An Executive Summary

A New Technology, Energized Dispersive Extraction, for Rapid, Simple, and Efficient Sample Preparation

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EDGE breaks the sample preparation bottleneck barrier

Introduction

Advances in the speed of modern instrumentation have made sample preparation the bottleneck in analytical laboratories. A new, rapid sample preparation technology called Energized Dispersive Guided Extraction (EDGE, from CEM Corporation) eliminates this problem and improves sample recoveries. EDGE's simple five-minute automated cycle time includes setup, extraction, filtration, cooling, collection, and system washing. This method is at least six times faster than other sample preparation techniques and uses less solvent. EDGE is applicable to many industries such as environmental, food, consumer products, and pharmaceuticals.

Novel Sample Preparation Technique

Shown in **Figure 1**, the compact, fully automated EDGE apparatus improves the extraction process for sample preparation in several ways. This widely applicable technology is capable of preparing 12 samples per hour. In addition, it requires only 30 mL of solvent, which is less than many other pressurized fluid extractors. Comparison of extraction times and solvent usage between the EDGE and other common techniques is shown in **Figure 2**.

EDGE's Q-Cup sample holder simplifies and combines the processes of pressurized fluid extraction and dispersive solid phase extraction in one assembly. This yields all the benefits of both techniques while providing much faster results. Regardless of sample size, matrix, or use of sorbents, extraction only takes five minutes. This includes the filtering, cooling, and washing processes.

The Q-Cup, constructed of ultra-thin aluminum for efficient heating, consists of only three parts and its assembly is easy. **Figure 3** illustrates its simple design. The wide mouth makes it simple to introduce sample and clean-up agents. The perforated bottom can be attached with a single turn, and the disposable Q-Discs are placed in the perforated bottom. The unique open-cell concept creates a dispersive effect that promotes rapid extraction and filtration.

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A schematic of the Q-Cup within a pressurized chamber is depicted in **Figure 4**. Once the Q-Cup is automatically loaded into the EDGE chamber by the autosampler, a pressure cap then creates a pressurized seal on the top of it. Thus, the top is sealed but the bottom is not. Solvent is first added through the bottom to fill the gap between the chamber and Q-Cup, thereby aiding heat transfer. Then, solvent is sprayed in from the top of the Q-Cup to fully wet the sample. As heating begins in the chamber, the solvent on the chamber walls is preferentially heated. This causes a thermal gradient, which, in turn, creates a pressure gradient. Pressure forces the solvent to move up through the bottom of the Q Cup, through the Q-Disc, and then disperse the sample. This creates the energized dispersive effect. The extraction is extremely efficient because the solvent is at an elevated temperature, which makes it disperse well with the sample. The entire extraction process is typically achieved within 60 to 90 seconds.

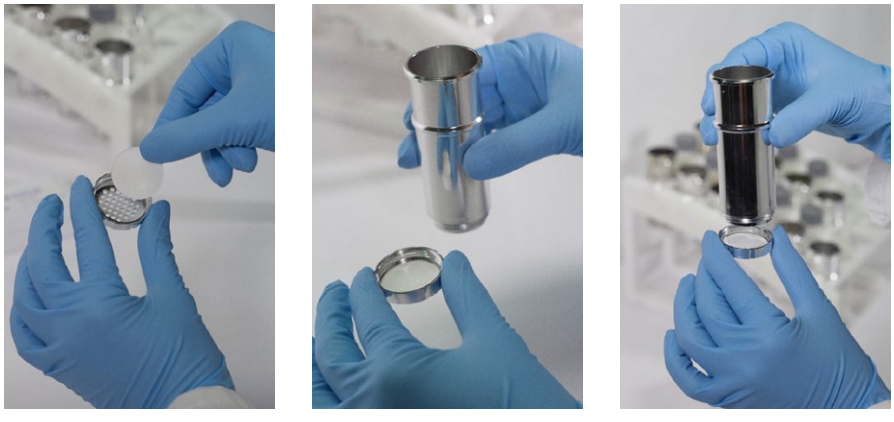
Figure 1: High throughput analysis of 48 samples/hr with four Energized Dispersive Guide Extraction (EDGE) systems.

