

# QuEChERS INSTRUCTIONS FOR USE

**BEKOLut®**  
QuEChERS KITS

The **QuEChERS** method, **Quick, Easy, Cheap, Effective, Rugged, and Safe**, was developed as a sample preparation method for the analysis of pesticides in fruits and vegetables with high water and low fat content, coupled with a clean-up method that removes e.g. sugars, lipids, organic acids, pigments, and excess water.

## MULTIRESIDUE QUECHERS PROCEDURES

The following procedures below are based on EN 15662:2018: Foods of plant origin – Multimethod for the determination of pesticide residues using GC- and LC-based analysis following acetonitrile extraction/partitioning and clean-up by dispersive SPE – Modular QuEChERS-method.

## THE TECHNIQUE INCLUDES TWO STEPS:

First, the homogenized samples are extracted by using a single phase consisting of acetonitrile and water. Then, extraction salts are added in order to initialize phase separation. After immediate centrifugation, the supernatant is cleaned-up using a dispersive solid-phase extraction (dSPE) technique for subsequent LC-MS/MS or GC-MS/MS.

## dSPE SAMPLE CLEAN-UP

Here, all QuEChERS clean-up methods based on dSPE can be applied in dependence of the respective matrix. BEKOLut dSPE kits are formulated in accordance with published methods and are listed in Table III. They are available either as 2 mL or 15 mL pre-filled centrifuge tubes for the respective organic extract volumes. Select tubes based on the method and sample type; general guidelines for different sample types are included in EN 15662:2018.

# QuEChERS EXTRACTION PROCEDURES (FIG. 1–7)

1. Homogenize the commodity to generate a uniform sample and weigh-in 10 g of homogenized product into a clean 50 mL tube (Fig. 1). Note that the 10 g sample size is for matrices >80% water\*. Add 10 mL of acetonitrile and an appropriate amount of an internal standard solution (Fig. 2) Intensively shake by hand for 1 minute (Fig. 3).

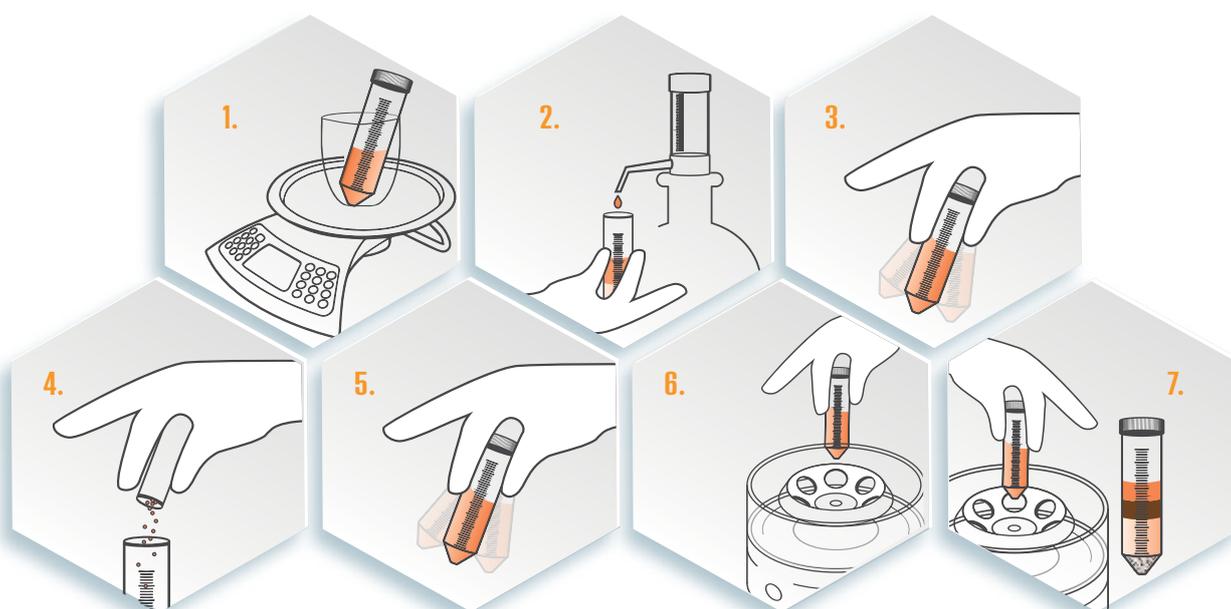
\* For samples with little or no water content, water must be added before adding extraction salts. For these samples, the sample weight must be reduced from 10 g to 5 g or less and 10 mL of water is added before extraction (Table I). For details, please refer to the EN 15662:2018 method.

