

DisQuE Dispersive Sample Preparation

What is “QuEChERS”?

The DisQuE™ Dispersive Sample Preparation Products use the internationally recognized QuEChERS method for pesticide residue analysis of fruits, vegetables, and other agricultural commodities. QuEChERS products can be and has been successfully used for residue analysis in other food matrices such as seafoods, animal tissues and processed foods. The acronym QuEChERS stands for Quick, Easy, Cheap, Effective, Rugged, and Safe and gives a descriptive inference to the methods universal utility as a “catcher” for pesticide analysis.

You mention that QuEChERS is suitable for fruits and vegetables. Is the similar DisQuE extraction procedure suitable for dry commodities like grains and cereals?

The DisQuE procedure is adaptable for both wet and dry commodities. Water is a critical component of the extraction procedure; without water present in the homogenized sample you will encounter poor extraction efficiency. Ideally the sample should contain about 75% water, which is typical to that of a fruit or vegetable. For a dry commodity such as a grain, water should be added to the sample in a ratio of 2:1 (10 mL water:5g sample) to increase the water content during the homogenization and extraction steps.

Due to the recent acetonitrile shortage, I would like to reduce the amount of acetonitrile used for the extraction step. Are there any other solvents suitable for the DisQuE extraction?

Typically, 10 to 15 mL of acetonitrile is used per sample and is the solvent of choice for QuEChERS extractions. The properties of acetonitrile allow for high extraction efficiency of the pesticides of interest, while minimizing the amount of co-extracted matrix found in the sample. However, any solvent that can partition with water is useful as an extraction solvent. Alternatives include ethyl acetate, acetone, and dichloromethane.

I use an ultra-high speed centrifuge in my lab. What is the maximum speed that I can centrifuge my samples without breaking the DisQuE Extraction Tubes?

The DisQuE Extraction Tubes are rated as follows:

- 50 mL tubes: 10,000 rcf
- 15 mL tubes: 10,000 rcf
- 2 mL tubes: 17,000 rcf

Some of the more challenging pesticides that are analyzed in my laboratory are unstable at neutral to high pH. Should I be concerned that the DisQuE extraction procedure will degrade my pesticide and reduce my recovery?

This is a concern that is faced in many laboratories dealing with very low analyte concentrations. The DisQuE extraction procedure follows established acetate and citrate buffered extraction procedures that control and minimize the pH fluctuations in the sample extract. Reproducible buffering of the sample extract is critical to achieving consistent results.

I am currently using QuEChERS to extract acidic pesticides, such as 2,4-D, from my samples. Why am I experiencing low recovery?

The low recovery of acidic pesticide results from the ionic interaction between the Primary Secondary Amine (PSA) sorbent and the acidic pesticide. Sugars and organic acids are the most common interferences found in fruit and vegetable samples. The PSA sorbent is added to reduce these interferences. An unfortunate consequence is that the acidic pesticides are also removed. Many scientists just analyze the extract from Tube 1 without further clean-up from Tube 2. This can be effective in some instances; however, Waters does not recommend this approach. A dispersive solid phase extraction (SPE) tube that does not have PSA can be used for clean-up, such as the tube containing 150 mg MgSO_4 and 50 mg C_{18} (Part number 186008075), or using the SPE in a cartridge format to enrich and clean your sample extract from Tube 1. Refer to the DisQuE Dispersive Solid Phase Clean-Up Tube Selection Guide in Table 1.

DisQuE Clean-Up Tubes come in a variety of flavors that contain different blends of sorbents. Why would you choose one sorbent over another?

The DisQuE Clean-Up Tubes contain different blends of sorbents to accommodate the variety of sample types that you are likely to encounter. The original method specified PSA (Primary-Secondary Amine) as the only clean-up sorbent. The PSA sorbent is effective for removing sugars and organic acid matrix interferences from your sample. Samples that contain high fat and oil content may require the addition of C₁₈ (part number WAT035672) sorbents to provide the necessary clean-up. Graphitized Carbon Black (GCB) (part number [186004835](#)) is often added to remove pigments, such as chlorophyll, from the sample extract. A word of caution: GCB is a very non-polar SPE sorbent. In some cases you may experience losses of very non-polar or planar aromatic pesticide if used in excess. When using any sorbent modification, there is a trade-off between final extract cleanliness and analyte recovery. For convenience, Waters provide a wide variety of pre-weighed dispersive SPE tubes for specific sample matrices and applications for both AOAC method and CEN method (see Table 1).

