APPLICATIONS

Determining the Effects of Harmful LC Solvent Vapors on GC Analysis

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Abstract

Losses of volatile mobile phase components to the atmosphere can result in poor chromatography. Using GC-MS, the changes are observed with and without a SecurityCAP™ product.

Introduction

The SecurityCAP mobile phase and solvent waste safety caps prevent dangerous vapors and gases from leaving LC solvent reservoirs and solvent waste containers. Over time, if not properly contained, these harmful solvents can have a negative impact on the health of employees and visitors in the lab. OSHA, ICH, and USP have placed allowable limits for harmful solvents in the workplace or in pharmaceutical ingredients that are listed in **Table 1**. Also, labs that do not have a dedicated area for both HPLC and GC instruments will suffer from cross contamination of HPLC mobile phase solvents in their GC analyses. False positive GC peaks can be detected on sensitive instruments, such as GC-Headspace or GC-MS due to escaped LC solvent vapors. To examine the presence of cross contamination by LC solvent vapors, an experiment was conducted using INFICON® Micro GC Fusion® Gas Analyzer. This experiment was designed to determine solvent contamination for low boiling solvents like methanol, acetonitrile, and dichloromethane in GC analysis with and without the use of SecurityCAP products. The SecurityCAP products not only protects HPLC/UHPLC results by preventing airborne particulates and contaminants from entering the solvent bottle, causing irreproducible results, bacterial growth, and ghost peaks, but it also prevents cross contamination of vapors. When laboratory safety and dependable results are a priority, SecurityCAP is an essential component to laboratory safety.

Experimental Conditions

The experimental design consisted of placing the portable INFICON Micro GC Fusion Gas Analyzer device inside a closed environment such as a 2' x 2' box to monitor the presence of solvent vapor. A pictorial representation of the experiment is shown in **Figure 1**. First, this experiment was performed in an empty box. A mini fan was kept in the corner of the box in order to circulate the vapors. Then an open, 1 L glass solvent bottle was kept inside the box. The box was then closed for 3-minutes to let the space equilibrate with the vapors, then a reading was taken at 3 minute intervals, in order for the vapors to fill in the space. Every reading requires box evacuation for 1 minute, 3 minutes of equilibration, and recording the solvent vapor using INFICON Micro GC-TCD. Box evacuation was performed for 1 minute by fanning out the vapors which prevented carry over from one experiment to another. Common reversed phase HPLC solvents such as methanol, acetonitrile, tetrahydrofuran, and normal phase HPLC solvents like dichloromethane and ethyl acetate were individually analyzed in a 1 L solvent bottle to monitor solvent contamination. The experiment was repeated for solvent bottles with and without SecurityCAP installed. All these experiments were performed to mimic real lab environment, where open solvent bottles can cause health hazard and false positive results.

GC-MS/MS Conditions

Column: 100 % PDMS
Dimensions: 20 m x 0.15 mm x 0.15 µm
Temperature Program: 60 °C hold for 65 °C/sec, 160 °C @ 0.8 °C/sec and hold for 10 sec at 160 °C
Column Pressure: 35 psi
Injection Time: 30 sec
Sampling Time: 15 sec (through a tube/straw)
Injection Temperature: 90 °C
Sample Inlet Heater Temperature: 50 °C
Detector: TCD

Table 1.
Allowable Limits for Harmful Solvents

Solvent	OSHA Permitted Exposure Limit (ppm)	Boiling Point (°C)	ICH/USP Residual Solvent Limits (ppm)
Acetonitrile	40	82	410
Methanol	200	64.7	3000
Tetrahydrofuran	200	66	720
Dichloromethane	25	39.6	600
Ethyl Acetate	500	77.1	5000

