

MACHEREY-NAGEL

CHROMABOND®

QuEChERS



Convenient solutions for  
your pesticide residue analysis

SPE



Be the early bird – Catch them with the right mix!

Proven CHROMABOND® Diamino  
(PSA) – pre-mixed in  
QuEChERS mixes



Chromatography

# CHROMABOND® QuEChERS

## QuEChERS methodology

“Quick, Easy, Cheap, Effective, Rugged and Safe” – these are the demands of sample preparation in modern food analysis. The QuEChERS (pronounced as “catchers”) method, introduced by Anastassiades et al. in 2003,<sup>[1]</sup> and the subsequent development of ready-to-use mixes meet these needs. QuEChERS became the method of choice in sample preparation for the analysis of pesticides and drugs in fruit, vegetables and other food products.



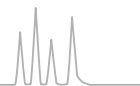
## Advantages of QuEChERS in comparison with classical clean-up methods:

- Easy to handle and time-saving procedure
- No need for glassware
- Low consumption of solvents
- No need for chlorinated solvents
- Broad range of pesticides can be detected
- Rugged method with high and safe recovery rates

**Improved lab  
productivity**

**Our products help you to keep a close eye on food!**





Ready! Set! Go!

Your benefits



Ready to use.



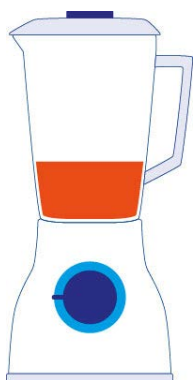


# CHROMABOND® QuEChERS

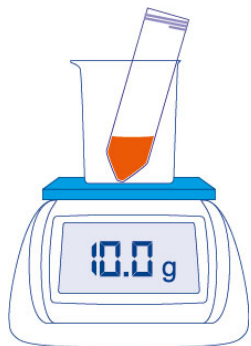
## QuEChERS procedure\*

It is simple and consists of two major steps:

### Step 1 – Extraction and salting-out



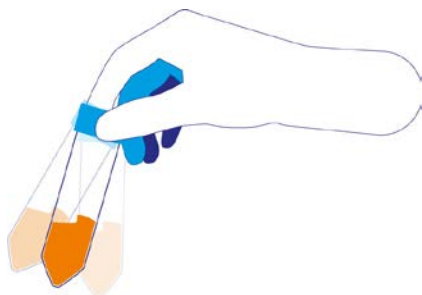
1. Sample is homogenized, e.g., with dry ice in a blender.



2. Weigh 10 g of the sample into a centrifuge tube.



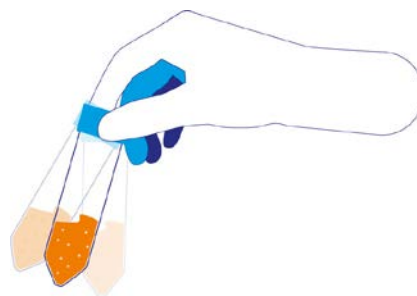
3. Add 10 mL of acetonitrile and internal standard.



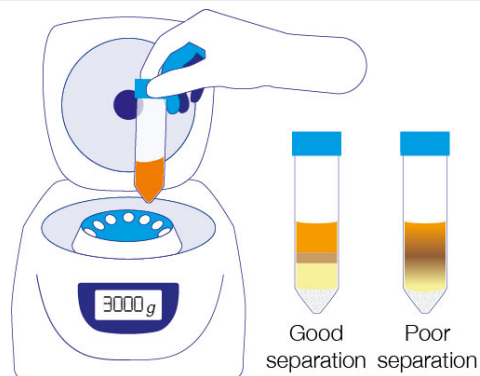
4. Shake vigorously for 1 min.



