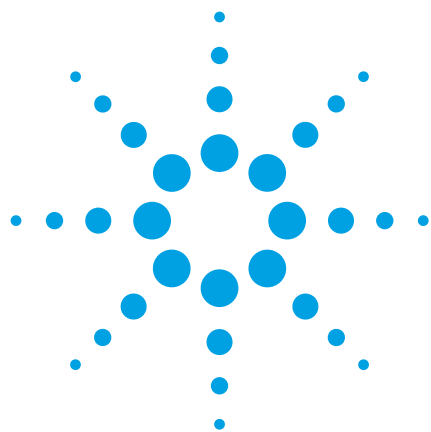




GC Separation of USP 467 Class 2 Solvents Using Agilent J&W FactorFour VF-624ms with EZ-Guard Column

Application Note

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Introduction

In United States, all drug substances, excipients and products are subject to the control of residual solvents under US Pharmacopeia method USP-NF General Chapter 467 Residual Solvents/Organic Volatile Impurities. The method sets out a testing procedure that covers 53 solvents, grouped into three classes of descending toxicity. Class 2 covers solvents of limited use. This application note demonstrates the value of the VF-624ms GC column with EZ-Guard in the analysis of Class 2 solvents according to the USP 467 method.

Guard columns are well known for protecting the analytical GC column, but can be a source for leakage through the coupling. As EZ-Guard is an integrated guard, it protects the analytical column and keeps the column leak-free.



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Materials and Methods

Column: VF-624ms, 30 m x 0.25 mm ID, df=1.4 μ m, + 10 m EZ-Guard (part number CP9029)

Samples: USP 467 Class 2 Mix A and Mix B

Sample Size: 0.2 μ L, splitter 200 mL/min

Sample Conc: 0.98-19.5 mg/mL (Mix A), 250-1450 μ g/mL (Mix B) in DMSO

Program Temp: 40 °C (20 min) to 240 °C (20 min), 10 °C/min

Carrier Gas: H₂

Pressure: 1 bar

Injector Temp: 275 °C, split

Detector Temp: 325 °C, FID

Results and Discussion

Figure 1 shows the separation of Class 2 Mix A solvents for USP 467. All the peaks have symmetrical shapes, indicating the inertness of the VF-624ms EZ-Guard.

Figure 2 shows the chromatogram of the Mix B solvents. Peak shapes are excellent for almost all components. Even the peak of pyridine is better than the industry standard.