Figure 1.
Set-up for Open Mobile Phase Bottle and SecurityCAP™ Installed Bottle

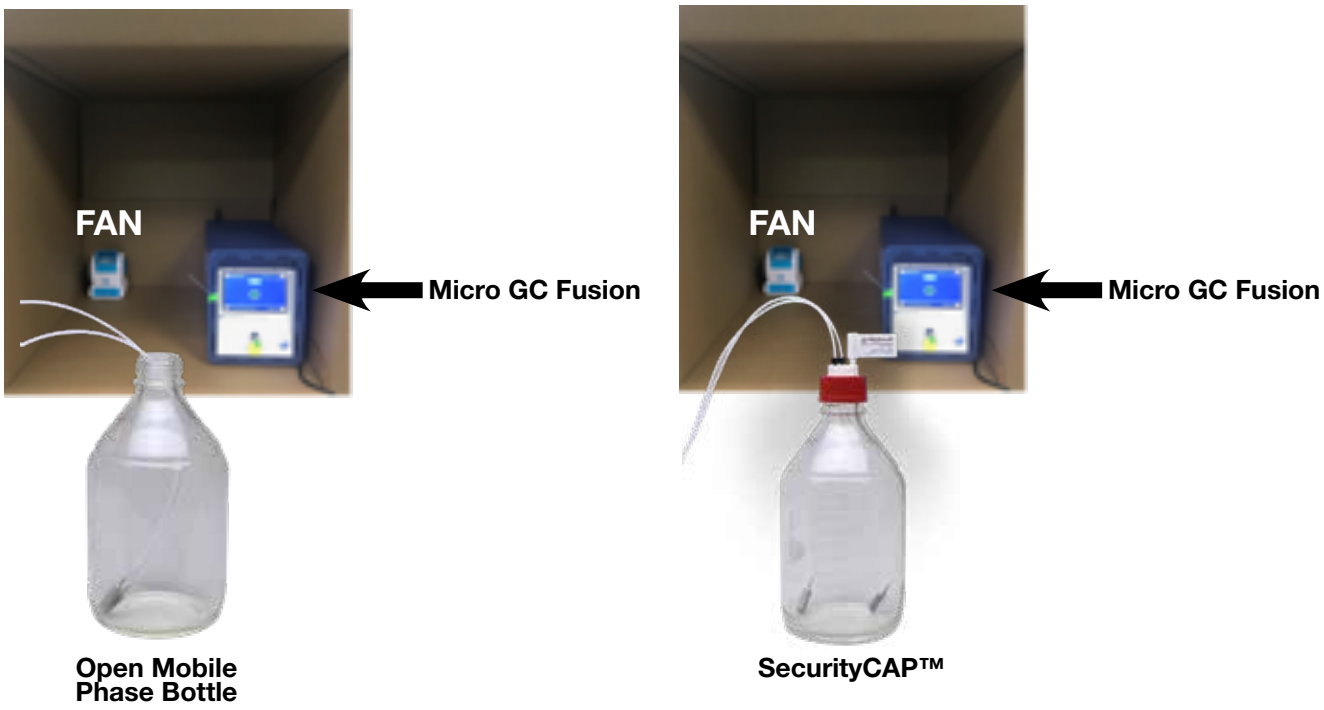


Figure 2.
Empty Box

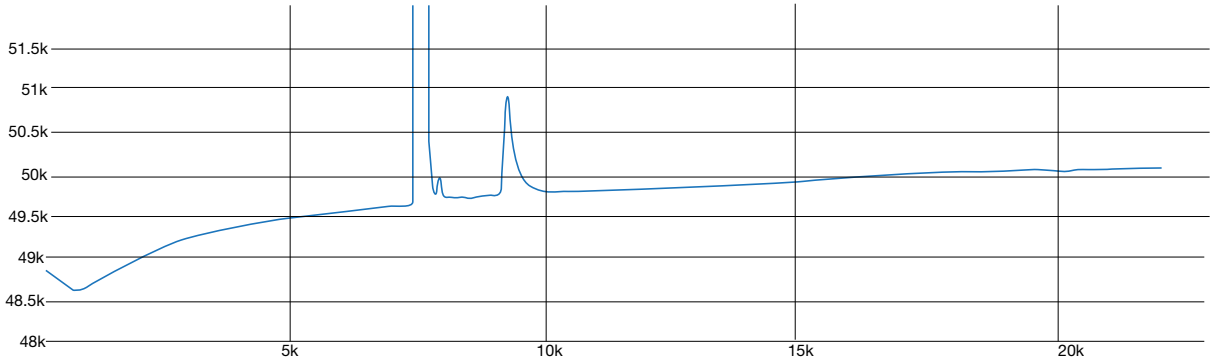


Figure 3.
Acetonitrile with Open Mobile Phase Bottle

