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

Distribútor pre SR:
AZ CHROM s.r.o., Robotnícka 10, 831 03 Bratislava
Tel. 0907 244526 azetchrom@hplc.sk, www.azetchrom.sk

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,[1] 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

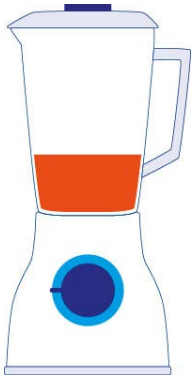
Ready to use.

CHROMABOND® QuEChERS

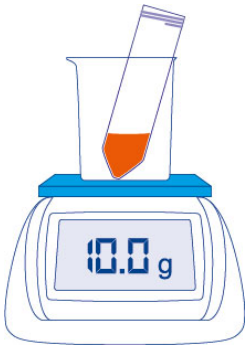
QuEChERS procedure*

It is simple and consists of two major steps:

Step 1 – Extraction and salting-out

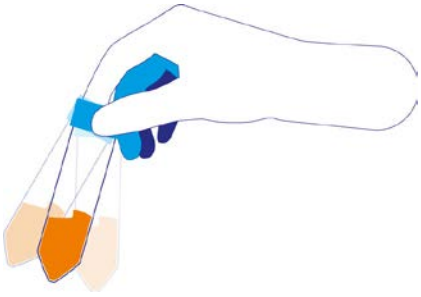


1. Sample is homogenized, e.g., with dry ice in a blender.

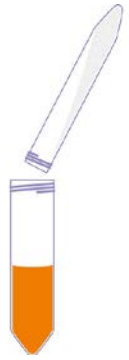


2. Weigh 10 g of the sample into a centrifuge tube.

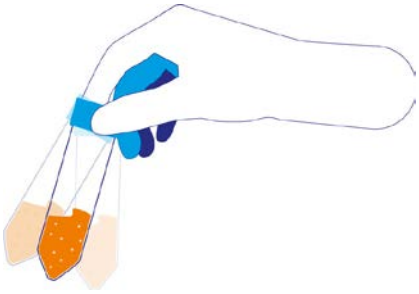
3. Add 10 mL of acetonitrile and internal standard.



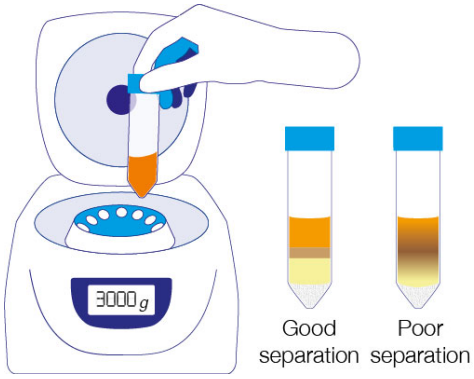
4. Shake vigorously for 1 min.



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



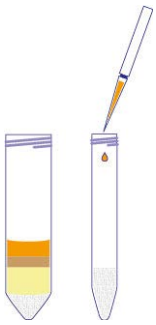
6. Shake vigorously for 1 min.



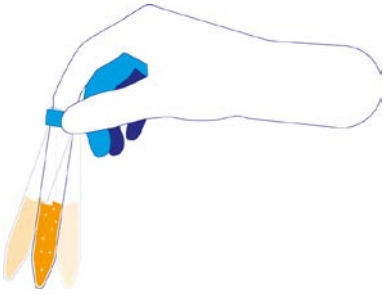
7. Centrifuge for 5 min at > 3000 *g*. For the determination of pesticides with acidic groups, the raw extract should be analyzed directly (preferably by LC/MS ESI neg.).

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

Step 2 – Clean-up by dSPE

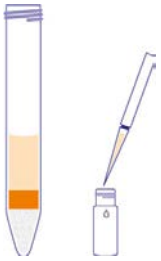


1. Transfer an aliquot of the supernatant to a centrifuge tube containing a clean-up mix. This dispersive solid phase extraction mix (dSPE) removes matrix components such as fats, sterols and pigments.



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 citrate-buffered [2]	10 g	10 mL acetonitrile	4 g $MgSO_4$, 1 g NaCl, 0.5 g Na_2H citrate · 1.5 H_2O, 1 g Na_3 citrate · 2 H_2O	Mix I
AOAC 2007.01 acetate-buffered [3]	15 g	15 mL 1 % acetic acid in acetonitrile	6 g $MgSO_4$, 1.5 g Na acetate	Mix II
Original non-buffered [1]	10 g	10 mL acetonitrile	4 g $MgSO_4$, 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. [2]

Sample type	Sample weight	Water added	Note
Fruit and vegetables > 80 % water content	10 g	–	
Fruit and vegetables 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 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.