2. **Extraction step:** Pour-in the entire contents of one tube BEKOLut Citrate Kit or Salt Kit-AC into the 50 mL tube (Fig. 4). For other extraction salt kits in accordance to the AOAC 2007.01 method, please refer to **Table II**. Immediately vortex or shake vigorously by hand for 1 minute (Fig. 5).

**TABLE I: WATER ADDITION**

Sample Pre-treatment			
Sample type	Weight	Water	Remarks
Fruits/Vegetables (Water content > 80 %)	10 g	-	
Fruits/Vegetables (Water content 30–80 %)	10 g	X g	X = 10 g - water content in 10 g sample
Cereals	5 g	10 g	
Dried fruits (Water content < 30 %)	5(00) g	5(50) g	Add water prior to homogenization. Weigh 13.5 g of the comminuted/homogenized sample
Honey	5 g	10 g	
Spices	2 g	10 g	

3. **Phase separation:** Centrifuge for 1 minute at >3.000 rpm to separate the solids (Fig. 6,7) from the organic phase.
4. Further proceed with dSPE sample clean-up or analyze raw extract directly without clean-up. This is recommended for pesticides with acidic groups like phenoxy herbicides.

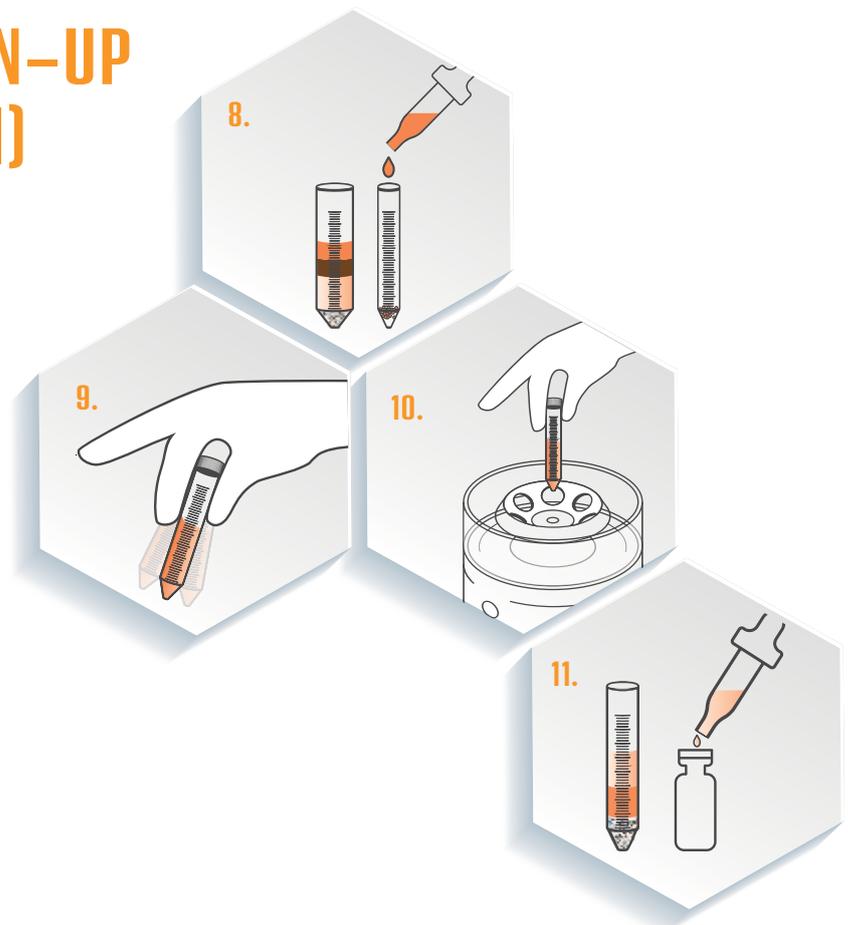


**TABLE II: EXTRACTION SALTS**

Kit	Article No. without 50 mL centrifuge tubes; 50 pcs./PK	Composition in grams							Method
		MgSO <sub>4</sub> anhydrous	Sodium chloride	tri-Sodium citrate dihydrate	Sodium hydrogen citrate sesquihydrate	NaHCO <sub>3</sub>	MgSO <sub>4</sub> ·H <sub>2</sub> O	NaAcetat	