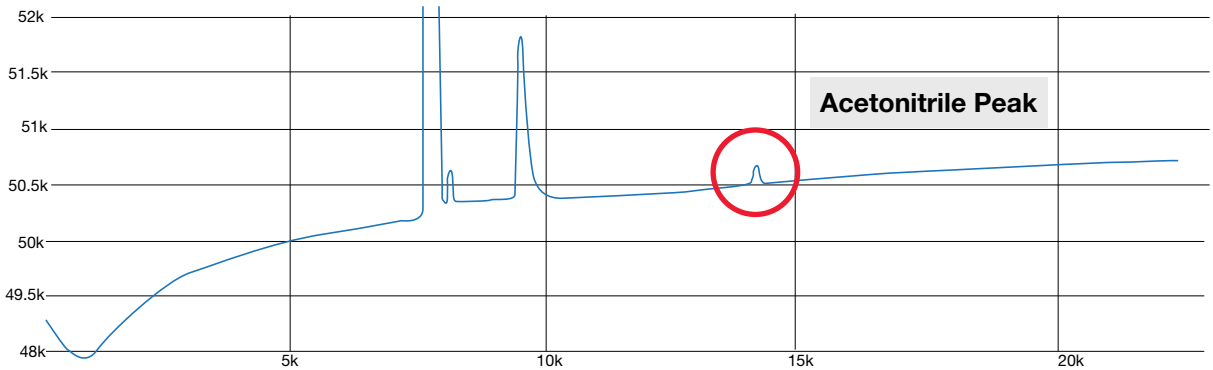


Figure 4.
Acetonitrile with SecurityCAP™ Installed

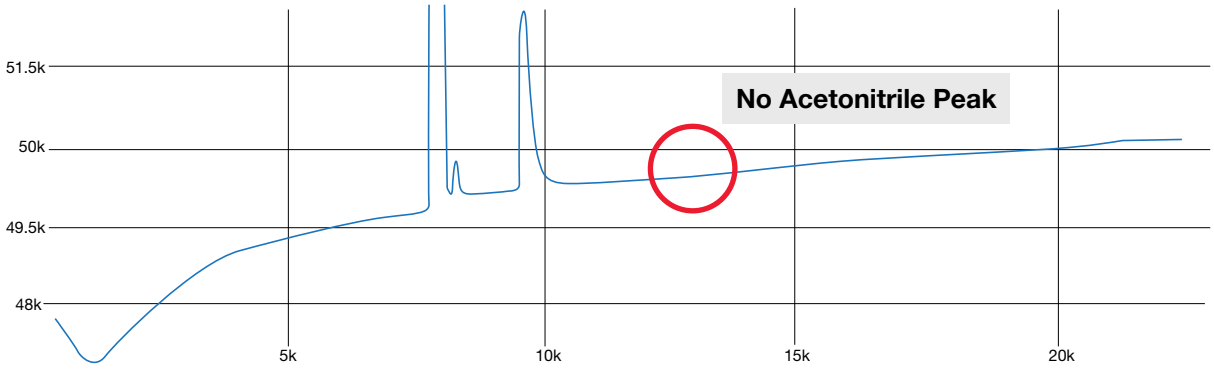


Figure 5.
Methanol with Open Mobile Phase Bottle

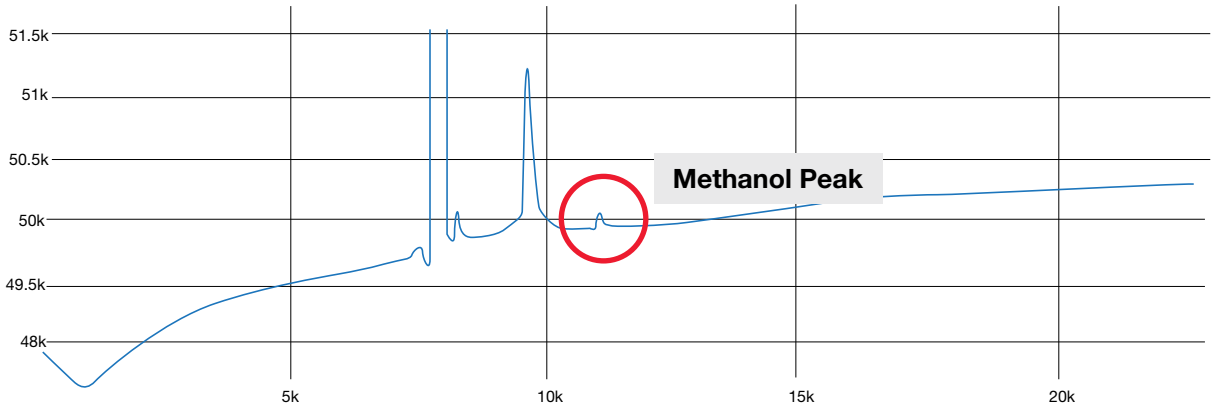


Figure 6.
