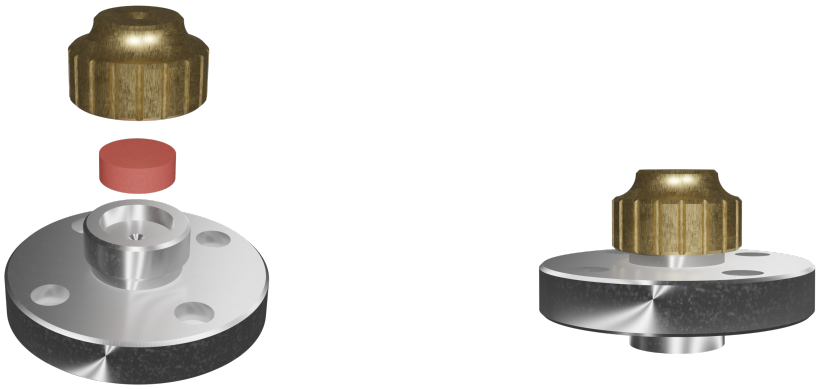
Ex-situ gas injection cell

EC-MS Technical Note #18

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Introduction

The gas injection cell provides an interface for introducing gas samples into the EC-MS ex-situ, for example a gas sample extracted from a battery pouch cell. The cell consists of a body with a slot for a septum and a screw cap with a needle injection port (see fig 1a). When the cell is assembled, the screw cap holds and compresses the septum to seal off the cell (see fig. 1b), which allows for controlled gas injections into the EC-MS.



(a) (b)

Figure 1: Gas injection cell

The gas injection cell is fastened to the EC-MS interface with four **M3 x 6** bolts tightened to 10 N cm, and also uses an O-ring to seal between the bottom face of the cell body and the chip, like other EC-MS cells. Note, that any chip (standard or non-aqueous) may be used, but the cell is only intended for gas injection - no liquids. Since standard chips are

generally more robust, these are recommended for this use. To protect the chip, the mass spectrometer and vacuum system components, no volume of gas should be forced into the cell abruptly. The pressure of the gas in the syringe (prior to injection) should be near ambient ($\approx$ 1 bar). Specifications for suitable septa and syringes (including needles) are given in table 1.

Accessory	Specifications
Syringe	
Type	Gas tight, with needle
Volume	$\approx$ 1 mL and above recommended
Needle	
Size	22-26 gauge (OD = 0.47-0.71 mm)
Point style	Style 2, 4, AS (bevel length < 1 mm)
Septum	
Diameter	10.0-11.0 mm
Thickness	3.0-3.8 mm

Table 1: Required accessories for gas injection

Note that syringe volumes less than 1 mL could be used for injection, but the sample dilution into the internal volume of the cell becomes increasingly dominant for smaller volumes, and this could significantly lower the signal observed with the mass spectrometer.

Gas injection procedure

Assuming the EC-MS system is in the safe state (including manual valves closed on EC-MS Premium systems), start a measurement with sampling of relevant m/z ratios and mass scan parameters. Then follow the standard chip pumpdown procedure. On EC-MS Premium systems, remember to open **MV**$_v$ before doing the pumpdown. Mount the cell body, insert a septum and screw on the screw cap until it is tight and the resistance increases significantly. The septum should sit firmly in place and be slightly compressed.

Cell volume pumpdown

Next step is to pump down the internal cell volume via the chip's membrane and built-in gas channel using the roughing pump:

(i) close **V1** to protect the turbo pump, then ensure **V14** is open

(ii) put the **PC** mode to purge. On EC-MS Premium systems, open **MV**$_o$ as well.

(iii) once the pressure on Pirani 3 has again dropped to an acceptable level, open **V1**.

(iv) wait for the **PC** readout to stabilize, indicating that the cell volume is fully pumped down. A **PC** readout < 10 mbar should be obtained, preferably < 5 mbar. It might take several minutes to achieve a stable, low pressure.

(v) now set the **PC** to flow off. On EC-MS Premium systems, close **MV**$_o$ as well.

Injection

With a measurement running in Zilien and both the chip and cell pumped down, the gas injection can be performed next.

(i) Extract the gas sample from its source using a suitable needle and syringe.

(ii) If possible, seal the syringe and needle immediately after extraction to avoid contamination.

(iii) Fully insert needle into the cell via the guide hole in the screw cap. The tip of the needle should pierce through the septum and reach the cone shaped volume right below.

Once the needle is correctly inserted, MID signals should promptly increase (depending on the m/z values selected and the species present in the injected gas). The differential pressure between the pumped down cell and the syringe acts as a driving force for getting in the gas sample, and will quickly equilibrate - pushing the syringe piston is not needed, and might cause an unwanted high pressure in the vacuum chamber. A stable MID and/or a full mass spectrum can now be acquired, after which the needle can be removed from the cell.

Example: Ar injection

A basic gas injection is demonstrated with the following example. A balloon is flushed a few times and then filled with Ar. A 1 mL luer tip syringe is filled with Ar by inserting the syringe tip into the baloon and pulling back the piston. A suitable needle with luer tip fitting is quickly placed onto the syringe. The syringe piston is used to push out ≈ 0.1 mL Ar in order to flush atmospheric species out of the needle volume before quickly inserting it into the cell. At t≈ 2400 s the needle is guided through the screw cap port, until it penetrates

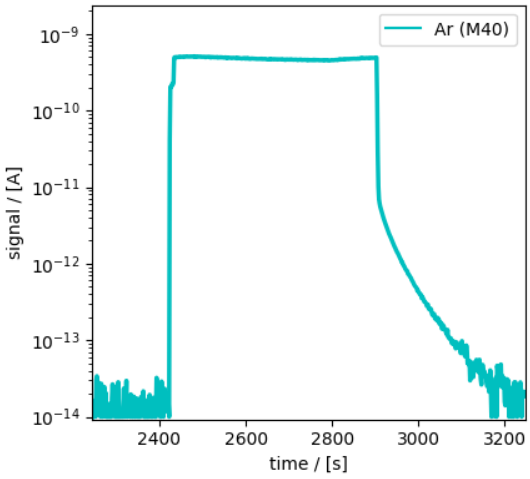


Figure 2: MID signal (**M40**) during Ar injection into the EC-MS

the septum and enters the cell. The M40 signal of the MID immediately increases several orders of magnitude (see figure 2). Note that the piston of the syringe is not pushed - the content of the syringe quickly equilibrates with the cell volume by itself. The needle is left in the cell until t≈ 2900 s, during which a quite stable M40 signal can be observed in the MID. After retracting the needle, the M40 signal immediately drops around two orders of magnitude and then further decreases at a more gradual pace.

Troubleshooting

If an acceptable PC readout is not obtainable when trying to pump down the cell volume (step (iv) of the procedure above), try tightening the screw cap further to ensure a tight sealing of the cell. If this resolves the issue, a further drop in the PC readout should be observable almost immediately. In the case that that further tightening does not help or that the screw cap is already adequately tightened, abort the cell pumpdown procedure and return to the safe state in Zilien. Remove the screw cap and replace the septum.

If the expected MID signals don't rise promptly upon injection, it could indicate that the needle has not fully gone through the septum. Push it in further if possible or retry the injection.