5. Add extraction mix to centrifuge tube.  
Optional: check pH and adjust pH to 5.0–5.5 with 5 mol/L aqueous NaOH.

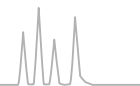


6. Shake vigorously for 1 min.

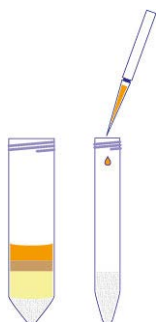


7. Centrifuge for 5 min at  $> 3000\text{ g}$ . For the determination of pesticides with acidic groups, the raw extract should be analyzed directly (preferably by LC/MS ESI neg.).

\*Procedure is described according to EN 15662:2008 method.



## Step 2 – Clean-up by dSPE



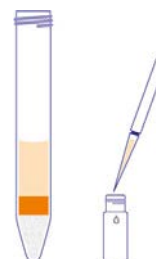
1. Transfer an aliquot of the supernatant to a centrifuge tube containing a clean-up mix. This dispersive solid phase extraction mix (dSPE) removes matrix components such as fats, sterols and pigments.



2. Shake for 30 s.



3. Centrifuge for 5 min at > 3000 g.



4. Transfer supernatant to vial, acidify with 5 % formic acid in acetonitrile (10 µL/mL extract) and analyze sample by LC/MS or GC/MS.

# CHROMABOND® QuEChERS

Select the right QuEChERS products for your sample!

For each of the two steps we provide the proper CHROMABOND® mixes. The contents are pre-weighed and mixed according to the standardized methods:

## Step 1 – Extraction and salting-out

| Method                                           | Sample weight | Solvent                                  | Content of mix                                                                                                                                   | Mix No. |
|--------------------------------------------------|---------------|------------------------------------------|--------------------------------------------------------------------------------------------------------------------------------------------------|---------|
| EN 15662:2008<br>citrate-buffered <sup>[2]</sup> | 10 g          | 10 mL acetonitrile                       | 4 g MgSO <sub>4</sub> , 1 g NaCl,<br>0.5 g Na <sub>2</sub> H citrate · 1.5 H <sub>2</sub> O,<br>1 g Na <sub>3</sub> citrate · 2 H <sub>2</sub> O | Mix I   |
| AOAC 2007.01<br>acetate-buffered <sup>[3]</sup>  | 15 g          | 15 mL 1 % acetic<br>acid in acetonitrile | 6 g MgSO <sub>4</sub> , 1.5 g Na acetate                                                                                                         | Mix II  |
| Original<br>non-buffered <sup>[1]</sup>          | 10 g          | 10 mL acetonitrile                       | 4 g MgSO <sub>4</sub> , 1 g NaCl                                                                                                                 | Mix XII |

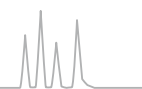
Samples with less than 80 % water content require the addition of sufficient cold water. The amount of water needed is listed in the table below. <sup>[2]</sup>

| Sample type                                   | Sample weight | Water added | Note                                          |
|-----------------------------------------------|---------------|-------------|-----------------------------------------------|
| Fruit and vegetables<br>> 80 % water content  | 10 g          | –           |                                               |
| Fruit and vegetables<br>25–80 % water content | 10 g          | x g         | x = 10 g minus water amount in sample         |
| Cereals                                       | 5 g           | 10 g        |                                               |
| Dried fruit                                   | 5 g           | 7.5 g       | Water can be added during<br>comminution step |
| Honey                                         | 5 g           | 10 g        |                                               |
| Spices                                        | 2 g           | 10 g        |                                               |

## References

- [1] M. Anastassiades, S. J. Lehotay, D. Stajnbaher, F. J. Schenck, J. AOAC Int. 86 (2003), 412–431.
- [2] EN 15662:2008 Foods of plant origin – Determination of pesticide residues using GC-MS and/or LC-MS/MS following acetonitrile extraction/partitioning and clean-up by dispersive SPE – QuEChERS method.
- [3] AOAC Official Method 2007.01, Pesticide Residues in Foods by Acetonitrile Extraction and Partitioning with Magnesium Sulfate.