Methanol with SecurityCAP Installed

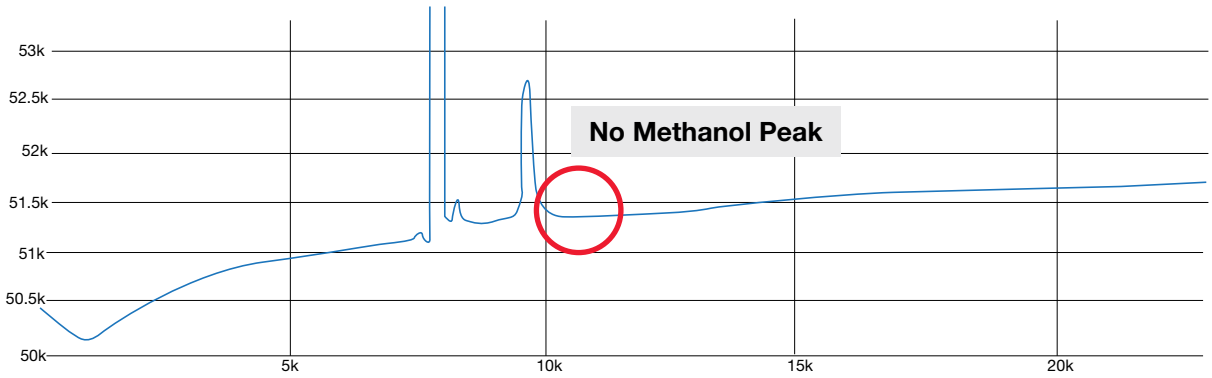


Figure 7.
Tetrahydrofuran with Open Mobile Phase Bottle

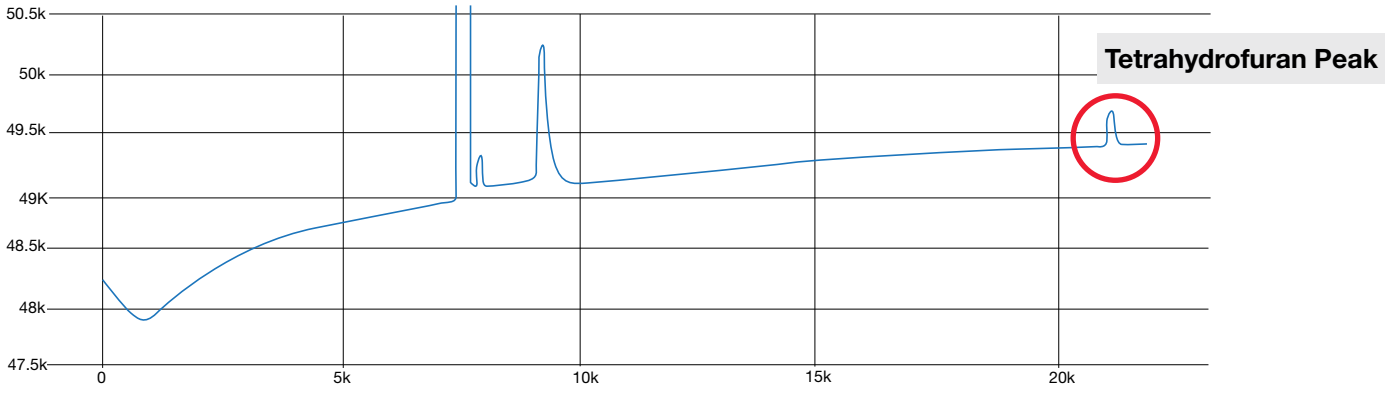


Figure 8.
Tetrahydrofuran with SecurityCAP™ Installed