CHROMABOND® QuEChERS

Step 2 – Clean-up by dSPE

	Sample property	Content of mix	EN 15662	AOAC 2007.01
	Low fat content · apple · apricot · asparagus · broccoli · pear · pineapple · strawberry	$MgSO_4$ Diamino (PSA)	Mix III	Mix XX
	Moderate content of chlorophyll and carotinoids · carrot · lettuce · coffee · tea	$MgSO_4$ Diamino (PSA) Carbon (GCB)	Mix IV	Mix XVII
	Higher content of chlorophyll and carotinoids · pepper · spinach · blackberry · raspberry	$MgSO_4$ Diamino (PSA) Carbon (GCB)	Mix V	–
	Higher fat content · avocado · cereals · nuts · beef · chicken · pork · dairy products · soil · oils · baby food	$MgSO_4$ Diamino (PSA) C_{18} ec	Mix VI	Mix XIX

Adsorbents and what they are used for

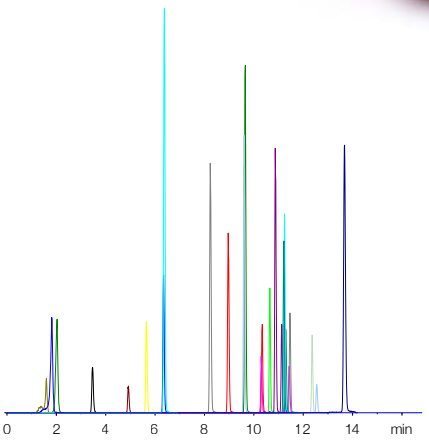
$MgSO_4$	Removes excess of water
NaCl	For phase separation
CHROMABOND® Diamino (PSA) (Primary Secondary Amine)	Removes organic and fatty acids, sugars and anthocyanin pigments
CHROMABOND® C_{18} ec (reversed phase modified silica)	Traps nonpolar compounds, e.g., lipids
CHROMABOND® Carbon (GCB) (Graphitized Carbon Black)	Removes pigments and sterols 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_{18} Gravity-SB (conditions see next page).



Sample pretreatment:

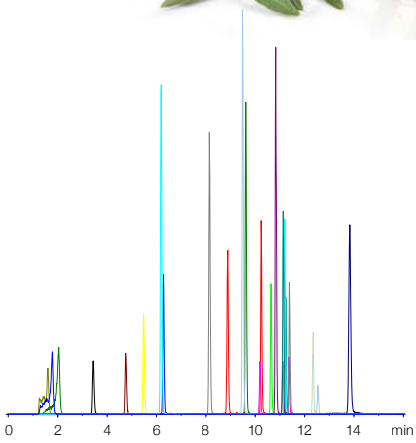
- Weigh 10 g homogenized sample into an empty 50 mL centrifuge tube (REF 730223)
- Add 9.2 mL acetonitrile, add 0.8 mL of pesticide standard solution (1 µg/mL each compound in acetonitrile)
- Shake the mixture for 1 min
- Shake sample mixture with Mix I and centrifuge (5 min at 4500 rpm, 5 °C)
- Transfer 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_{18} 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

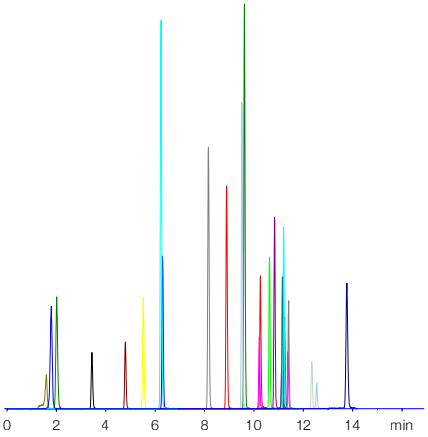
Pesticide	% Recovery	% RSD (n=5)	Pesticide	% Recovery	% RSD (n=5)
Acephate	85	4	Omethoate	99	13
Atrazine	82	3	Phosalone	78	3
Azoxystrobin	100	2	Phosmet	96	3
Carbaryl	97	1	Tebuconazole	86	3
Chlorpyrifos	59	5	Thiacloprid	94	2
Coumaphos	81	1	Propyzamide	84	1
Cyprodinil	54	3	Mevinphos	96	1
Diazinon	77	3	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_{18} Gravity-SB.



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

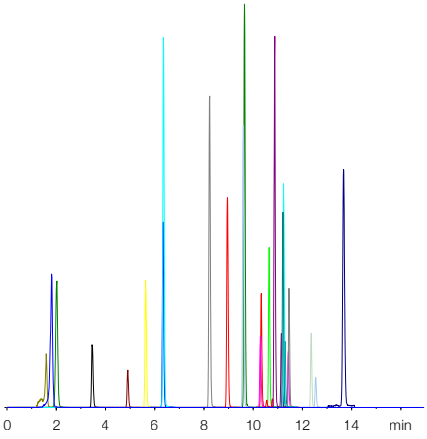
Pesticide	% Recovery	% RSD (n=5)	Pesticide	% Recovery	% RSD (n=5)
Acephate	78	1	Omethoate	84	2
Atrazine	95	2	Phosalone	75	2
Azoxystrobin	100	2	Phosmet	110	2
Carbaryl	103	2	Tebuconazole	85	2
Chlorpyrifos	58	4	Thiacloprid	99	2
Coumaphos	74	2	Propyzamide	98	2
Cyprodinil	62	1	Mevinphos	101	2
Diazinon	92	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 raw extract	after extraction	after clean-up	Limit of detection (LOD) µg/kg 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_{18} 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: 100 x 3 mm NUCLEODUR® C_{18} 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