## Step 2 – Clean-up by dSPE

|                                                                                    | Sample property                                                                                                                                                                                                                                            | Content of mix                                           | EN 15662 | AOAC 2007.01 |
|------------------------------------------------------------------------------------|------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|----------------------------------------------------------|----------|--------------|
|   | <b>Low fat content</b> <ul style="list-style-type: none"> <li>• apple</li> <li>• apricot</li> <li>• asparagus</li> <li>• broccoli</li> <li>• pear</li> <li>• pineapple</li> <li>• strawberry</li> </ul>                                                    | MgSO <sub>4</sub><br>Diamino (PSA)                       | Mix III  | Mix XX       |
|   | <b>Moderate content of chlorophyll and carotinoids</b> <ul style="list-style-type: none"> <li>• carrot</li> <li>• lettuce</li> <li>• coffee</li> <li>• tea</li> </ul>                                                                                      | MgSO <sub>4</sub><br>Diamino (PSA)<br>Carbon (GCB)       | Mix IV   | Mix XVII     |
|   | <b>Higher content of chlorophyll and carotinoids</b> <ul style="list-style-type: none"> <li>• pepper</li> <li>• spinach</li> <li>• blackberry</li> <li>• raspberry</li> </ul>                                                                              | MgSO <sub>4</sub><br>Diamino (PSA)<br>Carbon (GCB)       | Mix V    | –            |
|  | <b>Higher fat content</b> <ul style="list-style-type: none"> <li>• avocado</li> <li>• cereals</li> <li>• nuts</li> <li>• beef</li> <li>• chicken</li> <li>• pork</li> <li>• dairy products</li> <li>• soil</li> <li>• oils</li> <li>• baby food</li> </ul> | MgSO <sub>4</sub><br>Diamino (PSA)<br>C <sub>18</sub> ec | Mix VI   | Mix XIX      |

## Adsorbents and what they are used for

|                                                                       |                                                                          |
|-----------------------------------------------------------------------|--------------------------------------------------------------------------|
| MgSO <sub>4</sub>                                                     | Removes excess of water                                                  |
| NaCl                                                                  | For phase separation                                                     |
| CHROMABOND®<br>Diamino (PSA)<br>(Primary Secondary Amine)             | Removes organic and fatty acids, sugars and anthocyanin pigments         |
| CHROMABOND®<br>C <sub>18</sub> ec<br>(reversed phase modified silica) | Traps nonpolar compounds, e.g., lipids                                   |
| CHROMABOND®<br>Carbon (GCB)<br>(Graphitized Carbon Black)             | Removes pigments and sterols<br>Note: planar pesticides are also removed |

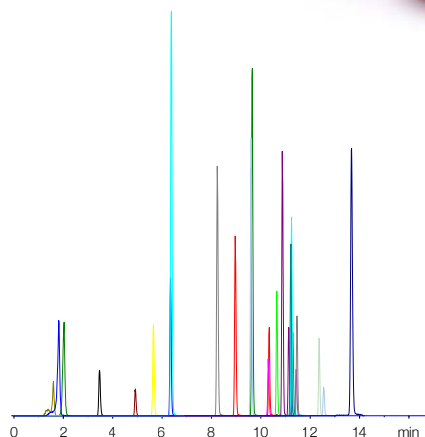


## Applications

### Apple

MN Appl. No. 306040/127410

Herein we describe the successful use of CHROMABOND® QuEChERS Mixes I and III for the analysis of 25 multiple pesticide class residues in apple\* which was purchased from a local grocery store. Subsequent LC/MS/MS analysis was performed with the new NUCLEODUR® C<sub>18</sub> Gravity-SB (conditions see next page).