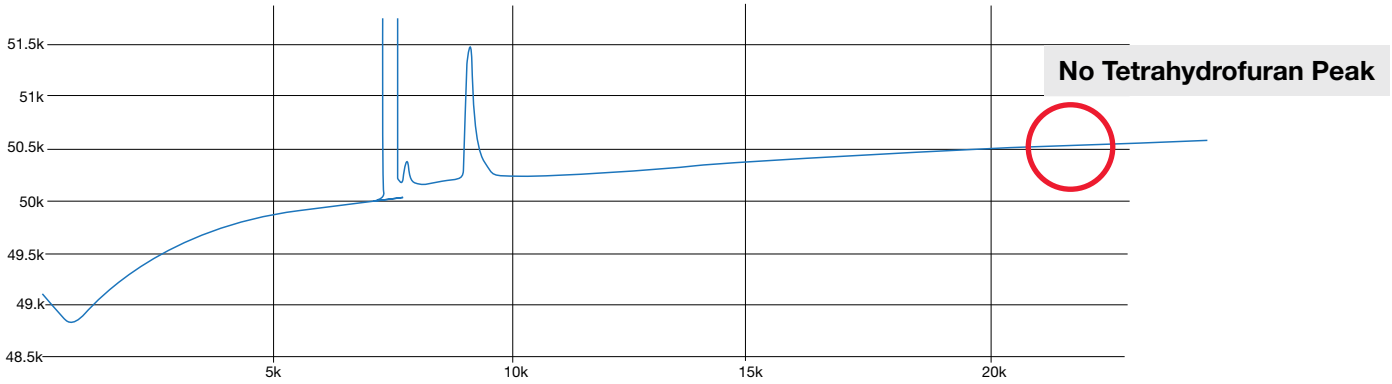


Figure 9.
Dichloromethane (DCM) with Open Mobile Phase Bottle

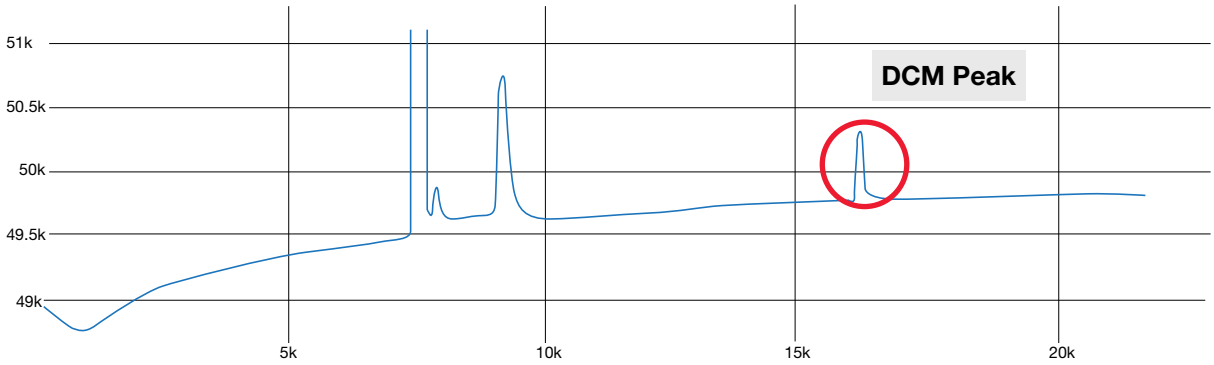


Figure 10.
Dichloromethane (DCM) with SecurityCAP Installed

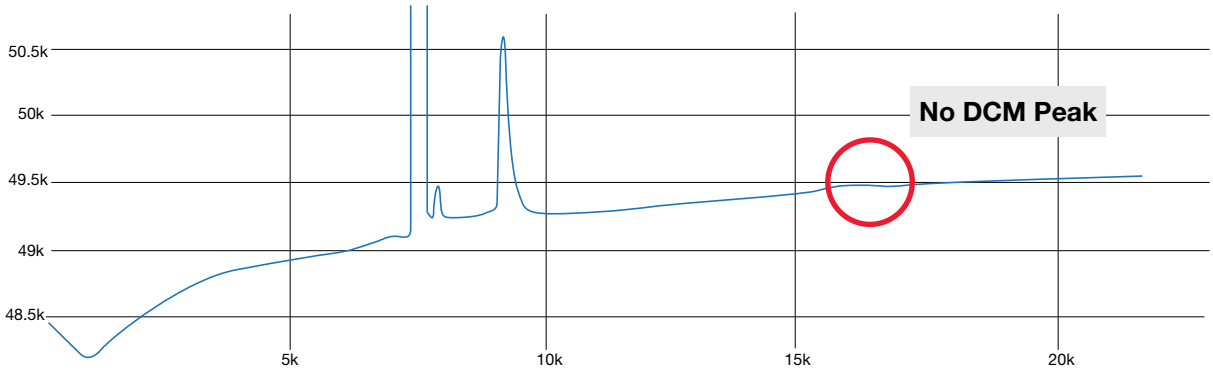


