„Shoot & Dilute GC“ – Captan und Folpet Analytik nach QuEChERS-Aufbereitung – schnell, robust und empfindlich

Um möglichst Empfindlichkeitsverluste bei der Pestizidanalyse nach QuEChERS-Aufbereitung zu vermeiden, halten sich viele Analytiker noch an die Splitlos-Injektion. Vor allem bei kritischen Pestiziden wie **Captan** und **Folpet** ist besondere Vorsicht geboten. Doch die zunächst sehr gute Empfindlichkeit nimmt nach einigen wenigen Injektionen bereits ab. Häufige Linerwechsel werden erforderlich.

⇒Unsere Lösung: die Verwendung eines blauen Precision-Liners und eine Split Injektion 1:10.

Die **Vorteile** liegen auf der Hand: schnellerer Probentransfer durch den Liner und damit weniger Zeit für Abbaureaktionen, nur 1/10 der Verschmutzung wie bei einer Splitlos-Injektion und damit längere Nutzung des Liners, der Säule und weniger Wartung Ihres GC Systems

Sie erreichen trotz der Split-Variante eine Nachweisempfindlichkeit von 1 ppb für Pestizide aus QuEChERS-Extrakten.

Lesen Sie mehr über das Ergebnis von jeweils 40 Splitless/Split-Injektionen zur Bestimmung kritischer Pestiziden aus Erdbeer-Extrakten in Resteks Poster.

Shoot and Dilute GC-ECD for Analysis of Problematic Pesticides

Introduction

⇒Sensitive pesticides like Captan, Folpet, DDT, and Deltamethrin degrade during hot splitless injection GC.

⇒This effect is magnified when analyzing food samples prepared using the QuEChERS procedure.

⇒Compound degradation leads to lower response factors and quantitative inaccuracies.

⇒To restore performance, more frequent maintenance on the splitless inlet and GC column are necessary.

⇒Maintenance includes splitless inlet liner and inlet seal changes, and GC column trimming.

⇒A way to keep the GC system up longer with more consistent response factors is to instead use **split injection.**

⇒**We call split injection GC—Shoot-and-Dilute“.**

Materials and Methods

⇒ EN QuEChERS extracts were prepared from organic strawberries.

⇒ The extracts were dSPE cleaned with 50mg per mL each, PSA and C18.

⇒ Formic acid was added to cleaned extracts (10 µL 5% per mL extract).

⇒ Extracts were spiked to 100 pg/µL with QuEChERS Performance Standard C.

⇒ The **splitless GC method conditions** were as follows:

Inlet: 250°C, 1µL splitless (1.4 min), **Sky 4mm single taper liner with quartz wool**.

Oven: 70°C (1.4 min), 15.2°C/min to 330°C (1.5 min).
Carrier: He, constant flow, 1.4 mL/min.
Column: Rxi-5ms, 15m x 0.25mm x 0.25µm.
ECD: 350°C, 20 Hz, nitrogen makeup + column flow 50 mL/min.

⇒ The **split GC method** used a **Sky 4 mm Precision split liner with quartz wool** at 250°C and split ratio 10.

⇒ The initial oven hold was 0.1 min for split; otherwise, the GC oven conditions were the same.

⇒ Ruggedness studies for **splitless** and split injections were 40 analyses each of strawberry extracts.

Results and Discussion

⇒The pesticides in QuEChERS Performance Standard C that could be detected with ECD are shown fully resolved from each other on the 15m x 0.25mm x 0.25µm Rxi-5ms GC column in **Figure 1**.

⇒Chromatograms at the start of the **splitless** ruggedness experiment (Splitless 1) and the end (Splitless 40) are shown in **Figure 2**, illustrating responses for difficult pesticides (Captan, Folpet, Deltamethrin) and a well-behaved pesticide like Bifenthrin. The red lines mark the 100% responses (Splitless 1 – first injection).

⇒During 40 **splitless injections** of strawberry extracts, the responses for Captan, Folpet, and Deltamethrin have declined significantly (**Figure 2**). Deltamethrin is degrading to its earlier eluting isomer.

⇒Chromatograms at the start of the **split** ruggedness experiment (Split 1) and the end (Split 40) are shown in **Figure 3**, again illustrating responses for difficult pesticides (Captan, Folpet, Deltamethrin) and a well-behaved pesticide like Bifenthrin. The red lines mark the 100% responses (Split 1 – first injection).