#### Sample pretreatment:

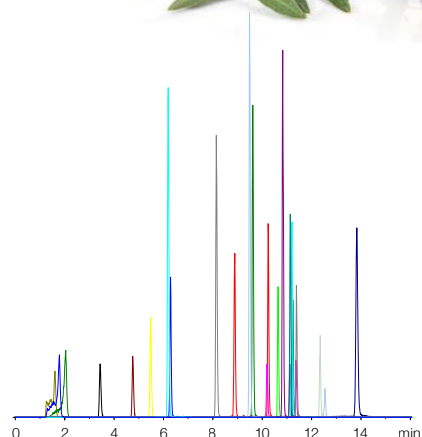
- Weigh 10 g homogenized sample into an empty 50 mL centrifuge tube (REF 730223)
- Add 9.2 mL acetonitrile, add 0.8 mL of pesticide standard solution (1 µg/mL each compound in acetonitrile)
- Shake the mixture for 1 min
- Shake sample mixture with Mix I and centrifuge (5 min at 4500 rpm, 5 °C)
- Transfer 6 mL supernatant to a 15 mL centrifuge tube, which is pre-filled with CHROMABOND® QuEChERS Mix III, shake for 1 minute and centrifuge again (5 min at 4500 rpm, 5 °C)
- Supernatant is analyzed by LC/MS/MS

| Pesticide     | % Recovery | % RSD (n=5) | Pesticide    | % Recovery | % RSD (n=5) |
|---------------|------------|-------------|--------------|------------|-------------|
| Acephate      | 85         | 2           | Omethoate    | 90         | 1           |
| Atrazine      | 92         | 3           | Phosalone    | 103        | 4           |
| Azoxystrobin  | 100        | 2           | Phosmet      | 92         | 3           |
| Carbaryl      | 102        | 2           | Tebuconazole | 90         | 4           |
| Chlorpyrifos  | 105        | 2           | Thiacloprid  | 83         | 6           |
| Coumaphos     | 100        | 3           | Propyzamide  | 101        | 2           |
| Cyprodinil    | 96         | 3           | Mevinphos    | 101        | 2           |
| Diazinon      | 102        | 2           | Fenazaquin   | 110        | 2           |
| Dimethoate    | 102        | 2           | Clofentezine | 94         | 3           |
| Ethion        | 155        | 4           | Tebufenozide | 102        | 5           |
| Ethoprophos   | 102        | 3           | Myclobutanil | 88         | 4           |
| Methamidophos | 88         | 3           | Imidacloprid | 89         | 6           |
| Methomyl      | 100        | 2           |              |            |             |

### Olive oil

MN Appl. No. 306030/127400

This application describes the use of CHROMABOND® QuEChERS Mixes for the analysis of 25 multiple pesticide class residues in olive oil\* (purchased from a local grocery store). The extraction and clean-up were successfully performed with Mixes II and XIX. Subsequent LC/MS/MS analysis was done with the new NUCLEODUR® C<sub>18</sub> Gravity-SB (conditions see next page).



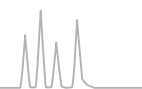
#### Sample pretreatment:

- Weigh 3 g sample into an empty 50 mL centrifuge tube (REF 730223)
- Add 7.0 mL water, add 9.2 mL acetonitrile (0.25 % formic acid) and 0.8 mL of pesticide standard solution (1 µg/mL each compound in acetonitrile)
- Shake the mixture for 1 min
- Shake sample mixture with Mix II and centrifuge (5 min at 4500 rpm, 5 °C)
- Transfer 6 mL supernatant to a 15 mL centrifuge tube, which is pre-filled with CHROMABOND® QuEChERS Mix XIX, shake for 1 minute and centrifuge again (5 min at 4500 rpm, 5 °C)
- Supernatant is analyzed by LC/MS/MS

| Pesticide     | % Recovery | % RSD (n=5) | Pesticide    | % Recovery | % RSD (n=5) |
|---------------|------------|-------------|--------------|------------|-------------|
| Acephate      | 85         | 4           | Omethoate    | 99         | 13          |
| Atrazine      | 82         | 3           | Phosalone    | 78         | 3           |
| Azoxystrobin  | 100        | 2           | Phosmet      | 96         | 3           |
| Carbaryl      | 97         | 1           | Tebuconazole | 86         | 3           |
| Chlorpyrifos  | 59         | 5           | Thiacloprid  | 94         | 2           |
| Coumaphos     | 81         | 1           | Propyzamide  | 84         | 1           |
| Cyprodinil    | 54         | 3           | Mevinphos    | 96         | 1           |
| Diazinon      | 77         | 3           | Fenazaquin   | 40         | 3           |
| Dimethoate    | 102        | 4           | Clofentezine | 66         | 3           |
| Ethion        | 71         | 3           | Tebufenozide | 88         | 3           |
| Ethoprophos   | 89         | 2           | Myclobutanil | 92         | 3           |
| Methamidophos | 103        | 2           | Imidacloprid | 96         | 2           |
| Methomyl      | 97         | 4           |              |            |             |

