When using SPME the fibre is very likely to bend and have problems penetrating the septa. In order to make penetration easier, we first reduced the crimp cap septa’s thickness from 3.0 mm (which is a standard in 20 mm Headspace Crimp Caps) to approximately half that thickness. However, in order to compensate this considerable decrease in septa thickness, the crimp neck of the vial had to be changed to ensure effective crimping. Whilst this change solved the SPME penetration problems, it meant using customised vials for SPME with additional cost and inconvenience for the user.

With the Precision Thread Headspace Vials an improvement for Headspace as well as for SPME was introduced that could universally be used for both applications. The septa used in the Magnetic Screw Caps of these vials only had a thickness of 1.3 mm up to 1.6 mm which helped to protect the SPME fibre. Furthermore the risk of incorrectly crimped vials has been eliminated and reproducible analysis results are guaranteed. The special precision thread forces the septa firmly against the glass rim to seal the vial and ensure gas-tightness.

With the pre-cut septa technology additional protection is offered to the SPME fibre. Here the Silicone layer is cut through in a star shape, without affecting the PTFE-lamination which remains intact. As the Silicone part of the septa represents the major share of the complete septa thickness, the needle can now easily penetrate through the star shape for most of the septa's thickness. The intact PTFE-lamination will give 100 per cent sample integrity avoiding any contamination by Silicone material. Besides the ease of penetration with a pre-cut septa, there is also very low fragmentation of the fibre during the penetration process (very important for SPME). The star shape of the pre-cut was used to give the needle a large target area and to give the septa enough movement to spread apart for penetration.

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Phone: (52) 521 131
Fax: (52) 470 069
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Web: http://hu.vwr.com

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Slovenia | +43 1 97002-203 | info@si.vwr.com | http://si.vwr.com |

For all other CEE countries, website in English: http://ru.vwr.com
Dear Chromatographer,

We are proud to present to you the new copy of our VWR ChromJournal with lots of interesting articles to support your chromatographic tasks.

Focussing on a very important aspect ‘Safety’, we have articles featuring SCAT products showing important solutions for your laboratory.

Features include; Safety caps for solvent bottles which offer three main advantages by keeping the solvents and their vapours inside the bottles: 1. Keeps the air in the laboratory free from solvent vapours and therefore protects the personnel. 2. Ensures the reliability of the analysis by keeping solvent concentration and therefore the retention times constant. 3. Saves solvent costs. It also features sophisticated safety funnels which help to safely collect waste solvents for disposal.

With interesting contributions on products and applications to make your life as an analyst easier, reduce cost and improve the quality of your analytical results.

The VWR chromatography team wishes you well in your analytical tasks!

Kind regards

Wolf-Dieter Beinert

Product specialist at VWR International, Darmstadt, Germany

Scientific Instruments & Applications

Editorial

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Wolf-Dieter Beinert

Product specialist at VWR International, Darmstadt, Germany

Scientific Instruments & Applications

Table of Contents

Acrodisc® PSF Syringe Filters from PALL Life Sciences .................................................. 3
SCAT: Keep your HPLC retention time constant and save expensive solvents! .................... 4-5
New PRE-CUT septa for HPLC applications ................................................................. 6
Prepare HPLC / UHPLC samples three times faster using Whatman Mini-UniPrep syringeless filters ........................................................................................................ 7
VWR Collection HPLC Syringes ...................................................................................... 8
VWR Teflon AF Degasser, Model 2003 - 2005 ................................................................. 9
SCAT Europe SafetyFunnels for collecting waste fluids .................................................. 10
New Headspace plunger from Hamilton for CTC PAL System ........................................... 11
VWR Collection PRE-CUT septa for SPME applications ................................................ 12
Acrodisc® PSF Syringe Filters

Acrodisc PSF syringe filters are available in many membrane types, sizes, and packaging configurations to fit all applications. These are the only syringe filters to receive Caliper Life Sciences Automation Certified guarantee, providing:

- Smooth operation and worry-free performance in automated applications with smooth filter-to-filter release, consistent turret advancement, exceptional housing strength, and strict outside filter geometry.
- Best protection for HPLC columns – extend column life as much as 46 times.
- Maximum throughput with the GxF multi-layer prefilter.