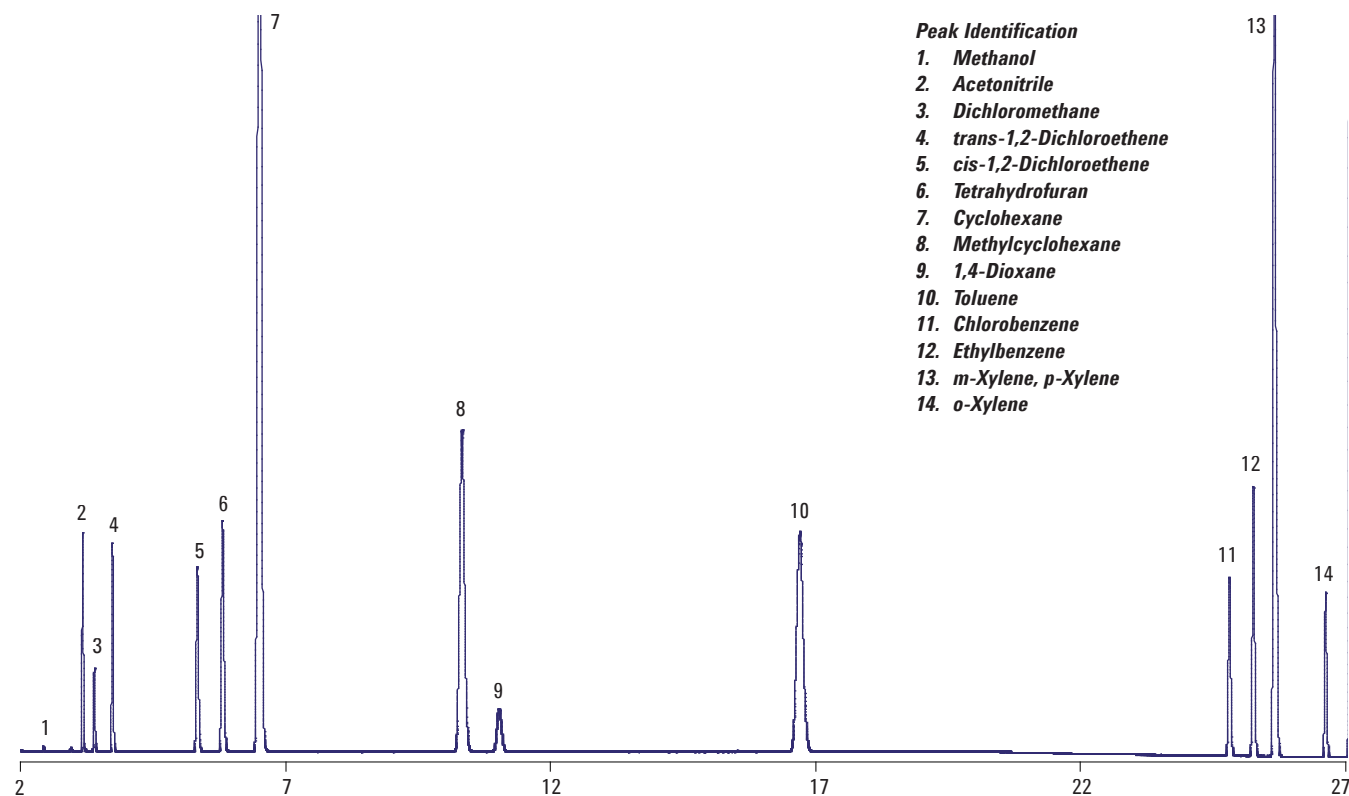


Figure 1. Separation of USP 467 Class 2 Mix A solvents on VF-624ms EZ-Guard

Peak Identification

1. Hexane
2. Nitromethane
3. Chloroform
4. 1,2-Dimethoxyethane
5. Trichloroethylene
6. Pyridine
7. 2-Hexanone
8. Solvent
9. Tetralin

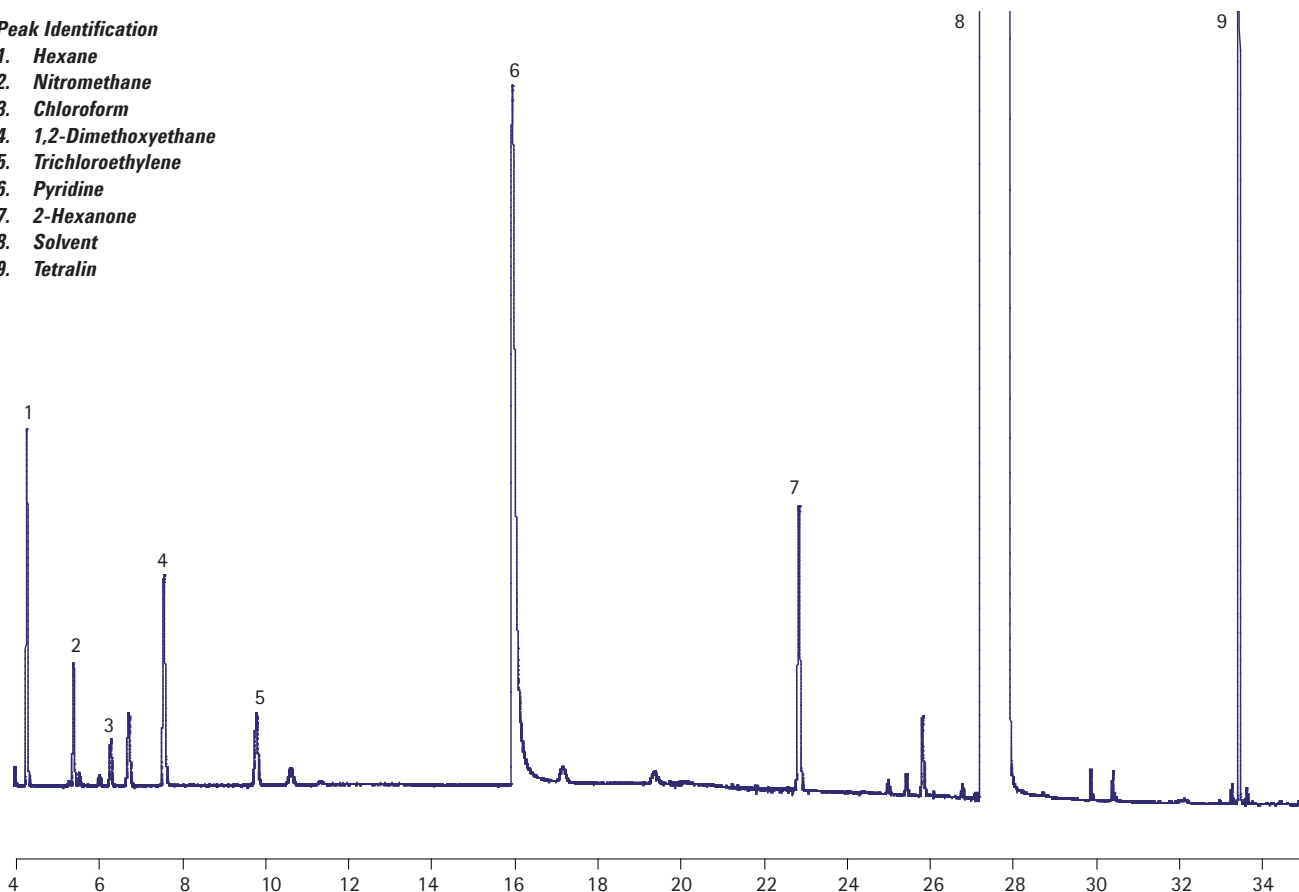


Figure 2. Separation of USP 467 Class 2 Mix B solvents on VF-624ms EZ-Guard

Conclusion

The VF-624ms successfully separated USP 467 Class 2 solvents with very good peak shapes and stable baselines. VF-624ms columns set a new standard for the analysis of volatile organic compounds. Their improved phase technology reduces bleed, thereby increasing signal to noise ratios. These columns are especially suited to analyzing solvents according to EPA Methods 524, 624 and 8260, as well as USP 467.

The EZ-Guard enhances the lifetime of the VF-624ms column when a complex matrix is being analyzed. When resolution or response in the chromatogram diminishes, a coil is removed from the EZ-Guard column so that column performance will improve.

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