Figure 2: Comparison of extraction times and solvent usage between the EDGE and other techniques.

Technique	Time (minutes)	Solvent Usage (mL)	Cost [1]
EDGE	5	30	$
QuEChERS	30	30	$
Pressurized Fluid Extraction	30	35	$$$
Soxhlet	360	150	$$
Automated Soxhlet	120	90	$$$
Ultrasonic	60	300	$$

[1] Includes instrument cost and running cost

Figure 3: The simple Q-Cup design.

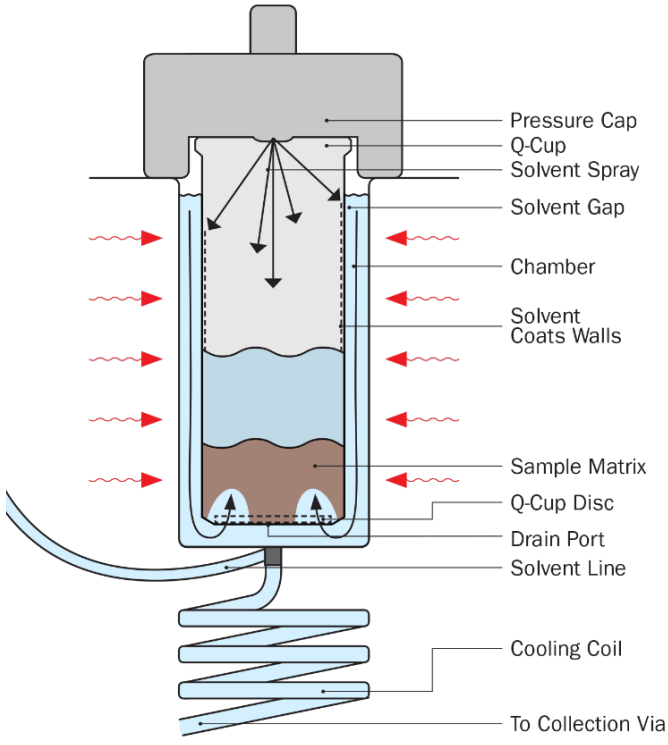


The extract is then drained through a cooling coil into a collection vessel. Next, a rinse occurs in order to wash away any residual solvent that may be left in the sample. Subsequently, the Q Cup is removed from the chamber and the chamber is washed. Cleaning typically uses a combination of two different solvents: a polar solvent and a non-polar solvent. This provides excellent cleaning of the entire wetted path length that the solvent will go through for the next test and eliminates carryover.

Sorbent and salt or other materials can be layered in the Q-Cup, which will allow the removal of interfering analytes. Thus, extraction of analytes and matrix clean-up can be accomplished in a one-step process. No additional steps are required before analysis.

Conveniently, the EDGE system uses removable racks that each hold 12 Q Cups and 12 collection vessels. This enables the analyst to place a rack in the system and prepare another rack while the first rack is running. Once the racks are in the system, the entire extraction and washing process is automated,

Figure 4: Schematic of the Q-Cup within a pressurized chamber.



- Q-Cup within a pressurized chamber
- Open Cell creates dispersive effect
- Promotes rapid extraction and filtration
- Provides filtered and cooled solvent solution ready for analysis or concentration

requiring no direct operator involvement.

Adding to the simplicity of the EDGE process, the instrument has pre-installed methods that offer effortless programming. One Touch™ methods are accessible right on the home screen, making the process straightforward and fast.

Figure 5: % Recovery data of spiked 15 g soil samples.