Pall have developed unique technical guides and support to help you select the correct device for your application. This information added with our product HPLC certification will give you the confidence in reproducible and accurate analytical results.

Low Drug Binding with API Chemistries

The purpose of sample filtration prior to HPLC injection is to remove non-dissolved solids. Non-dissolved solids interfere with the resulting chromatography by continuing to dissolve throughout the period of the analysis, and by plugging the HPLC column.

It has been shown that sample preparation using filtration results in more reproducible chromatography and longer column life, however there are thought to be potential drawbacks with filtration as a preparation for HPLC analysis in dissolution testing. The first is that the filter may adsorb active pharmaceutical ingredients (API) from the drug mixture. Unwanted drug adsorption as well as the presence of possible extractables eluted from the filter during routine pharmaceutical sample analysis could be a problem and can cause out-of-specification results.

To determine the effects on drug analysis from the syringe filter, Pall commissioned an independent lab to perform a study to measure the amount of drug that the filter removed, thus showing the impact on the results.

The results of the study demonstrate that the use of Pall syringe filters does not affect the HPLC determination of dissolution test samples, whilst still maintaining reproducible results and maintaining column life.

To obtain a copy of this report and information about our extensive range of analytical sample prep products please email labpromotionsEU@pall.com and request either an electronic or printed copy.
Keep your HPLC retention time constant and save expensive solvents!

A simple test was carried out to compare the use of ‘open to atmosphere’ eluent caps versus SCAT Europe SafetyCaps. A test mixture of 3 PAHs (Polycyclic Aromatic Hydrocarbons) was used for a 20 day comparison of the chromatograms. Water and acetonitrile at low flow rate (to reduce solvent consumption) was selected as the mobile phase.

The test revealed clearly that without using a closed solvent delivery system such SCAT Europe SafetyCaps, unreliable retentions times may be encountered even after a relative short time. Also volatile expensive solvents may be wasted!

Test conditions

Bottle A: This bottle was closed with a SCAT SafetyCap (590-0506), precisely fitting the standard GL45 glass bottle thread.

Bottle B: This bottle was closed with a cap with a 10 mm hole in the plastic material, giving an ‘open to atmosphere’ area of ~0.785 cm².

Procedure

- Both bottles were filled at the start of the test with an identical mixture of water: acetonitrile (20:80 w/w).
- Using Bottle A as the reference, a chromatogram of a mixture of three PAHs (Polycyclic Aromatic Hydrocarbons) - Benzo(a)pyrene, Benzo(ghi)perylene, and Indeno(1,2,3-c,d)pyrene, was acquired for comparison.
- After the reference chromatogram was obtained, both bottles were stored at room temperature in a fume hood, this provided a gentle air flow over the top of the bottles.
- The analysis of the test mixture was repeated after 9 and 20 days.

HPLC system

A VWR Hitachi LaChrom Elite® HPLC system with Diode Array Detector, and under the control of EZChrom Elite™ Software was used. Isocratic pump conditions with the premixed mobile phase were set.

HPLC Column: Merck Superspher® 100, RP-18e (5 µm), 125 x 2 mm

Flow rate = 500 µl/ min

Results

As expected, bottle A did not show any significant change in weight, concluding that no solvent vapours escaped from this bottle, see Tab. 1.

Bottle B showed significant and varied loss of liquid by vaporization. The composition of water and acetonitrile in the vapour may have varied over the time due to the fact that mixtures of water and acetonitrile form azeotropes.

<table>
<thead>
<tr>
<th>Day</th>
<th>Bottle A (g)</th>
<th>Bottle B (g)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>666.44</td>
<td>673.59</td>
</tr>
<tr>
<td>9</td>
<td>666.22</td>
<td>650.85</td>
</tr>
<tr>
<td>20</td>
<td>665.26</td>
<td>609.12</td>
</tr>
</tbody>
</table>

Fig. 1: graphical representation
The significant loss of more acetonitrile than water from the ‘open to atmosphere’ bottle was revealed by the chromatographic results; see Fig. 2.