CHROMABOND® QuEChERS

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 $MgSO_4$, 1 g NaCl, 0.5 g Na_2H citrate · 1.5 H_2O, 1 g Na_3 citrate · 2 H_2O	15 mL	50	730970
AOAC 2007.01	Mix II	6 g $MgSO_4$, 1.5 g NaOAc	15 mL	50	730971
Original	Mix XII	4 g $MgSO_4$, 1 g NaCl	15 mL	50	730648

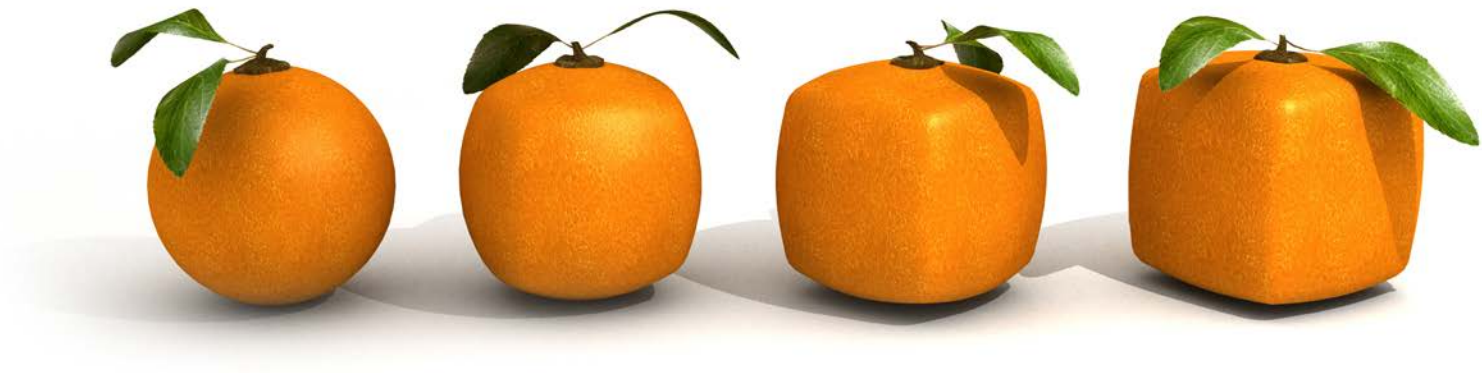
Clean-up mixes

- 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 $MgSO_4$, 0.15 g CHROMABOND® Diamino	15 mL	50	730972
EN 15662	Mix IV	0.90 g $MgSO_4$, 0.15 g CHROMABOND® Diamino, 15 mg CHROMABOND® Carbon	15 mL	50	730973
EN 15662	Mix V	0.90 g $MgSO_4$, 0.15 g CHROMABOND® Diamino, 45 mg CHROMABOND® Carbon	15 mL	50	730975
EN 15662	Mix VI	0.90 g $MgSO_4$, 0.15 g CHROMABOND® Diamino, 150 mg CHROMABOND® C_{18} ec	15 mL	50	730974
AOAC 2007.01	Mix XVII	0.15 g $MgSO_4$, 50 mg CHROMABOND® Diamino, 50 mg CHROMABOND® Carbon	2 mL	50	730996.2
AOAC 2007.01	Mix XIX	0.15 g $MgSO_4$, 50 mg CHROMABOND® Diamino, 50 mg CHROMABOND® C_{18} ec	15 mL	50	730657
AOAC 2007.01	Mix XX	1.20 g $MgSO_4$, 0.40 g CHROMABOND® Diamino	15 mL	50	730658

CHROMABOND® QuEChERS

Special service for special needs



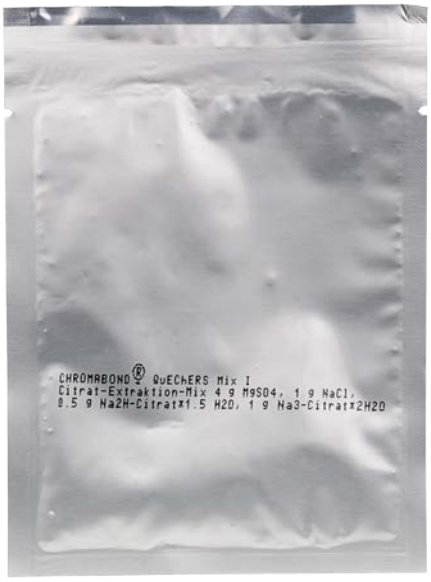
Custom-made mixes are available on request.

Extraction mix adsorbents:

- $MgSO_4$
- NaCl
- Na_2H citrate · 1.5 H_2O
- Na_3 citrate · 2 H_2O
- Na acetate
- other

Clean-up mix adsorbents:

- $MgSO_4$
- CHROMABOND® Diamino (PSA)
- CHROMABOND® C_{18} ec
- CHROMABOND® Carbon (GCB)
- NaCl
- Alox N
- Florisil®
- $CaCl_2$
- $NaHCO_3$
- other

Sizes (from left to right):

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