Compound	Sand	Loam	Clay
Pyridine	100	88	93
1,4-dichlorobenzene	88	88	96
2-methylphenol	84	95	115
3-methylphenol	90	102	104
hexachlorobutadiene	86	92	97
2,4,6-trichlorophenol	90	105	103
2,4,5-trichlorophenol	89	113	99
2,4-dinitrotoluene	90	102	99
hexachlorobenzene	86	86	81

Applications

The EDGE process can be useful for a myriad of analytes in environmental matrices, food, pesticides, polymers, pharmaceutical products, personal care products, and other media. For the extraction of semi-volatile organic compounds (SVOCs) in soils before GCMS analysis, EDGE adheres to US EPA 3545A, while Soxhlet extraction adheres to US EPA 3540C. These two techniques yield comparable results, yet there are significant differences in the requirements of the methods themselves. EDGE utilizes only 30 mL of a 50:50 mixture of hexane:acetone, whereas Soxhlet calls for 100 mL of the same solvent blend. While EDGE takes five minutes, Soxhlet takes 16–20 hours. Not only does the EDGE method save solvent, but it also saves considerable time and is a much simpler process.

Figure 5 shows the excellent recovery of several analytes in 15 g of spiked soil samples using EDGE. The table focusses on the BNA (base, neutrals, and acids) components of the SVOCs because they are generally the most difficult to extract. Similar recoveries were also achieved with 30 g of spiked soil samples and a relevant certified reference material. It

should be noted that the EDGE method described here heated the system to 100 °C, but the system is capable of heating to 200 °C. A temperature of 100 °C appears to be the "sweet spot" for SVOC extraction.

For the extraction of pesticides from food matrices before mass spectrometric analysis, EDGE has proven to be a simple alternative method to QuEChERS extraction. Although QuEChERS means Quick, Easy, Cheap, Effective, Rugged, and Safe, the EDGE extraction is actually quicker and much easier. QuEChERS is quite labor intensive, requiring multiple steps: add salts to the homogenized sample, shake it, and centrifuge it. Then decant, add sorbents, shake, centrifuge, and remove the supernatant for analysis. QuEChERS involves a solid partitioning extraction and then a dispersive solid-phase extraction clean-up.

In comparison, EDGE does the extraction and the clean-up in one step, which is fully automated. Although the actual extraction process is different than in QuEChERS, EDGE combines pressurized fluid extraction with dispersive solid phase extraction and takes just five minutes from start to finish. The only manual steps are simply putting sample in the Q-Cup and placing the Q-Cup in the EDGE. This process saves a considerable amount of time and effort.

Salts can be added to the Q-Cup to help dry wet samples such as strawberries. Sorbents added to the Q-Cup are very effective at cleaning up samples, even in difficult matrices. For example, lipids can be removed from hops, which are very waxy and onerous to clean-up using QuEChERS. Avocados are also challenging for QuEChERS due to their high fat content, whereas they work well in the EDGE system. **Figures 6 and 7** demonstrate the efficiency of the EDGE system in difficult matrices by showing the excellent recovery of several pesticides in rice, strawberries, avocado, and hops.

Figure 6: % Recovery data of spiked rice and strawberries.

Pesticide	Rice	Strawberry
Tokuthion	87	93
Guthion	90	90
Dichlorvos	88	120
Methyl parathion	95	107
Dursban	89	100
Ronnel	90	102
Disulfoton	92	92
Mocap	94	109

Figure 7: % Recovery data of spiked avocado and hops.

Pesticide	Avocado	Hops
Tokuthion	86	102
Guthion	85	102
Dichlorvos	116	98
Methyl parathion	107	98
Dursban	93	107
Ronnel	97	105
Disulfoton	89	101
Mocap	93	102

Conclusion

Energized Dispersive Guided Extraction (EDGE) with Q-Cup technology accelerates and simplifies sample preparation, thereby eliminating the bottleneck associated with high-performance analytical instruments. Its five-minute process includes the setup, extraction, filtration, cooling, collection, and system washing steps. Fully automated, it significantly reduces time spent on sample preparation and combines the processes of pressurized fluid extraction and dispersive solid phase extraction. Requiring only 30 mL of solvent, EDGE works well with difficult matrices that are challenging to other extraction methods. The technique has wide applicability to environmental, food, pesticide, polymer, pharmaceutical, and other types of samples.