\* Food was spiked with pesticide standard.

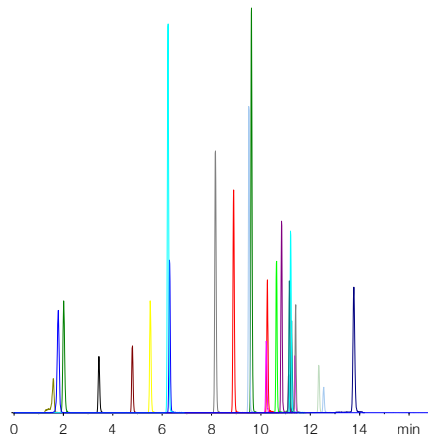




## Baby food

MN Appl. No. 306050/127420

This application describes the use of CHROMABOND® QuEChERS Mixes for the analysis of 25 multiple pesticide class residues in baby food\* which was purchased from a local grocery store. The extraction and clean-up were successfully performed with Mixes I and VI. Subsequent LC/MS/MS analysis was done with the new NUCLEODUR® C<sub>18</sub> Gravity-SB.



Sample pretreatment: analog to apple procedure  
Extraction: Mix I Clean-up: Mix VI

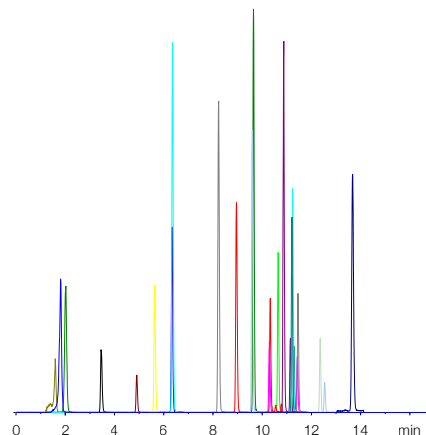
| Pesticide     | % Recovery | % RSD (n=5) | Pesticide    | % Recovery | % RSD (n=5) |
|---------------|------------|-------------|--------------|------------|-------------|
| Acephate      | 78         | 1           | Omethoate    | 84         | 2           |
| Atrazine      | 95         | 2           | Phosalone    | 75         | 2           |
| Azoxystrobin  | 100        | 2           | Phosmet      | 110        | 2           |
| Carbaryl      | 103        | 2           | Tebuconazole | 85         | 2           |
| Chlorpyrifos  | 58         | 4           | Thiacloprid  | 99         | 2           |
| Coumaphos     | 74         | 2           | Propyzamide  | 98         | 2           |
| Cyprodinil    | 62         | 1           | Mevinphos    | 101        | 2           |
| Diazinon      | 92         | 3           | Fenazaquin   | 42         | 4           |
| Dimethoate    | 97         | 2           | Clofentezine | 73         | 2           |
| Ethion        | 77         | 4           | Tebufenozide | 99         | 6           |
| Ethoprophos   | 99         | 1           | Myclobutanil | 100        | 2           |
| Methamidophos | 80         | 8           | Imidacloprid | 99         | 2           |
| Methomyl      | 97         | 1           |              |            |             |

