

The Open-Split Capillary Interface

Part No. 113532

INTRODUCTION

With the advent of fused silica capillary columns and their high degree of flexibility, it is obviously attractive to be able to take the end of the column directly into the ion source of the mass spectrometer.

This is fine in theory but in practice the vacuum system changes the chromatographic retention data making it difficult to transfer data obtained on a conventional gas chromatograph to a GC-MS system. An additional problem that can arise is overloading of the ion source when all the effluent from the column is loaded directly into the MS.

The Open-Split Capillary Interface is a device which overcomes many of these shortcomings.

Firstly, it enables the capillary column

to terminate at atmospheric pressure so retention data is unaffected.

Secondly, a helium purge enables the amount of material entering the mass spectrometer to be controlled.

Thirdly, columns may be changed without shutting down the mass spectrometer and without allowing air to enter the ion source.

Finally, the material not used by the mass spectrometer may be split by the purge gas to another detector e.g. FID or ECD.

THEORY OF OPERATION

The amount of component entering the mass spectrometer (Y) is governed by the relationship:

$$Y = \frac{F_2}{F_1 + F_3}$$

WHERE:

F1= flow in capillary column

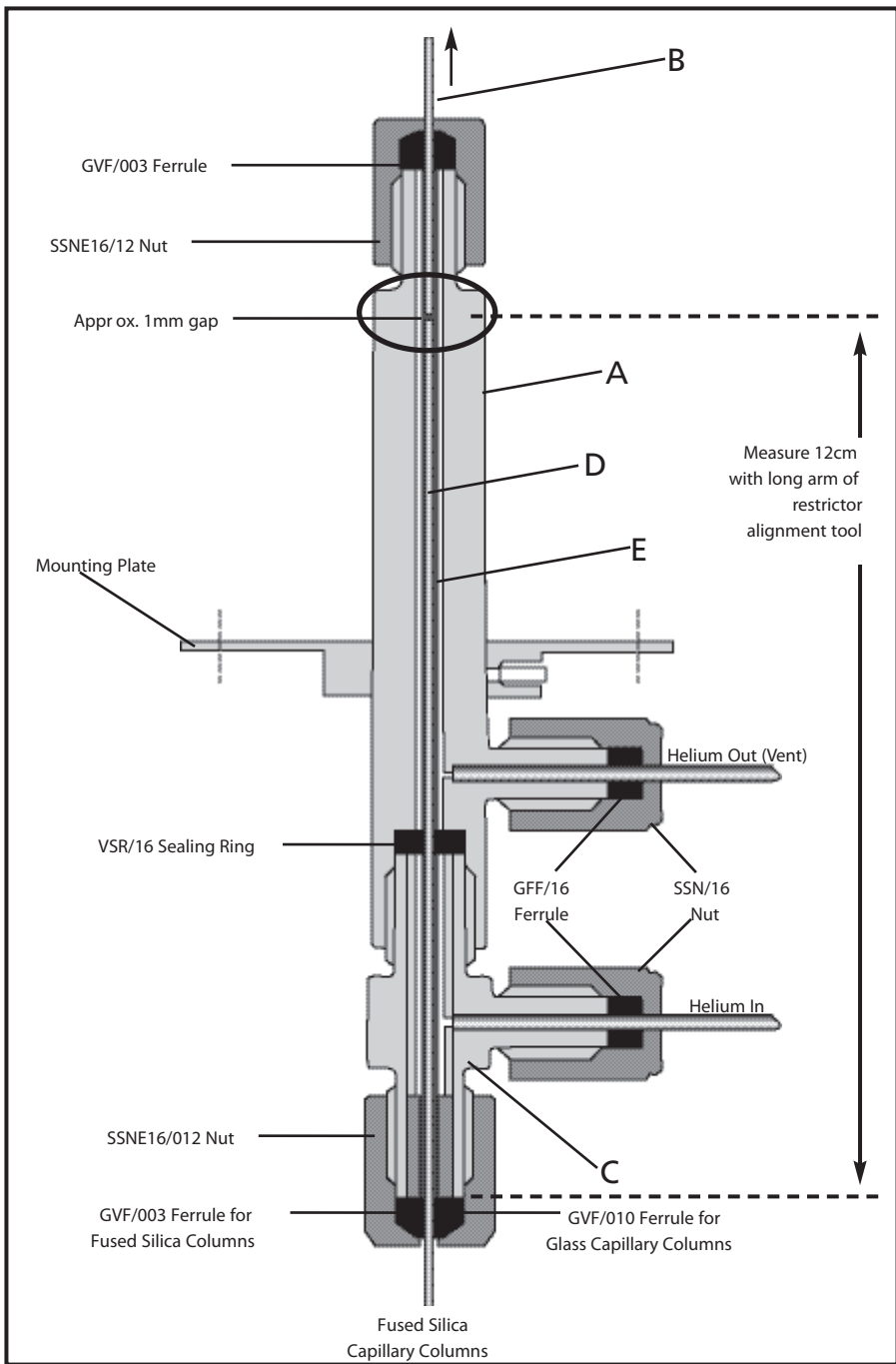
F2= flow into mass spectrometer

F3= purge flow

The flow F1, is dictated by the nature of the chromatography required and is therefore "Fixed". Similarly the flow F2 is determined by the volume of gas that the mass

spectrometer pumping system can handle in maintaining optimum pressure in the ion source. This flow is controlled by the length and diameter of the deactivated fused silica transfer line between the interface and the ion source. F2 is therefore also "Fixed".