As a result of storing the solvent bottle with a premixed mobile phase ‘open to atmosphere’, a significant shift in retention time of the test compounds was observed. This would make a retention time based compound identification completely impossible. If one suggests roughly the same “linear behaviour” of the vaporization of volatile HPLC solvents out of a eluent mixture, it becomes obvious, that even after one day of using non-sealed mobile phase bottles the user may expect changes of retentions times.

**Conclusion:**

Without using tightly sealed solvent bottles as containers for premixed HPLC eluents there is clear risk of uncontrolled vaporization of solvents, resulting possibly in wrong retention time assignment for eluting compounds.

The use of SCAT SafetyCaps definitely prevents such uncontrolled evaporation, and in addition preserves your laboratory environment from contamination of toxic compounds!

**Not this way!**

[Image of an unsealed bottle with a notecard saying “CLOSED FOR MATT REASONS”]

**Much better!**

[Image of three sealed bottles]

---

**Tab. 2: Retention time shift over 9 and 20 days**

<table>
<thead>
<tr>
<th>Compound</th>
<th>Day 0</th>
<th>Day 9</th>
<th>Days 20</th>
</tr>
</thead>
<tbody>
<tr>
<td>Compound 1</td>
<td>2.29</td>
<td>2.32</td>
<td>2.88</td>
</tr>
<tr>
<td>Compound 2</td>
<td>3.45</td>
<td>3.51</td>
<td>4.52</td>
</tr>
<tr>
<td>Compound 3</td>
<td>4.77</td>
<td>4.86</td>
<td>6.39</td>
</tr>
</tbody>
</table>

---

For more information on these products contact your local VWR sales office, send an e-mail to chromjournal@eu.vwr.com or visit our website http://eu.vwr.com/chrom
New PRE-CUT septa for HPLC applications

In HPLC often slitted or cross-slitted Silicone/PTFE Septa are used, in order to offer a penetration aid for the fairly thick and dull needles used in the HPLC instruments. While the instrument needle requires the slitted septa, the user may run into the risk of concentration changes due to the evaporation of solvents. The risk increases with the volatility of the solvent used and especially in cases of for example, Hexane this problem may end in non-reproduceability of analysis results.

In order to avoid these risks, the manufacture has developed the so-called pre-cut septa. Here the Silicone carrier material is cut through in Y-form, however, the PTFE-lamination remains unhurt. As the Silicone part of the septa represents 95 per cent of the complete septa thickness, the needle can easily penetrate through the Y-slit for most of the septa’s thickness. Only the 0.05mm thick PTFE-lamination which is uncut, needs to be penetrated by the HPLC needle itself, but this goes without any problems even for these needles. The intact PTFE-lamination will also give 100 per cent protection to the sample with regard to any possible contamination until the needle pierces through it to pull out the sample for injection. The Y-form of the pre-cut was chosen, in order to give the needle a lot of target area for penetration, even if the needle does not come down centrically. Also the Y-form of the pre-cut allows a better flow of air during sample pick up, in order to avoid a vacuum in the vial. Samples are available on request.

Furthermore there is a risk that the volatile solvent may extract something out of the carrier material of the septa (ie. Mainly out of the Silicone), as the chemically inert PTFE-lamination has been cut through as well as the Silicone layer with slitted or crossslitted liners. Thus two risks (concentration change + contamination through extraction) with a lot of impact to the analysis go along with slitted septa, even though the needle may require it.

VWR Collection

The solution to your everyday needs

High quality products at competitive prices

The VWR Collection is a continually evolving range designed to meet your everyday laboratory needs. Selected by and created for VWR International, the VWR Collection is the fast growing source of products making a real difference to your lab’s productivity.