| Pesticide     | Limit of quantification (LOQ) µg/kg |                  |                | Limit of detection (LOD) µg/kg |                  |                |
|---------------|-------------------------------------|------------------|----------------|--------------------------------|------------------|----------------|
|               | raw extract                         | after extraction | after clean-up | raw extract                    | after extraction | after clean-up |
| Acephate      | 0.26                                | 0.18             | 0.16           | 0.08                           | 0.05             | 0.05           |
| Atrazine      | 0.29                                | 0.21             | 0.18           | 0.09                           | 0.06             | 0.05           |
| Azoxystrobin  | 0.10                                | 0.08             | 0.04           | 0.03                           | 0.02             | 0.01           |
| Carbaryl      | 0.15                                | 0.13             | 0.10           | 0.04                           | 0.04             | 0.03           |
| Chlorpyrifos  | 2.45                                | 4.93             | 1.96           | 0.74                           | 1.48             | 0.59           |
| Coumaphos     | 0.87                                | 0.98             | 0.65           | 0.26                           | 0.29             | 0.19           |
| Cyprodinil    | 1.89                                | 3.08             | 1.33           | 0.57                           | 0.92             | 0.40           |
| Dimethoate    | 0.42                                | 0.16             | 0.16           | 0.13                           | 0.05             | 0.05           |
| Ethion        | 1.45                                | 2.75             | 1.17           | 0.44                           | 0.82             | 0.35           |
| Ethoprophos   | 0.88                                | 0.37             | 0.27           | 0.27                           | 0.11             | 0.08           |
| Methamidophos | 0.49                                | 0.33             | 0.37           | 0.15                           | 0.10             | 0.11           |
| Methomyl      | 0.49                                | 0.24             | 0.18           | 0.15                           | 0.07             | 0.05           |
| Omethoate     | 0.33                                | 0.22             | 0.21           | 0.10                           | 0.07             | 0.06           |
| Phosalone     | 0.27                                | 0.30             | 0.20           | 0.08                           | 0.09             | 0.06           |
| Phosmet       | 0.12                                | 0.09             | 0.06           | 0.03                           | 0.03             | 0.02           |
| Tebuconazole  | 0.29                                | 0.22             | 0.19           | 0.09                           | 0.07             | 0.06           |
| Thiacloprid   | 0.09                                | 0.06             | 0.04           | 0.03                           | 0.02             | 0.01           |
| Propyzamide   | 0.68                                | 0.41             | 0.36           | 0.20                           | 0.12             | 0.11           |
| Mevinphos     | 0.33                                | 0.20             | 0.19           | 0.10                           | 0.06             | 0.06           |
| Clofentezine  | 1.27                                | 0.79             | 0.54           | 0.38                           | 0.24             | 0.16           |
| Tebufenozide  | 0.30                                | 0.27             | 0.27           | 0.09                           | 0.08             | 0.08           |
| Myclobutanil  | 0.39                                | 0.23             | 0.20           | 0.12                           | 0.07             | 0.06           |
| Imidacloprid  | 0.23                                | 0.16             | 0.14           | 0.07                           | 0.05             | 0.04           |

## Spinach

MN Appl. No. 306060/127430

Herein we describe the successful use of CHROMABOND® QuEChERS Mixes I and V for the analysis of 25 multiple pesticide class residues in spinach\* which was purchased from a local grocery store. Subsequent LC/MS/MS analysis was performed with the new NUCLEODUR® C<sub>18</sub> Gravity-SB.



Sample pretreatment: analog to apple procedure  
Extraction: Mix I Clean-up: Mix V

| Pesticide     | % Recovery | % RSD (n=5) | Pesticide    | % Recovery | % RSD (n=5) |
|---------------|------------|-------------|--------------|------------|-------------|
| Acephate      | 100        | 4           | Omethoate    | 85         | 1           |
| Atrazine      | 94         | 2           | Phosalone    | 80         | 2           |
| Azoxystrobin  | 96         | 2           | Phosmet      | 87         | 3           |
| Carbaryl      | 96         | 2           | Tebuconazole | 95         | 3           |
| Chlorpyrifos  | 74         | 3           | Thiacloprid  | 98         | 2           |
| Coumaphos     | 73         | 2           | Propyzamide  | 95         | 3           |
| Cyprodinil    | 65         | 4           | Mevinphos    | 96         | 2           |
| Diazinon      | 96         | 2           | Fenazaquin   | 74         | 3           |
| Dimethoate    | 95         | 2           | Clofentezine | 62         | 2           |
| Ethion        | 106        | 3           | Tebufenozide | 96         | 3           |
| Ethoprophos   | 97         | 2           | Myclobutanil | 97         | 2           |
| Methamidophos | 87         | 3           | Imidacloprid | 102        | 2           |
| Methomyl      | 95         | 2           |              |            |             |