Figure 11.
Standard Test for Ethyl Acetate

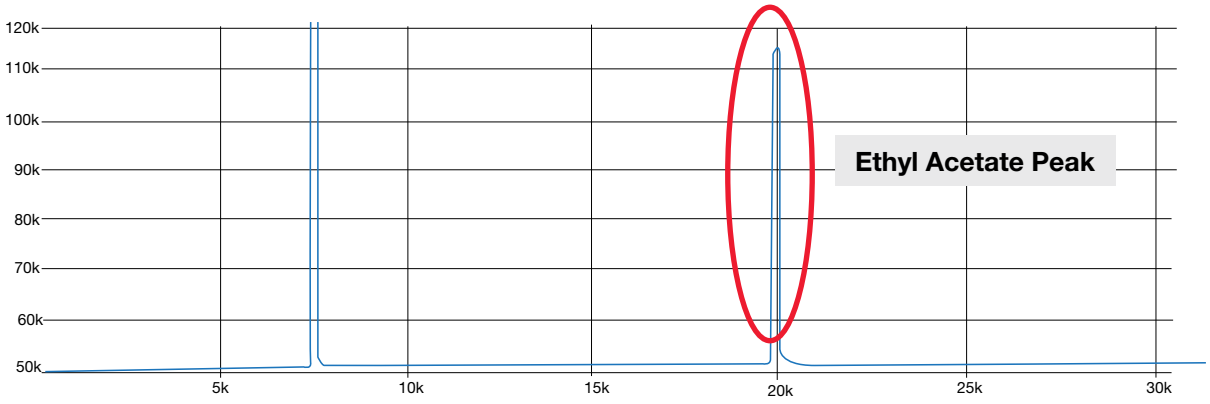


Figure 12.
Ethyl Acetate with Open Mobile Phase Bottle

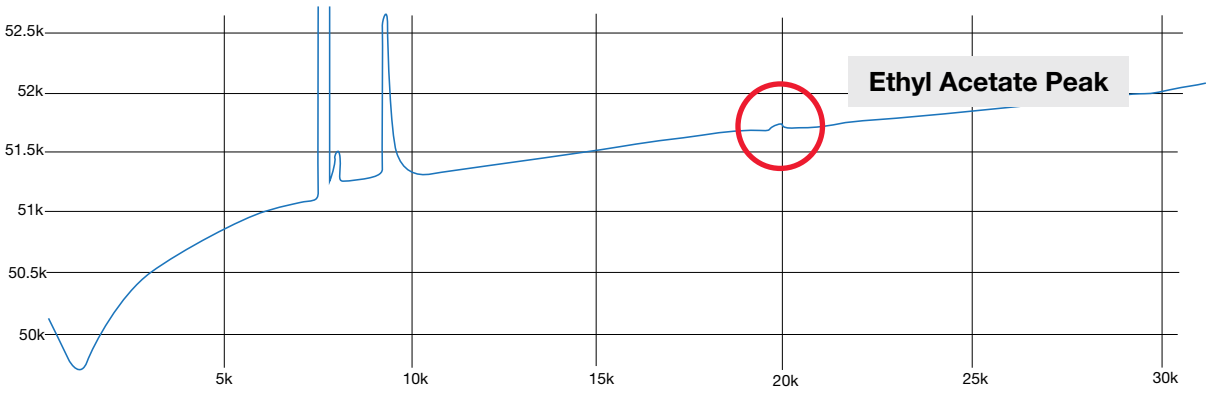
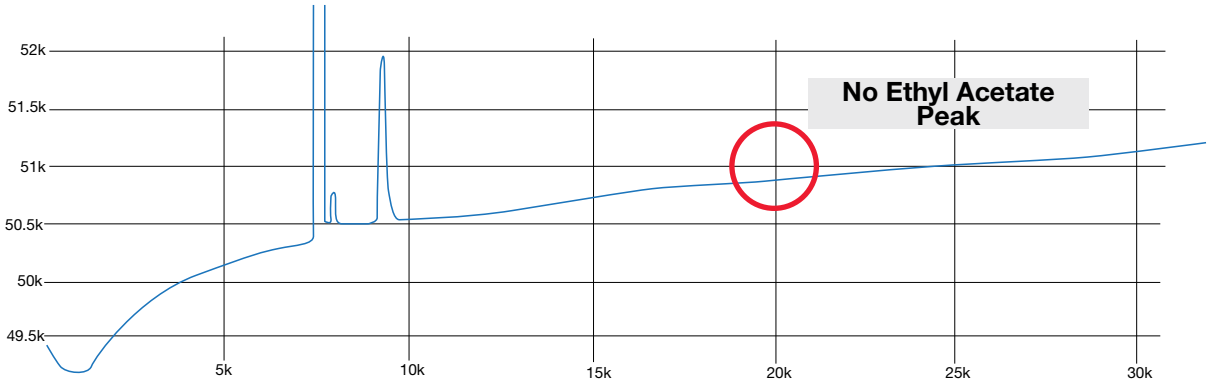