Please contact our sales office or go to www.vwr.com for the latest news and special offers
Prepare HPLC / UHPLC samples three times faster using Whatman Mini-UniPrep syringeless filters

Whatman
Part of GE Healthcare

Whatman Mini-UniPrep™ Syringeless Filters provide a faster, easier way to remove particulates from samples and will provide a visual confirmation that all samples analysed are filtered. Instead of using a syringe, syringe filter, vial, and septum, just use one Mini-UniPrep and perform the same manipulation in just a few seconds.

Because of the specific demands for sample preparation in chromatography applications, sample preparation can become a bottleneck in the daily activity of the laboratory. Protection of columns for HPLC applications has always been advised and with the latest developments of instruments moving towards Ultra HPLC, this has become increasingly important. Using smaller particle sizes and higher pressures, columns can achieve increased resolution of separated components. However, the cleanliness of the sample preparation is critical.

Continuously striving to improve and innovate, Whatman has listened to these new needs and created a whole family of Mini-UniPrep filters.

You will see the huge benefits for your laboratory with the time and money saved by removing the need for multiple consumables from the sample preparation process.

For customers using slit septa, Whatman offers Slit Septa Mini-UniPrep.

For customers who need to protect samples from light damage, there is Amber Mini-Uniprep.

To maximise the sample preparation throughput, Whatman developed the 6 Place Compressor. This will allow you to process 6 Mini-UniPrep at the same time and will reduce the risk of hand stress.

The filter membrane is housed at one end of a plunger, with a cap/septum at the other. By pressing the plunger through the sample placed into the chamber, positive pressure forces the filtrate into the reservoir of the plunger. Air escapes through the vent holes until the evaporation seal is engaged, providing an airtight seal. Within seconds the Mini-UniPrep can be placed into your autosampler for injection into your instrument.

Six position compressor makes your job even easier

Please contact your local VWR customer support for availability of amber coloured housing or slit septum.

Mini-UniPrep benefits:

- Reduces sample prep time and improves efficiency
- Reduces costs
- Reduces laboratory waste
- Protects columns – as filtration is in vial
- Gives visual conformation sample has been filtered
- Flexible: designed to fit in autosamplers that take standard 2 ml vials
- Flexible: available with a choice of membranes

Technical specification

<table>
<thead>
<tr>
<th>Dimensions</th>
<th>Equivalent in size to 12 mm x 32 mm vials</th>
</tr>
</thead>
<tbody>
<tr>
<td>Materials of construction</td>
<td>Housing and cap: Polypropylene</td>
</tr>
<tr>
<td>Filter media: As specified</td>
<td>Septa: PTFE membrane and silicone rubber</td>
</tr>
<tr>
<td>Filtering capacity</td>
<td>0.4 ml</td>
</tr>
<tr>
<td>Nominal force needed to compress</td>
<td>Approximately 8 psi (0.6 bar)</td>
</tr>
<tr>
<td>Max operating temperature</td>
<td>120° F (50 °C)</td>
</tr>
</tbody>
</table>

Choose the right Mini-UniPrep filtering media

<table>
<thead>
<tr>
<th>Sample media</th>
<th>Suitable Mini-UniPrep media</th>
</tr>
</thead>
<tbody>
<tr>
<td>Particulate laden liquids</td>
<td>Glass Microfibre (GMF)</td>
</tr>
<tr>
<td>Aqueous/organic samples in 3 to 10 pH range</td>
<td>Nylon (NYL)</td>
</tr>
<tr>
<td>General filtration media/solvent based samples</td>
<td>Polypropylene (PP)</td>
</tr>
<tr>
<td>Chemically aggressive solutions</td>
<td>Polytetrafluoroethylene (PTFE)</td>
</tr>
<tr>
<td>Biological samples requiring low protein binding media</td>
<td>Polyethersulphone (PES)</td>
</tr>
<tr>
<td>Aqueous/organic solvents – low nonspecific protein binding media</td>
<td>Polyvinylidenefluoride (PVDF)</td>
</tr>
<tr>
<td>Aqueous/organic solvents – high flow and loading capacity</td>
<td>Polypropylene Depth (dp PP)</td>
</tr>
<tr>
<td>Aqueous/organic solvents – low nonspecific protein binding media</td>
<td>Regenerated Cellulose (RC)</td>
</tr>
</tbody>
</table>

Simple
Place unfiltered sample in chamber.