### Subsequent HPLC analysis

250 µL QuEChERS extract were filled up to 1 mL with Eluent A, mixture was filtered through syringe filter CHROMAFIL® Xtra PET-20/13 (REF 729222) before injection

### HPLC conditions

Column: 100x3 mm NUCLEODUR® C<sub>18</sub> Gravity-SB, 3 µm, (REF 760606.30)  
 Eluent: A) 5 mmol/L ammonium formate in water  
 B) 5 mmol/L ammonium formate in methanol  
 (solve ammonium formate in 5 mL water and fill up to 1 L with methanol)  
 Gradient: 25–90 % B in 10 min, 90 % B for 5 min  
 Flow rate: 0.5 mL/min  
 Temperature: 30 °C  
 Injection: 10 µL  
 Detection: MS

## Ordering information

### Extraction mixes

- 15 mL centrifuge tube with screw cap

| Method       | Mix No. | Content of mix                                                                                                                                   | Volume | Quantity | REF    |
|--------------|---------|--------------------------------------------------------------------------------------------------------------------------------------------------|--------|----------|--------|
| EN 15662     | Mix I   | 4 g MgSO <sub>4</sub> , 1 g NaCl,<br>0.5 g Na <sub>2</sub> H citrate · 1.5 H <sub>2</sub> O,<br>1 g Na <sub>3</sub> citrate · 2 H <sub>2</sub> O | 15 mL  | 50       | 730970 |
| AOAC 2007.01 | Mix II  | 6 g MgSO <sub>4</sub> , 1.5 g NaOAc                                                                                                              | 15 mL  | 50       | 730971 |
| Original     | Mix XII | 4 g MgSO <sub>4</sub> , 1 g NaCl                                                                                                                 | 15 mL  | 50       | 730648 |