⇒During 40 **split injections** of strawberry extracts, the responses for Captan, Folpet, and Deltamethrin are holding up well (**Figure 3**). Deltamethrin is not degrading to its earlier eluting isomer.

⇒**Figure 4** shows Average Response Factor (Avg RF) and % RSD RF for the pesticides of interest. The initial RF's (not shown here) were proportionately higher for split injection of difficult pesticides, especially Deltamethrin, which suggests a better quantitative transfer to the GC column for split versus **splitless** injection.

⇒**Figure 4** demonstrates that the Average Response Factor RSD% values are better (lower) for the split injection approach for difficult pesticides. RSD%'s are high for the **splitless injection**, a function of the declining response factors as the splitless inlet liner gets dirty from the QuEChERS strawberry extracts.

⇒Calibration curves shown in **Figure 5** indicate that split injection GC-ECD can easily meet the low ppb requirements for analysis of these difficult pesticides.

⇒**Split injection works well because the higher flow through the hot inlet (in this case almost 15 mL/min) with low pesticide residence time mitigates degradation that can occur during slow transfer (splitless).**

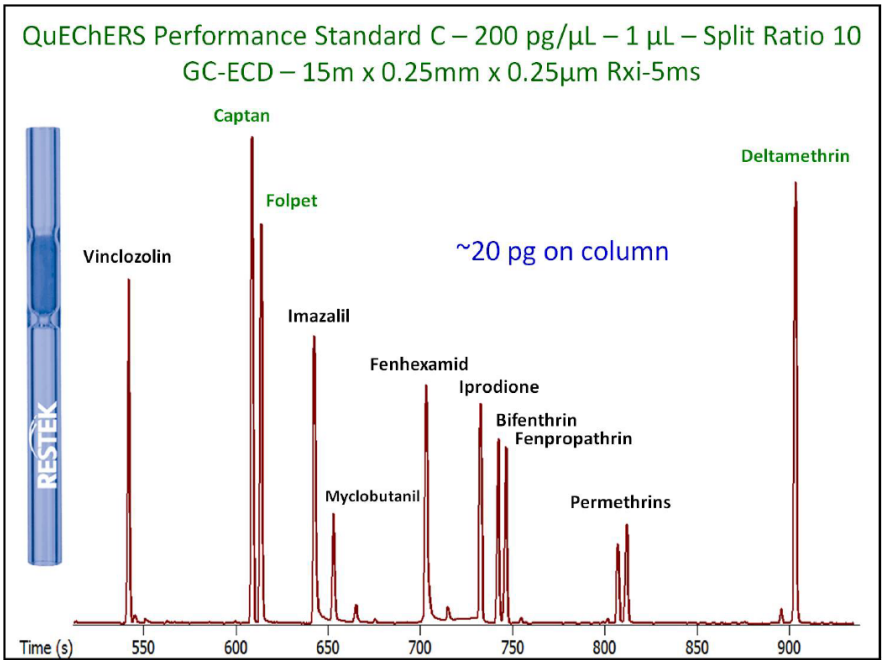

Figure1: QuEChERS Performance Standard C, including difficult pesticides like Captan, Folpet and Deltamethrin, analyzed under **split injection** GC-ECD conditions.

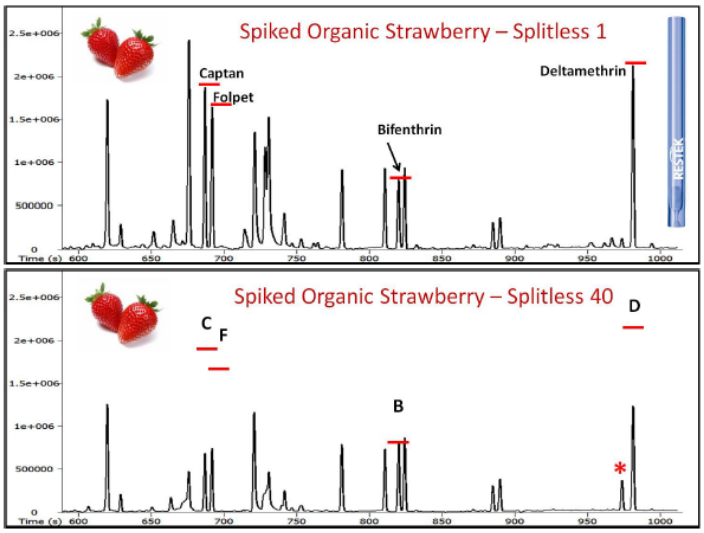

Figure 2: Captan, Folpet, and Deltamethrin show severely decreased response factors under splitless GC conditions during analysis of 40 QuEChERS strawberry extracts.