Innovative
Compress filter plunger into sample chamber. Clean filtrate fills reservoir from bottom up.

Convenient
The Mini-UniPrep vial shape fits easily in autosamplers.
VWR is proud to present the new VWR Collection Chromatography Syringes. This new line from VWR is the optimum addition to your laboratory providing easy liquid transfer.

VWR introduce a broad range of different syringes for manual or automated sample preparation, or sample injection into HPLC or GC systems. The syringes give optimum precision and accuracy together with excellent price-performance ratio.

One of the new VWR Collection Chromatography Syringe product lines is:

**Super Elastic Plunger models**

The Super Elastic Plunger models are a new age of syringes with a special Titanium-Nickel alloy plunger which have stronger resistance to chemicals and bending.

**Super Elastic Plunger features:**

- **Superior flexibility** of a special Ti-Ni alloy which will bend but won’t kink.
  - Plunger can be wound into a 16 mm diameter coil
  - Caution: Super-elasticity is weakened below -10°C and disappears over 200 °C

- **Superior chemical resistance**
  - The robust Ti-Ni alloy is more resistant to acid, alkalis and salt solutions than standard stainless steel (ref. Fig. 2)

- **Accuracy and reproducibility**
  - Accuracy and reproducibility of injection is within ± 1 %

<table>
<thead>
<tr>
<th>Description</th>
<th>Cat. No.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Syringe micro 10 μl STD</td>
<td>549-0199</td>
</tr>
<tr>
<td>fixed needle</td>
<td></td>
</tr>
<tr>
<td>Syringe micro 100 μl Gas</td>
<td>549-0539</td>
</tr>
<tr>
<td>Tight luer lock</td>
<td></td>
</tr>
<tr>
<td>Syringe micro 10 μl</td>
<td>549-0567</td>
</tr>
<tr>
<td>23 Gauge, Agilent 7673A</td>
<td></td>
</tr>
</tbody>
</table>

Compatible with Agilent and Rheodyne HPLC valves.

The Super Elastic plunger provides extended syringe life time.
VWR Teflon AF Degasser Model 2003 - 2005

Teflon® AF is 50x more permeable than the old Teflon® PTFE

The new Teflon® AF membrane outperforms the older Teflon® PTFE membranes used in many other degassing systems today. This translates into the ability to use shorter tubing for removal of dissolved gases.

On-Line degassing has been shown to be more effective at removing dissolved gas from mobile phases than sonication or helium sparging.

Up to five solvent lines can be degassed simultaneously by one unit. The extremely low volume of each Teflon® AF channel (480 µl) offers very quick equilibration and very short start-up times.

Benefits

• Considerably shorter equilibration/re-equilibration times
• Very easy to prime
• Short vacuum pull-down times, typically 3 minutes
• Teflon AF has the ability to efficiently remove air bubbles from solvents and therefore to increase the reliability of the HPLC analysis

Vacuum Pump Technology

New is the ZHCR® (Zero Hysteresis Constant Run) stepper motor driven vacuum pump, specifically designed and developed for membrane degassing of HPLC mobile phase. Employing a micro-stepping closed-loop vacuum control strategy permits the pump to maintain a constant vacuum level set-point (50 mm Hg) by varying the RPM of the stepper motor. The pump initially runs at a high speed, which provides for a quick pull-down and, as it approaches the vacuum control point, the RPM is gradually reduced until the desired vacuum level is reached. This patented control strategy allows the On-Line Degasser to maintain a virtually constant vacuum that is unaffected by varying degassing loads. As a consequence, fluctuations in baseline due to vacuum repeatedly stop and start as is done in many older and existing systems.