Some of the pesticides that I work with are unstable and will degrade when they are exposed to increased temperature. How can I avoid this issue?

When dealing with thermally unstable pesticides, Waters recommends homogenizing your sample using a cryogenic blender, liquid nitrogen or dry ice to minimize analyte losses during the extraction steps.

When I add the homogenized sample to the DisQuE Extraction Tube with pre-weighed buffer salts, the magnesium sulfate “clumps” together with my sample and it is difficult to break apart. Should this be happening?

For some commodities, especially very “wet” samples, the water in the sample reacts with the anhydrous magnesium sulfate in the extraction tubes. The resulting clump is very hard to break and is undesirable. To overcome this issue, it is recommended that you add the acetonitrile or extraction solvents to the tube prior to adding your homogenized sample. The addition of solvent “wets” the magnesium sulfate and prevents “caking” without jeopardizing the extraction procedure.

In some cases, adding acetonitrile to the tube prior to adding sample may be undesirable. The buffer salts in pouch format should be used for sample extraction instead of the tube with pre-weighed salt mixture. For example, when the test sample is dry commodities such as cereals or dried fruits, it would require soaking the sample in water for a period of time to allow the residues released from the dry matrix. In this case, the homogenized dry sample would be first transferred into the 50 mL centrifuge tube, and then water is to be added to the tube. After the tube is shaken and soaked for sufficient time, the extraction solvent is added. Vigorously mix and shake the extraction tube for at least one minute. Empty the whole content of a pouch with buffer salts into the tube, and then again shake the tube vigorously for one minute. The sample tube is ready for centrifugation.

When I extract honey samples, I experience problems due to the high sugar content. What can I do?

Even though honey is a liquid, it only contains approximately 30% water. The remaining 70% is dissolved sugar. For the DisQuE extraction to be effective, the homogenized sample should contain approximately 75% water. Simple dilution of the honey overcomes many of the common problems associated with this type of extraction.

Can I inject the acetonitrile extract from Tube 2 directly onto my GC and LC instrumentation?

It is possible to inject the final acetonitrile extract directly onto your gas or liquid chromatograph. However, there may be some potential chromatographic problems. Acetonitrile is a very strong elution solvent for reversed-phase HPLC. If used as an injection solvent, you may experience chromatographic peak distortion, especially for early eluting, polar analytes. You can overcome this in two ways: Inject less sample volume or dilute your sample with water or buffer to weaken the solvent strength and eliminate the peak distortion. For GC applications, it is important to consider the detection method. For example, acetonitrile creates problems for GC-NPD (nitrogen-phosphorous detection) systems and should be avoided. An option is to exchange the solvent, through evaporation and reconstitution, for a solvent (e.g. toluene) that is more compatible with the GC system.

Can QuEChERS method be used for residue analysis other than pesticides?

Yes. QuEChERS extraction technique has been successfully used for other residues such as microtoxins and veterinary drugs in food stuffs. The degrees of success for the specific applications are mostly dependent upon the nature of the compounds. The applicable compounds are those that have similar properties like pesticides which have medium to high polarity and low to moderate water solubility. In the case of veterinary drugs, aminoglycosides are not appropriate because they mostly stay in the aqueous layer during the liquid-liquid partitioning step. However, the modified QuEChERS extraction protocol was demonstrated in the sample pretreatment for the multi-residue analysis of mycotoxins in rice. Then a special formulated sorbent mixture (part no. [186008080](#) in Table 1) was used for the dispersive Solid-Phase Extraction (SPE) clean-up step.