Figure 13.
Ethyl Acetate with SecurityCAP™ Installed



Results and Discussion

The results of the experiment are represented in the form of chromatograms. **Figure 2** shows a blank chromatogram that shows three distinct peaks. The first peak represents atmospheric nitrogen, second peak represents carbon dioxide, and the third peak represents the moisture present in air. Since TCD is a universal detector, it may be able to detect even more permanent gases. **Figure 3** shows the comparative chromatogram for experiment with the acetonitrile bottle without a SecurityCAP and **Figure 4** shows the chromatogram with the SecurityCAP installed. The open bottle without a SecurityCAP showed contamination of acetonitrile while the SecurityCAP resulted in a chromatogram that displayed no acetonitrile peak. **Figures 5 - 8** shows the same pattern where SafetyCAP installed bottles showed no solvent contamination with methanol, tetrahydrofuran respectively. **Figure 9** shows a higher peak intensity for an open bottle of dichloromethane. This is because dichloromethane has a lower boiling point than other solvents considered for this study. So, it readily goes to the vapor phase at room temperature and contaminates the atmosphere easily. However, it is a chlorinated solvent and it has a very low permitted exposure limit of 25 ppm as per OSHA as shown in **Table 1**. **Figures 12** and **13** show chromatograms for ethyl acetate with and without a SecurityCAP. Since ethyl acetate has a higher boiling point, a very low peak intensity was observed using the open bottle.

Table 1 shows the permitted exposure limit as per OSHA for all the solvents that are considered for this study. Exceeding these limits will cause various health hazards and can even be fatal. Not only do open solvent bottles cause health hazards, but also the saturated solvent vapors can cause potential fire accidents in the laboratory. Aside from this, a contaminated lab environment with a mixture of solvents can show false positive and ghost peaks on complementary chromatographic techniques. For example, there are numerous labs that have HPLC and GC instruments side by side. In GC, the analytes of interest are commonly volatiles, while in HPLC, these are used as mobile phase. If the mobile phase bottles are kept open or if they are not sealed completely with a SecurityCAP, solvent contaminants will be observed in GC data. The table also shows the ICH limits for residual solvents as per guidelines Q3C(R6). USP has the same limits for these solvents in pharmaceutical ingredients. Prior to testing the pharmaceutical ingredients for residual solvents, it is highly recommended to run blanks on a GC instrument. If the lab environment is contaminated with solvent vapors from open mobile phase bottles, the contamination peaks for methanol, acetonitrile, and methylene chloride, etc. will be observed. The intensity of contaminated peaks in the blank will be enhanced if the adopted method is headspace or mass spec based detection as these techniques have very low detection limits. If a blank chromatogram was not analyzed, false positive results would show up on samples due to HPLC solvent contamination in the GC instrument.

With SecurityCAP installation to the mobile phase reservoir and solvent waste reservoir, environmental contamination can be prevented. By adopting SecurityCAP mobile phase and solvent waste safety systems:

- health hazards due to solvent can be prevented
- fire accidents due to saturated solvent vapor can be prevented
- And ghost peaks or false positives for GC analysis can be prevented

Conclusion

The results demonstrate that solvent peak contamination can occur from open mobile phase bottles and can alter results due to vapor contamination. Using SecurityCAP mobile phase and solvent waste safety systems, lab environment contamination can be prevented, making SecurityCAP an essential component for safe laboratory practice.