Benefits

• Extremely quiet operational noise
• Long life Expectancy - 5 years continuous run
• Pump head purge eliminates need for purge valve
• Eliminate baseline fluctuations due to the vacuum pump
• Short vacuum pull-down times, typically 3 minutes
• Closed loop vacuum control means constant vacuum (variable RPM)
• Advanced error and leak checking functions

Specification

<table>
<thead>
<tr>
<th>Specification</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Number of channels:</td>
<td>3, 4 or 5</td>
</tr>
<tr>
<td>Internal Channel Volume</td>
<td>480 µl</td>
</tr>
<tr>
<td>Max. Flow rate:</td>
<td>10 ml/min</td>
</tr>
<tr>
<td>Degasging Capacity:</td>
<td>~ 2 ppm at 1 ml/min</td>
</tr>
<tr>
<td>Wetted Parts:</td>
<td>Peek, PTFE and Teflon AF</td>
</tr>
<tr>
<td>Dimensions:</td>
<td>127 mm (H) x 273 mm (W) x 249 mm (D)</td>
</tr>
<tr>
<td>Power:</td>
<td>15 to 24 VDC, 0.85 A max. (0.5 A typical)</td>
</tr>
<tr>
<td>Weight:</td>
<td>2.7 kg</td>
</tr>
</tbody>
</table>
In line with the SCAT Europe target to offer quality, safety and overall functionality, SafetyFunnels are now available from VWR. Not only used in the laboratory but also in production and storage areas; the SafetyFunnels with cover, stainless steel sieve and grounding connection is especially developed for use in tight spaces. There’s no safer way! Ignition sources are avoided by the electroconductive HDPE which offers perfect protection against electrostatic charges.

In combination with the electroconductive 10 litre waste container with mechanical level control the total height is just 39.5 cm including the angled adapting connecting piece - used to keep the funnel upright. Available with different thread diameters; the SafetyFunnel can be used on every standard waste container. Also there’s a special connector available for barrels.

Simple to install and remove, the funnel has a diameter of 13.5 cm and in combination with the stainless steel sieve that acts as an additional splash guard the disposal of large quantities of liquid waste becomes easy. The cut-way drawing explains the functional details of the funnel. It’s clear to be see that SCAT Europe does what ever is possible to reduce dangerous vapours in the laboratory.
New Headspace plunger from Hamilton for CTC PAL System

A spring supported seal increases the performance in Headspace analysis.

- The new sealing system allows the plunger to work with enhanced tightness over the whole temperature range from 40-150 °C.
- The lifetime is 3-5 times higher compared to today’s Headspace syringes on the market.*

* lifetime may vary on conditions used on application.

With this new plunger design Hamilton has set a new standard for Headspace syringes. The new patented plunger design has been optimised for higher throughput in Headspace analysis.

<table>
<thead>
<tr>
<th>Description</th>
<th>Pack size</th>
<th>Cat no</th>
</tr>
</thead>
<tbody>
<tr>
<td>Model 1001; Volume 1000 µL; Gauge 23; (23/51/5) CTC</td>
<td>1 pk</td>
<td>549-1324</td>
</tr>
<tr>
<td>Model 1002; Volume 2500 µL; Gauge 23; (23/51/5) CTC</td>
<td>1 pk</td>
<td>549-1325</td>
</tr>
<tr>
<td>Model 1005; Volume 5000 µL; Gauge 23; (23/51/5) CTC</td>
<td>1 pk</td>
<td>549-1326</td>
</tr>
<tr>
<td>Model 1001; Volume 1000 µL; Gauge 26; (26/51/5) CTC</td>
<td>1 pk</td>
<td>549-1327</td>
</tr>
<tr>
<td>Model 1002; Volume 2500 µL; Gauge 26; (26/51/5) CTC</td>
<td>1 pk</td>
<td>549-1328</td>
</tr>
<tr>
<td>Model 1005; Volume 5000 µL; Gauge 26; (26/51/5) CTC</td>
<td>1 pk</td>
<td>549-1329</td>
</tr>
</tbody>
</table>

CTC Instrument are original equipped with HAMILTON syringes, ask VWR for the HAMILTON syringe catalogue.

For more information on these products contact your local VWR sales office, send an e-mail to chromjournal@eu.vwr.com or visit our website http://eu.vwr.com/chrom