Table 1 Selection Guide for DisQuE Dispersive Solid Phase Clean-Up Tubes

Part Number	Description	Contents	Usage Guide
186004572	DisQuE 2 mL dSPE Tube -AOAC, white cap	150 mg MgSO ₄ and 50 mg PSA	AOAC method, small clean-up tube for fruits and vegetables in general, remove fatty acids, sugars and lipids
186004830	DisQuE 2 mL dSPE Tube -AOAC with C ₁₈ , blue cap	150 mg MgSO ₄ , 50 mg PSA and 50 mg C ₁₈	AOAC method, small clean-up tube for fruits and vegetables with fatty matrix, remove fatty acids, sugars, fats and waxes and other non-polar components
186004831	DisQuE 2 mL dSPE Tube -CEN, yellow cap	150 mg MgSO ₄ and 25 mg PSA	CEN method, small clean-up tube for fruits and vegetables in general, remove fatty acids, sugars and lipids
186004832	DisQuE 2 mL dSPE Tube -AOAC with C ₁₈ , green cap	150 mg MgSO ₄ , 25 mg PSA and 25 mg C ₁₈	CEN method, small clean-up tube for fruits and vegetables with fatty matrix, remove fatty acids, sugars, fats and waxes and other non-polar components
New 186008071	DisQuE 2 mL dSPE Tube with C ₁₈ and GCB, blue cap	150 mg MgSO ₄ , 25 mg PSA, 25 mg C ₁₈ and 7 mg GCB	Samples with high pigments matrix such as spices and tea, clean-up tube for LC-MS analysis
New 186008075	DisQuE 2 mL dSPE Tube with C ₁₈ , yellow cap	150 mg MgSO ₄ , 50 mg C ₁₈	Clean-up tube for acidic pesticides and non-polar components
New 186008076	DisQuE 2 mL dSPE Tube with GCB, blue cap	150 mg MgSO ₄ , 25 mg PSA and 2.5 mg GCB	CEN method, samples with some pigments matrix
New 186008081	DisQuE 2 mL dSPE Tube with C ₁₈ and Al-N, blue cap	150 mg MgSO ₄ , 50 mg PSA, 30 mg C ₁₈ and 30 mg Al-N	Small clean-up tube for multi-residue mycotoxins LC-MS/MS analysis for grains, for use with QuEChERS extraction procedure
186004833	DisQuE 15 mL dSPE Tube -CEN, blue cap	900 mg MgSO ₄ and 150 mg PSA	CEN method, large clean-up tube for fruits and vegetables in general, remove fatty acids, sugars and lipids
186004834	DisQuE 15 mL dSPE Tube -CEN with C ₁₈ , white cap	900 mg MgSO ₄ , 150 mg PSA and 150 mg C ₁₈	CEN method, large clean-up tube for fruits and vegetables with fatty matrix, remove fatty acids, sugars, fats and waxes and other non-polar components
New 186008072	DisQuE 15 mL dSPE Tube -AOAC, clear cap	1200 mg MgSO ₄ and 400 mg PSA	AOAC method, large clean-up tube for fruits and vegetables in general, remove fatty acids, sugars and lipids
New 186008073	DisQuE 15 mL dSPE Tube -AOAC with C ₁₈ , blue cap	1200 mg MgSO ₄ , 400 mg PSA and 400 mg C ₁₈	AOAC method, large clean-up tube for fruits and vegetables with fatty matrix, remove fatty acids, sugars, fats and waxes and other non-polar components

[FREQUENTLY ASKED QUESTIONS]

Part Number	Description	Contents	Usage Guide
186008074 New	DisQuE 15 mL dSPE Tube -AOAC with C ₁₈ and GCB, blue cap	1200 mg MgSO ₄ , 400 mg PSA, 400 mg C ₁₈ and 400 mg GCB	AOAC method, large clean-up tube for samples with high pigments matrix such as spices and tea, for GC-MS analysis, not suitable for planar compounds
186008077 New	DisQuE 15 mL dSPE Tube, clear cap	900 mg MgSO ₄ and 300 mg PSA	Clean-up tube for samples with fatty acids matrix
186008078 New	DisQuE 15 mL dSPE Tube with C ₁₈ , blue cap	900 mg MgSO ₄ , 300 mg PSA and 300 mg C ₁₈ , and 50 mg GCB	Clean-up tube for fatty produce and cereals
186008079 New	DisQuE 15 mL dSPE Tube with C ₁₈ and GCB, blue cap	900 mg MgSO ₄ , 450 mg PSA and 300 mg C ₁₈	Clean-up tube for samples with fatty and pigments matrix
186008080 New	DisQuE 15 mL dSPE Tube with C ₁₈ and Al-N, blue cap	750 mg MgSO ₄ , 250 mg PSA, 150 mg C ₁₈ and 150 mg Al-N	Large clean-up tube for multi-residue mycotoxins LC-MS/MS analysis for grains, for use with QuEChERS extraction procedure

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