Citrate-Kit-01	CK-01-050	4	1	1	0.5				EN 15662
Citrate-Kit-1/10	CK-1/10-050	0.4	0.1	0.1	0.05				EN 15662
Citrate-Kit-1/3	CK-1/3-050	1.335	0.335	0.335	0.017				EN 15662
Citrate-Kit-1/5	CK-1/5-050	0.8	0.2	0.2	0.1				EN 15662
Citrate-Kit-1/2	CK-1/2-050	2	0.5	0.5	0.25				EN 15662
Citrate-Kit-01-S	CK-01-050-S	4	1	1	0.5	5			EN 15662
SALT-Kit-AC	SK-AC-050	4	1						Original method (10 g samples)
SALT-Kit-AC-1/5	SK-AC-1/5-050	0.8	0.2						
SALT-Kit-AC2	SK-AC2-050	6						1.5	AOAC 2007.01
SALT-Kit-AC3	SK-AC3-050	6	1.5						Original method (15 g samples)
SALT-Kit-AC4	SK-AC4-050	4	0.5						Acrylamide
SALT-Kit-AC5	SK-AC5-050		1				4		

## dSPE SAMPLE CLEAN-UP (FIG. 8-10, TABLE III)

- Once tubes are selected, dSPE sample clean-up can be performed according to the procedure shown in the Fig. 8-10. Transfer an aliquot of the supernatant obtained after the extraction step to the pre-filled dSPE tube as shown in Fig. 8.
- Vortex or shake vigorously by hand for 30 seconds or 2 minutes (Fig. 9). Centrifuge for 5 minutes at >3.000 rpm to separate the solids (Fig. 10).
- Transfer sample extract after centrifugation to an autosampler vial and stabilize the analytes with 5% formic acid in acetonitrile (approx. 10 µL/mL extract). This stabilized solution is ready to use for pesticide multi-residue analyses using GC or LC methods (Fig. 11).