F3, the purge flow, is the only "variable" entity in the relationship and its value must be known and accurately controlled. For this reason we advocate a mass flow controller for this purge even when the unit is used isothermally.



INSTALLATION

Installation of GMCC/90

1. Mount the GMCC in a convenient place inside the column oven of the GC-MS, close to the entrance of the ion source, using the mounting plate provided. If possible locate the Long end A of the GMCC in a heated section of the instrument preferably at constant temperature.
2. Select the appropriate MS connector and install it in the entrance to the ion source (SSMF/16S to fit Male 1/16" Swagelok®, SSFM/16S to fit Female 1/16" Swagelok as the case may be). Use SSMF/16E with the Finnigan 4000.
3. Connect a mass flow controller (0-10cc) to the inlet port of the MGCC using 1/16" OD stainless steel tubing.
NB: The inlet port is the closest to the end at which the column is inserted.
4. The vent may be connected to a detector, e.g. FID or ESD using 1/16" stainless steel tubing or may be led outside the oven through a convenient point.
5. Cut a piece of fused silica tubing from the deactivated material supplied. The length of this restrictor will be dictated by the distance from the GC to the ion source and by pumping capacity of the mass spectrometer. Begin with about 110µm ID (supplied) and adjust the length as required until the balance of ion source pressure and flow is achieved. Other bore size are available.
NB: Remember that the viscosity of gas INCREASES with temperature, so take this into account. e.g. Viscosity of Helium at 20°C is 194 micropoise while at 250°C it rises to 285 micropoise.
6. Insert one end of restrictor right into the ion source and lock into position using GVF/005 ferrule.
7. Insert the other end into the Long end A of the GMCC and locate its position by means of the restrictor alignment tool (12cm long) inserted into the Short end C of the GMCC.
8. Lock the restrictor into position in GMCC with GVF/005 ferrule.

INSTALLATION

Installation of Fused Silica Column

The column D is inserted into the GMCC until it meets the end of the restrictor. It is then withdrawn about 1mm.

NB: When using wide bore columns, it is possible to push the column over the end of the restrictor. If you

are in doubt, mark the column with erasing paint e.g. Liquid Paper® or Tipp-Ex® then withdraw it and compare the length of the inserted portion with length of the restrictor alignment tool.

Installation of Glass Capillary Column

Glass capillary columns are butt joined to the interface, using a GVF/010 ferrule.

OPERATION

From the preliminary discussion it will be obvious that maximum yield to the Mass Spectrometer can only be achieved when the purge flow is very low.

1. Set the inlet pressure to the mass flow controller at 50psi.
2. Set flow to about 0.5mL/min. These settings are purely a guide and will need to be adjusted as required to give optimum performance.

GENERAL MAINTENANCE

In the event that leakage occurs due to normal “wear and tear”, it will be necessary to replace seals as follows:

1. Remove restrictor from GMCC by loosening nut (SSNE16/012) and withdrawing the silica tubing. If it is intended that the same restrictor is to be used again, carefully remove the nut entirely with silica still inserted. This will facilitate reassembly.
2. Remove column from GMCC by loosening nut (SSNE16/012) and withdrawing the column.
3. Disconnect vent line.
4. Remove bottom T, C and GLT liner E.
5. Using the Sealing Ring Removal Tool to remove the Vespel® sealing ring (VSR/16).
6. Replace Vespel sealing ring on GLT liner and reassemble T to GMCC, making sure that GLT liner is “flush” with end of GMCC.
7. Replace restrictor and column as outlined in the installation procedure.

PARTS LIST

Open Split Adaptor	1
MS Connector 1/4", 1/8", 1/16"	3
SSMF/16E, Part No. 1034603	1
Graphite Ferrules GFF/16, Part No. 072603	5
Graphite Ferrules GFF/010, Part No. 072656	2
Graphitized Vespel® Ferrules GVF/003, Part No. 072659	5
Vespel Sealing Rings, Part No. 072653	5
2VSD-110 I.D. Deactivated Silica, Part No. 064226	1
8g x 1/2" Self Tapping Screw, Part No. 0932706	2
1/16" Allen Key, Part No. 18000100	1
SGE Spanner 1/14" - 5/16"	1
Sealing Ring Removal Tool, Part No. 0932704	1
Restrictor Alignment Tool for GMCC/90	1



SGE, Incorporated (USA)
Toll Free: (800) 945 6154
Tel: (512) 837 7190
Fax: (512) 836 9159
Email: usa@sge.com

SGE International Pty. Ltd.
Toll Free: 1800 800 167
Tel: +61 (0) 3 9873 4200
Fax: +61 (0) 3 9874 5672
Email: support@sge.com

SGE Europe Ltd (UK)
Tel: (0) 1908 568 844
Fax: (0) 1908 566 790
Email: uk@sge.com

SGE (France) Sarl
Tel: +33 (0) 1 6929 8090
Fax: +33 (0) 1 6929 0925
Email: france@sge.com

SGE (Deutschland) GmbH
Tel: +49 (0) 6151 860486
Fax: +49 (0) 6151 860489
Email: germany@sge.com

SGE Italia Srl.
Tel: 06 4429 0206
Fax: 06 4429 0724
Email: sge.italia@tin.it

SGE Japan Inc.
Tel: +81 45 222 2885
Fax: +81 45 222 2887
Email: japan@sge.com

SGE India
Tel: +91 (022) 471 5896
Fax: +91 (022) 471 6592
Email: sgeindia@vsnl.com

SGE China
Tel: +86 (10) 6588 8666
Fax: +86 (10) 6588 6577