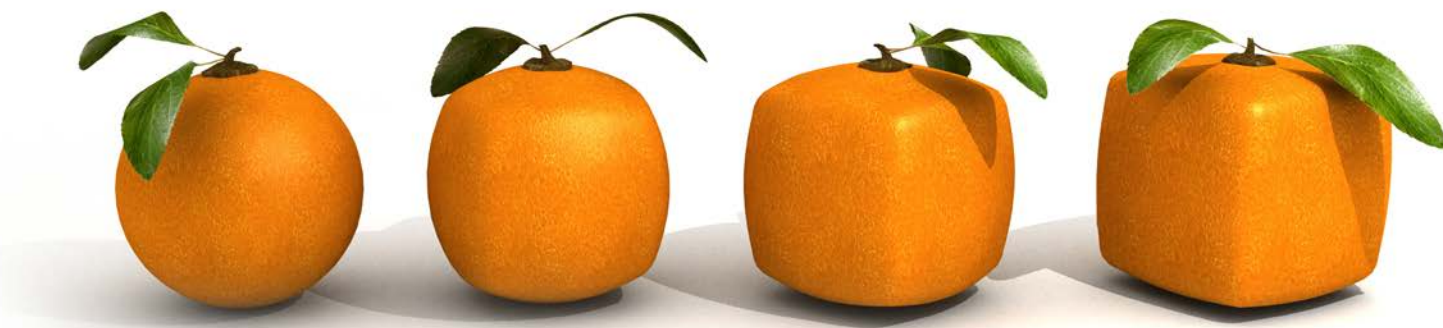
### Clean-up mixes

- 15 mL centrifuge tube with screw cap
- 2 mL centrifuge tube with cap

| Method       | Mix No.  | Content of mix                                                                                     | Volume | Quantity | REF      |
|--------------|----------|----------------------------------------------------------------------------------------------------|--------|----------|----------|
| EN 15662     | Mix III  | 0.90 g MgSO <sub>4</sub> ,<br>0.15 g CHROMABOND® Diamino                                           | 15 mL  | 50       | 730972   |
| EN 15662     | Mix IV   | 0.90 g MgSO <sub>4</sub> ,<br>0.15 g CHROMABOND® Diamino,<br>15 mg CHROMABOND® Carbon              | 15 mL  | 50       | 730973   |
| EN 15662     | Mix V    | 0.90 g MgSO <sub>4</sub> ,<br>0.15 g CHROMABOND® Diamino,<br>45 mg CHROMABOND® Carbon              | 15 mL  | 50       | 730975   |
| EN 15662     | Mix VI   | 0.90 g MgSO <sub>4</sub> ,<br>0.15 g CHROMABOND® Diamino,<br>150 mg CHROMABOND® C <sub>18</sub> ec | 15 mL  | 50       | 730974   |
| AOAC 2007.01 | Mix XVII | 0.15 g MgSO <sub>4</sub> ,<br>50 mg CHROMABOND® Diamino,<br>50 mg CHROMABOND® Carbon               | 2 mL   | 50       | 730996.2 |
| AOAC 2007.01 | Mix XIX  | 0.15 g MgSO <sub>4</sub> ,<br>50 mg CHROMABOND® Diamino,<br>50 mg CHROMABOND® C <sub>18</sub> ec   | 15 mL  | 50       | 730657   |
| AOAC 2007.01 | Mix XX   | 1.20 g MgSO <sub>4</sub> ,<br>0.40 g CHROMABOND® Diamino                                           | 15 mL  | 50       | 730658   |



Special service for special needs



Custom-made mixes are available on request.

## Extraction mix adsorbents:

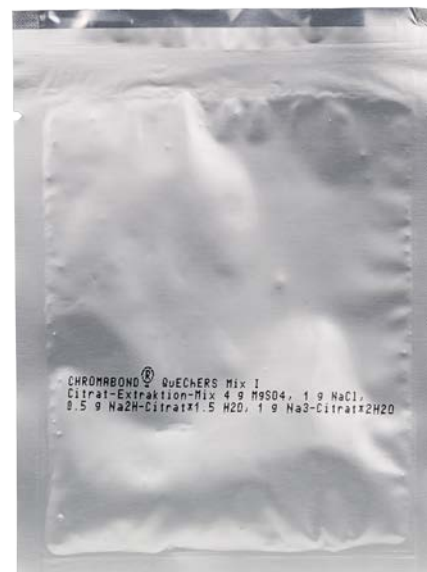
- $\text{MgSO}_4$
- $\text{NaCl}$
- $\text{Na}_2\text{H citrate} \cdot 1.5 \text{ H}_2\text{O}$
- $\text{Na}_3 \text{ citrate} \cdot 2 \text{ H}_2\text{O}$
- $\text{Na acetate}$
- other

## Clean-up mix adsorbents:

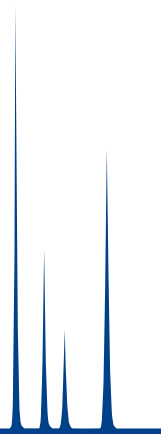
- $\text{MgSO}_4$
- CHROMABOND® Diamino (PSA)
- CHROMABOND®  $\text{C}_{18}$  ec
- CHROMABOND® Carbon (GCB)
- $\text{NaCl}$
- Alox N
- Florisil®
- $\text{CaCl}_2$
- $\text{NaHCO}_3$
- other

## Sizes (from left to right):

- 2 mL
- 15 mL
- 50 mL
- Aluminium bags



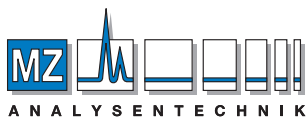
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