**TABLE III: dSPE KITS**

EN 15662:2018	MATRIX	AOAC 2007.01
<b>General samples (with low fat content)</b>		
<b>2 mL - PSA-KIT-01</b> 25 mg PSA, 150 mg MgSO <sub>4</sub> 100 pcs., Part no.: PK-01		<b>2 mL - PSA-KIT-01A</b> 50 mg PSA, 150 mg MgSO <sub>4</sub> 100 pcs., Part no.: PK-01A
<b>15 mL - PSA-KIT-02</b> 150 mg PSA, 900 mg MgSO <sub>4</sub> 50 pcs., Part no.: PK-02		<b>15 mL - PSA-KIT-02A</b> 400 mg PSA, 1200 mg MgSO <sub>4</sub> 50 pcs., Part no.: PK-02A
Color code		Color code
<b>Samples with high fat &amp; wax content</b>		
<b>2 mL - PSA-KIT-03</b> 25 mg PSA, 25 mg C18e, 150 mg MgSO <sub>4</sub> 100 pcs., Part no.: PK-03		<b>2 mL - PSA-KIT-03A</b> 50 mg PSA, 50 mg C18e, 150 mg MgSO <sub>4</sub> 100 pcs., Part no.: PK-03A
<b>15 mL - PSA-KIT-04</b> 150 mg PSA, 150 mg C18e, 900 mg MgSO <sub>4</sub> 50 pcs., Part no.: PK-04		<b>15 mL - PSA-KIT-04A</b> 400 mg PSA, 400 mg C18e, 1200 mg MgSO <sub>4</sub> 50 pcs., Part no.: PK-04A
Color code		Color code
<b>Pigmented samples (chlorophyll, carotenoids)</b>		
<b>2 mL - PSA-KIT-05</b> 25 mg PSA, 2.5 mg GCB, 150 mg MgSO <sub>4</sub> 100 pcs., Part no.: PK-05		<b>2 mL - PSA-KIT-05A</b> 50 mg PSA, 50 mg GCB, 150 mg MgSO <sub>4</sub> 100 pcs., Part no.: PK-05A
<b>15 mL - PSA-KIT-06</b> 150 mg PSA, 15 mg GCB, 900 mg MgSO <sub>4</sub> 50 pcs., Part no.: PK-06		<b>15 mL - PSA-KIT-06A</b> 400 mg PSA, 400 mg GCB, 1200 mg MgSO <sub>4</sub> 50 pcs., Part no.: PK-06A
Color code		Color code
<b>Highly pigmented samples (chlorophyll, carotenoids)</b>	<b>Pigmented samples with fats &amp; waxes</b>	
<b>2 mL - PSA-KIT-07</b> 25 mg PSA, 7.5 mg GCB, 150 mg MgSO <sub>4</sub> 100 pcs., Part no.: PK-07		<b>2 mL - PSA-KIT-07A</b> 50 mg PSA, 50 mg C18e, 50 mg GCB, 150 mg MgSO <sub>4</sub> 100 pcs., Part no.: PK-07A
<b>15 mL - PSA-KIT-08</b> 150 mg PSA, 45 mg GCB, 900 mg MgSO <sub>4</sub> 50 pcs., Part no.: PK-08		<b>15 mL - PSA-KIT-08A</b> 400 mg PSA, 400 mg C18e, 400 mg GCB, 1200 mg MgSO <sub>4</sub> 50 pcs., Part no.: PK-08A
Color code		Color code

### **FOR SAMPLES WITH CO-EXTRACTED FATS OR WAXES:**

The centrifuged samples are put in a refrigerator overnight. Cold samples are then re-centrifuged and fats or waxes are removed. If fat remains, the supernatant is cleaned-up with 25 mg PSA, 150 mg MgSO<sub>4</sub>, and 25 mg C18e per mL of extract (Fig. 8, BEKOLut PSA-KIT-03/04).

If no fat remains, clean-up with 25 mg PSA and 150 mg MgSO<sub>4</sub> per mL of extract is sufficient (Fig. 8, BEKOLut PSA-KIT-01/02).

### **FOR SAMPLES WITH INTENSELY COLORED EXTRACTS:**

Clean-up with 25 mg PSA, 150 mg MgSO<sub>4</sub>, and 7.5 mg graphitized carbon black (GCB) per mL of extract is recommended (Fig 8, BEKOLut PSA-KIT-07/08).

### **FOR SAMPLES WITH LESS INTENSELY COLORED EXTRACTS, OR HIGH CAROTENOID OR CHLOROPHYLL LEVELS:**

Clean-up with 25 mg PSA, 150 mg MgSO<sub>4</sub>, and 2.5 mg GCB per mL of extract is applicable (Fig. 8, BEKOLut PSA-KI-05/06).

### **FOR ALL OTHER SAMPLE EXTRACTS:**

Clean-up with 25 mg PSA and 150 mg MgSO<sub>4</sub> per mL of extract is sufficient (Fig. 8, BEKOLut PSA-KIT-01/02).

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Disclaimer: All instructions, illustrations and technical information are based on best knowledge and experience by BEKOlut in accordance with the EN 15662:2018 standard, but no guarantee can be given to the customer for successful application. This requires individual tests depending on the application.

Furthermore, BEKOlut accepts no guarantee or liability for damage or subsequent damage resulting from improper use.

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