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Septum seal without elastomer component for vial closures

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In instrumental analysis, sample bottles, which are also known as vials, threaded sample bottles, fine-thread bottles, flared rim bottles, rolled rim bottles, snap cap bottles, snap ring neck bottles or injection bottles, are fitted with closures, which usually consist of a perforated cap with an inserted septum seal. Usually, septum seals consist of an elastomer such as natural rubber, butyl rubber, silicone rubber or fluororubber.

For better chemical resistance, seals are laminated with thin films of fluoropolymers, generally on product side. Depending on the application, elastomers can also be laminated with thin films made of metal or another polymer such as homopolymers, copolymers, polymer blends.

For sampling, the septum is punctured with the cannula of a microlitre syringe, an aliquot of the gaseous or liquid samples is taken and injected automatically or manually into the analyzer. The septum seal has the following functions:

- ⇨ sealing the vial's neck by means of a perforated cap.
- ⇨ sealing the microlitre syringe cannula when puncturing the septum to fill the microlitre syringe.
- ⇨ after pulling out the cannula, the septumcloses tightly again by contracting the elastomer (analogous to a Bunsen valve).

However, the puncture in the seal's lamination remains open, as a fluoropolymer such as PTFE, for example, has little elasticity. This hole corresponds approximately to the diameter of the cannula of the microlitre syringe. Due to this permanent damage to the seal, parts of the analytically rather "impure" elastomer can contaminate the sample solution. MS detectors working at the detection limit can detect substances (e.g. plasticizers) from the elastomer component, which makes the evaluation of the analysis more difficult or even impossible because they cause disruptive interfering peaks.

In trace analysis, the requirements for optimum sample integrity of the septum seal in closures for sample bottles are very high. The analytes are preferably dissolved in low-boiling solvents and double or triple determinations are carried out from the same sample, for quality assurance. This means that the syringe needle penetrates the same septum several times (multiple injection). The period in which further injections through the same septum take place depends on the subsequent analysis, usually the subsequent injections are carried out within a few hours.

In trace analysis, vial closures with thin, analytically pure layers (e.g. fluoropolymers, aluminum, polyethylene) with a thickness of approx. 0.05 to 0.3 mm are frequently used instead of laminated elastomer septa with a thickness of approx. 1 to 3.5 mm. Disadvantages of such "thin" foil septa in vial closures are:

- ⇨ after puncturing the septum's foil with the cannula of a microliter syringe once, the septum gets a hole which does not close again due to lack of elasticity. This allows solvents to evaporate from the sample solution. This alters the analyte solution's concentration and prevents a reproducible multiple injection.
- ⇨ due to the lower thickness of the septum seal, the flanging tool has to be readjusted for roller rim closures.
- ⇨ due to puncturing with the cannula, the smooth, thin foils can be pressed into the sample, since in the case of a thin film no sufficiently large pretension can be applied with the contact pressure of the cap.
- ⇨ during transport, the inserted septum seals may fall out of the pre-assembled closures.

The only advantage of the analytically pure, "thin" film septum is that the septum consists of only one material and there is no elastomer that can contaminate the sample.

Closures with the ZeroSept®AIR septum "PTFEvirginal/AIR/PTFEvirginal" have an air cushion instead of an elastomer. A

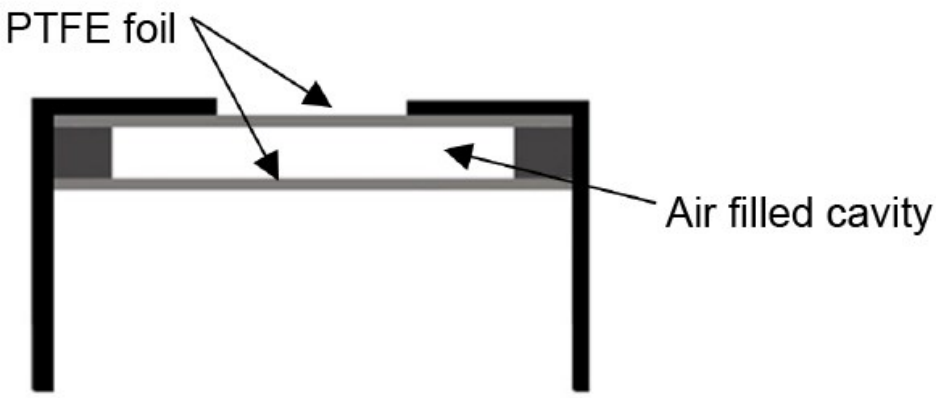

Fig. 1: Scheme of the ZeroSept®AIR septum "PTFEvirginal/AIR/PTFEvirginal"

special fluoropolymer perforated disc is connected to a thin PTFE film at the inlet and outlet ends, creating an air-filled cavity between the two (see Figure 1).

The structure of the ZeroSept®AIR septum has the following advantages:

- ⇨ the thickness of the ZeroSept®AIR septum is similar to that of a conventional elastomer septum. Therefore, the flanging tool does not have to be readjusted for roller rim closures.
- ⇨ due to the firm connection of the PTFE foils with the fluoropolymer perforated disk, the inserted ZeroSept®AIR septum does not fall out of the pre-mounted closures during transport and the smooth, thin foils can no longer be pressed into the sample when punctured by the cannula.
- ⇨ the syringe needle cannot punch out any elastomer material when puncturing the septum and thus possibly contaminate the sample.
- ⇨ the air-filled cavity and two punctured PTFE foils considerably reduce the evaporation of solvents and the associated change in concentration of the sample solution in the sample vial. This results in an exact and reproducible measurement of the same sample, e.g. within 24-36 hours after the first injection (see also table 1 below (PTFE/AIR/PTFE)).

Impermeability comparison of different septa

Application

20 mL headspace vials were filled with 15ml acetone and closed with fine-thread screw caps, which were equipped with four different septum materials.

The closed vials were punctured with an injection needle (AD 0.47 mm) and stored at 23°C. Depending on the time, the gross weight was measured by double determination.

Results

As shown in table 1, none of the samples stored for 24 hours at 23°C, lost weight due to solvent evaporation, which could affect the measurement results.

Conclusion

The ZeroSept®AIR-Septum, in contrast to foil septa, has a sufficiently high impermeability compared to conventional septa and is therefore suitable for multiple injections.

Due to its air cushion the ZeroSept®AIR septum corresponds in thickness to the elastomer septa, so flanging tools do not have to be readjusted and seals cannot fall out of the pre-assembled closures during transport.

In addition, ZeroSept®AIR for standard vial closures eliminates the aforementioned disadvantages of conventional laminated elastomer septa, as no elastomer components can contaminate the sample. Therefor it is very interesting for trace analysis.

In combination with standard vials, they fit into all common GC/HPLC systems and, in magnetic design, can also be moved with an autosampler.

Tab. 1 Impermeability comparison of different septa

time (h)	1A	1B	2A	2B	3A	3B	4A	4B
	PTFE/AIR/PTFE		Silicone white/ PTFE blue		Butyl red/PTFE grey		Butyl moulding disc dark/PTFE	
0	29,27	29,03	28,82	28,85	29,08	29,09	29,12	29,15
1,5	29,26	29,03	28,82	28,85	29,08	29,09	29,12	29,15
24	29,20	29,00	28,82	28,84	29,08	29,09	29,11	29,15
78,5	29,11	28,94	28,80	28,82	29,08	29,08	29,08	29,11
102,5	29,06	28,91	28,80	28,82	29,08	29,08	29,07	29,10
145,5	28,99	28,87	28,79	28,81	29,08	29,08	29,05	29,08