References

1. https://www.osha.gov/dts/chemicalsampling/data/CH_253450.html
2. http://www.ich.org/products/guidelines/quality/article/quality-guidelines.html
3. http://www.ich.org/fileadmin/Public_Web_Site/ICH_Products/Guidelines/Quality/Q3C/Q3C__R6___Step_4.pdf

SecurityCAP™ Ordering Information

Starter Kits

SecurityCAP Mobile Phase (Eluent) Safety Starter Kits	
Part No.	Description
AC2-1245	2-port GL45 Cap and 6-month Safety Filter
AC2-4245	2-port GL45 Caps (x4) and 6-month Safety Filters (x4)
AC2-4240	2-port Merck S40 Caps (x4) and 6-month Safety Filters (x4)
AC2-1345	3-port GL45 Cap and 6-month Safety Filter
AC2-4345	3-port GL45 Caps (x4) and 6-month Safety Filters (x4)
AC2-4445	4-port GL45 Cap (x1) and 2-port Caps (3x) and 6-month Safety Filters (x4)
AC2-1445	4-port GL45 Cap and 6-month Safety Filter
AC2-1545	5-port GL45 Cap and 6-month Safety Filter
AC2-1561	5-port S60/S61 Cap and 6-month Safety Filter

SecurityCAP Waste Safety Starter Kits		
Part No.	Description	Unit
AC1-1245	2-port GL/DIN45 Cap and 6-month Exhaust Filter and Barbed Connector	ea
AC1-1545	5-port GL/DIN45 Cap and 6-month Exhaust Filter	ea
AC1-1551	5-port DIN51 Cap and 6-month Exhaust Filter	ea
AC1-1553	5-port B53 Cap and 6-month Exhaust Filter	ea
AC1-1561	5-port S61 Cap and 6-month Exhaust Filter	ea

Replacement Filters

SecurityCAP Mobile Phase Safety Filters		
Part No.	Description	Unit
AC2-0161	6-month Capacity, ¼ in.-28 Threads	ea
AC2-0961	6-month Capacity, ¼ in.-28 Threads	10/pk

SecurityCAP Waste Safety Filters		
Part No.	Description	Unit
AC1-0161	6-month Exhaust Filter for SecurityCAP, ¼ in.-28 Threads	ea
AC1-0361	6-month Exhaust Filter for SecurityCAP, ¼ in.-28 Threads	3/pk
AC1-0162	6-month Exhaust Filter for Wide-port Caps, GL14 Threads	ea
AC1-0362	6-month Exhaust Filter for Wide-port Caps, GL14 Threads	3/pk

Fittings and Accessories

SecurityCAP Fittings		
Part No.	Description	Unit
AC3-1101	for 1/16 in. or 2.0 mm ID Tubing, ¼ in.-28 Threads (POM), blue	ea
AC3-1201	for 2.3-2.6 mm ID Tubing, ¼ in.-28 Threads (POM), white	ea
AC3-2101	for ⅛ in. ID Tubing, ¼ in.-28 Threads (POM), black	ea

SecurityCAP Connectors		
Part No.	Description	Unit
AC3-1001	Barbed Connector, for 5-8 mm ID Tubing (PTFE), white	ea
AC3-1301	Y-connector, for 6-8 mm ID Tubing (POM), white	ea

POM = polyoxymethylene
PTFE = polytetrafluoroethylene (Teflon®)

SecurityCAP Adapter		
Part No.	Description	Unit
AC2-1138	Cap Thread Adapter, PTFE, GPI/GL 38 Female to GL45 Male	ea
AC3-1111	Waste Adapter for Male ¼ in. NPT-port (PTFE)	ea

SecurityCAP Sealing Plug		
Part No.	Description	Unit
AC3-2001	¼ in.-28 Threads (POM), white	ea

SecurityCAP Waste Safety Compatibility Table

	Phenomenex SecurityCAP Filters	
Supplier	ea	3/pk
S.C.A.T® SafetyWasteCaps	AC1-0162	AC1-0362
AIT Smart Healthy Caps	AC1-0162	AC1-0362
Agilent® InfinityLab Stay Safe Caps	AC1-0162	AC1-0362
VICI Jour® Waste Caps	AC1-0161	AC1-0361
Canary-Safe™ Safety Caps	AC1-0162	AC1-0362
DURAN® DG Safety Caps	AC1-0162	AC1-0362
VapLock™ Safety Caps (with AC3-1111)	AC1-0161	AC1-0361

guarantee

If SecurityCAP Safety Products do not perform as well or better than your current solvent safety products of similar type, dimensions, and material, return the product with comparative data within 45 days for a FULL REFUND.

APPLICATIONS

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