
8. Fluoranthene  
9. Pyrene  
10. Benz[a]fluorene  
11. Benz[a]anthracene  
12. Cyclopenta[c]dipyrrene  
13. Chrysene  
14. Methylchrysene  
15. Benz[b]fluoranthene  
16. Benz[g]fluoranthene  
17. Benz[j]fluoranthene  
18. Benz[k]pyrene  
19. 3-Methylcholanthrene  
20. Dibenzo[a,h]acridine  
21. Dibenzo[a,i]acridine  
22. Indeno[1,2,3-cd]pyrene  
23. Dibenzo[a,h]anthracene  
24. Benz[a,h]perylene  
25. 7H-Dibenzo[c,g]carbazole  
26. Dibenzo[a]pyrene  
27. Dibenzo[a]pyrene  
28. Dibenzo[a]pyrene  
29. Dibenzo[a]pyrene

**Tech Tip:**
Inlet deactivation is critical for obtaining stable calibration curves. Use a liner style that has a taper at the bottom to help focus analytes onto the column. Avoid liners with glass wool because it adds activity. Also remember to change your gold seal regularly when working with Agilent® 5890 or 6890 instruments.
DIN Method 51527: Polychlorinated Biphenyls Separation

Resolving Critical Isomers

Polychlorinated Biphenyls (PCBs) are a class of priority environmental pollutants that have been identified for international regulation. The similarity in structure and polarity makes the resolution of certain isomers challenging on standard phases. German law requires separation of specific PCB congeners under DIN Method 51527.

The unique selectivity offered by Zebron™ MultiResidue™ columns makes them a versatile solution for many applications outside of pesticide analysis. Both the MR-1 and the MR-2 columns provide resolution of all the required PCB congeners in less than 30 minutes, allowing for simultaneous confirmation of each sample.
Ordering Information

Choose the Best Column Set for Your Needs

We know every lab is different and we want to help make choosing the right GC column easy. Zebron™ MultiResidue™ columns are available in a variety of formats to meet your lab’s needs.

0.25 mm ID

Provides the overall best resolution, lowest bleed, and greatest sensitivity. Recommended when using GC/MS or when needing the lowest detection levels possible. Does not handle highly contaminated samples well, column lifetime might be reduced.

0.32 mm ID

Good compromise between resolution and column stability. Recommended when working with dual column setups where high level samples will be encountered. Works well on newer MS systems. High flow rates decrease residence time in the inlet.

0.53 mm ID

Recommended when working with highly contaminated samples. Good for screening instruments. Resolution and sensitivity will be slightly reduced when compared to 0.32 or 0.25 mm ID columns. However, Zebron MultiResidue phases have very high selectivity for pesticides. Thus, resolution will be greater than other phases recommended for this work.

Phenomenex Recommended Liners

<table>
<thead>
<tr>
<th>Description</th>
<th>GC Instruments &amp; Model No.</th>
<th>Dimensions ID x L x OD (mm)</th>
<th>Part No.</th>
<th>Unit</th>
<th>Price</th>
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<tbody>
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<td>Agilent 5880/5890/6890</td>
<td>4 x 78.5 x 6.3</td>
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<td>Splitless Liner</td>
<td>Varian 1075/1077</td>
<td>2 x 74 x 6.3</td>
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<td>Single Taper Direct Connect with Side Hole (top)</td>
<td>Agilent 5880/5890/6890</td>
<td>4 x 78.5 x 6.3</td>
<td>AG0-7850</td>
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<td>Single Taper Direct Connect with Side Hole (bottom)</td>
<td>Agilent 5880/5980/6890</td>
<td>4 x 78.5 x 6.3</td>
<td>AG0-7851</td>
<td>5/pk</td>
<td></td>
</tr>
</tbody>
</table>
Gold Inlet Base Seals

<table>
<thead>
<tr>
<th>ID (mm)</th>
<th>Temp. Limits °C</th>
<th>df (µm)</th>
<th>Part No.</th>
<th>df (µm)</th>
<th>Part No.</th>
<th>Price</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>-60 to 320/340</td>
<td>0.25</td>
<td>7HG-G016-11</td>
<td>0.20</td>
<td>7HG-G017-10</td>
<td></td>
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<tr>
<td>0.25</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>0.32</td>
<td></td>
<td>0.50</td>
<td>7HM-G016-17</td>
<td>0.25</td>
<td>7HM-G017-11</td>
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<tr>
<td>0.53</td>
<td></td>
<td>0.50</td>
<td>7HK-G016-17</td>
<td>0.50</td>
<td>7HK-G017-17</td>
<td></td>
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</tbody>
</table>

Replacement Inlet Seal Washers

<table>
<thead>
<tr>
<th>Part No.</th>
<th>Description</th>
<th>Similar to Mfr. No.*</th>
<th>Unit</th>
<th>Price</th>
</tr>
</thead>
<tbody>
<tr>
<td>AG0-7522</td>
<td>Inlet Seal Washers, for Agilent® GC injection port</td>
<td>5061-5869</td>
<td>12/pk</td>
<td></td>
</tr>
</tbody>
</table>
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