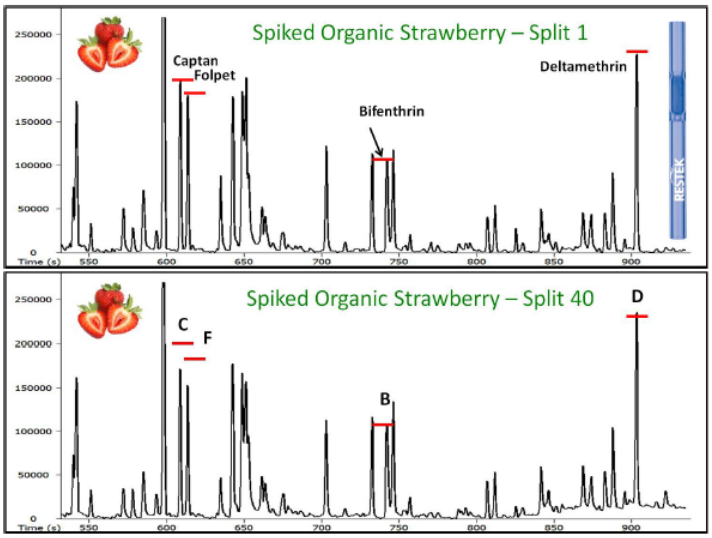

Figure 3: Captan, Folpet, and Deltamethrin show more stable response factors under split GC conditions during analysis of 40 QuEChERS strawberry extracts.

Pesticide	Splitless Avg RF	Split Avg RF		Splitless % RSD RF	Split % RSD RF
Captan	24800	4550		31	5.6
Folpet	25400	4200		26	5.7
Bifenthrin	23600	3490		1.9	3.0
Deltamethrin	43800	6620		15	2.4

Figure 4: The average response factor RSD% values for Captan, Folpet, and Deltamethrin are much better for split versus splitless injection over analysis of 40 strawberry extracts.

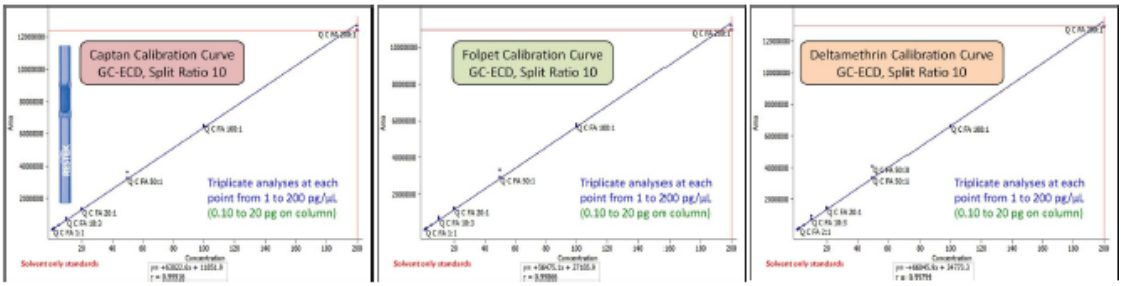

Figure 5: Split injection GC-ECD calibration curves for Captan, Folpet, and Deltamethrin are linear and easily reach the 1 ppb level for QuEChERS extracts of pesticides.

Conclusion

⇒ **Shoot-and-Dilute GC (split injection GC) with a Sky Precision split liner with quartz wool is recommended to prevent sensitive pesticide degradation that occurs during splitless injection. In addition to keeping the GC inlet up longer, the GC column lasts longer because less „dirt" is placed on it during split injection.**

Haben Sie Fragen zu dieser Problematik oder benötigen Sie weitere Informationen dazu? Kontaktieren Sie uns!

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