The Mass Spec Source

VOL. XVI, NO. 3 NOVEMBER 1993

For Users of Mass Spectrometers and Gas Chromatographs

FEATURES

NEW MASS SPEC TIPS SECTION

CONTENTS

New product introductions at
Eastern Analytical Symposium

Scientific Instrument Services will once again be participating in the Eastern Analytical Symposium in Somerset, New Jersey on November 14-19. We anticipate this will be a good meeting, as we have a lot of activities planned including new product introductions, workshops and other presentations.

S.I.S. will occupy three booths at this years show. In addition to displaying our GC and MS accessories as well as our Thermal Desorption System, we will be introducing several NEW products at this show. The first of these new products is an Automated GC Cryo-Trap/Heater which will permit the cryo-trapping of organics on-column inside a GC oven during the injection process using liquid CO_2 as a cooling gas. After the trapping is complete, the trap can be temperature ramped in excess of 500 degrees per minute to elute the volatiles from the trap for subsequent GC or GC/MS analysis. This system can be used for both Thermal Desorption and Headspace GC techniques. This system is fully described later in this newsletter. In addition we will be introducing several new Mass Spectrometer Probes including a High Temperature Probe that can be used up to 800° C, a new Calibration Gas Probe, and a new Probe Temperature Controller. Be sure to stop by and see us for more information on these new products as well as our other supplies and services.

Workshop: "Recent Advances in Mass Spectrometer Inlet Techniques"

Chris Baker of S.I.S. will be chairing this workshop to update you on the latest techniques and hardware being utilized for the introduction of samples into the mass spectrometer. Several speakers will discuss the latest advancements in LC, thermospray, Capillary Electrophoresis, Fast Atom Bombardment and automated sample introduction techniques. This should be a good chance for you to become informed on the latest techniques and solutions to problems that you may have encountered in this area. For more information, contact Chris Baker at S.I.S.

Workshop: "GC Introduction Techniques - Thermal Desorption"

S.I.S. is sponsoring this workshop on Thermal Desorption to aid both the experienced user as well as individuals interested in the various thermal desorption techniques. Topics will be covered such as an introduction to the thermal desorption technique; description of the various techniques applicable to environmental, food, forensic, and other sample testing; selection and use of adsorbent resins and techniques for quantification using thermal desorption. If you want to know more about the techniques of thermal desorption and its capabilities plan to attend this workshop. For more information contact John Manura at S.I.S.

(continues on page 2)

New Product Introductions (continued from pg 1)

Both of these workshops are described elsewhere in this newsletter.

Posters

S.I.S. is presenting several posters during the technical session. The first of these is on the use of Tenax TA and Tenax GR Adsorbent Resins. This paper studies the breakthrough volumes of more than 100 volatile and semi-volatile organics on these two resins to determine the adsorption and desorption characteristics of these resins for use in thermal desorption applications. In addition we are presenting three other posters on the application of our Short Path Thermal Desorption System. These include the Flavor Analysis of Honeys, Analysis of Mushrooms and the Analysis of Air Samples from Commercial Buildings and Restaurants.

For more information on the Eastern Analytical symposium or to register for one of the workshops, consult your EAS program or contact Terry Nielsen, Chris Baker or John Manura at S.I.S. (908) 788-5550.

S.I.S. Short Path Thermal Desorption System

Featured on National Network

On Thursday, August 5, 1993, the ABC morning show Good Morning America aired a special report on foods and health as part of their regular morning programming. As part of this program, Good Morning America showed some footage in the Mass Spectrometer Laboratory at Rutgers University Center for Advance Food Technology (CAFT) in New Brunswick, NJ. Dr. Tom Hartman of the CAFT staff demonstrated the use of the S.I.S. Short Path Thermal Desorption System in the analysis of the flavor components of tomato products. Dr. Hartman demonstrated the attachment of the thermal desorption tube to the system and the desorption of the volatiles and semi-volatiles into the GC for subsequent analysis and identification via the Mass Spectrometer. The Short Path Thermal Desorption system was attached to a Varian 3400 GC and a Finnigan MAT 8200 GC/MS. The research on Tomatoes that is presently being done is part of a graduate research project by graduate student Karl Karmas of CAFT at Rutgers under the direction of Dr. Hartman. This group is studying the flavor profiles of various varieties of tomatoes as well as different stages of ripening and the effect of processing on the flavors of the consumer product. This work is being done utilizing the Short Path Thermal Desorption System. In addition to tomatoes, the group at CAFT is also studying potatoes, re-fried beans, spices, and a wide variety of other food products for their flavor components. CAFT is a research and support facility serving not only the university research but also serves as a problem solving source for companies throughout the food, pharmaceutical, & chemical industries. The CAFT group presently has four of the S.I.S. Short Path Thermal Desorption Systems for conducting much of this research.

Terms and Conditions

Scientific Instrument Services (S.I.S.) continues to supply "The Mass Spec Source" newsletter as a service to our customers. Printed six times a year, it includes articles and notes on new products and procedures of interest to mass spec and GC users. Papers from all fields of scientific inquiry in which mass spectrometry and gas chromatography can play a role will be considered and subject to review. However, S.I.S. reserves the right to reject any article that is in direct competition with S.I.S. products.

Articles and Application Notes

Editorials and reviews on new instrumentation and techniques for GC/MS will be considered for publication. These articles can be any length and our Graphics Department will aid you in any way you may need.

All articles and application notes in this publication are reviewed by two peer reviewers from the mass spectrometer community.

Mass Spec Tips

Any new ideas or tips that could benefit other mass spectroscopists can be submitted for inclusion in this section. Authors will be compensated $50.00 for each tip published n this newsletter. For each article or tip submitted, the authors name will be included in a yearly drawing at ASMS for a "free color TV" or gift certificate.

For Sale/Wanted

We advertise, for those looking to sell or buy, various mass spectrometers, leak detectors, gas chromatographs or other instrument parts. These parts may be new, used or reconditioned. Items are listed as described by the seller. If you wish to sell any mass spec parts or if you are looking for some particular part, please call Sandy Overton, editor (908) 788-5550. Be prepared to describe the item fully and indicate prices.

Laboratory Cartoons

S.I.S. will pay you for original cartoons related to the laboratory or GC/MS. We will consider cartoons related to GC/MS or any laboratory situation. Authors of cartoons printed in the Mass Spec Source will be paid $50.00 for their contribution. Our Graphics department can aid you with illustrations.

For More Information

Anyone interested in writing in any of the areas above should contact Sandy Overton, the editor of the Mass Spec Source, at (908) 788-5550. We are always trying to improve this newsletter, if you have any suggestions please give us a call. Thanks for your continued support.

Warranty

S.I.S. does not warranty that the items described herein are usable or fit for a particular purpose. Our company makes no representation as to condition or character of the merchandise. S.I.S. will not be responsible for consequential or special damages.

"The Mass Spec Source"

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Scientific Instrument Services, Inc.
1027 Old York Road
Ringoes, NJ 08551-1039

908-788-5550
FAX: 908-806-6631

Scientific Instrument Services Sponsored

WORKSHOPS

at the 1993 Eastern Analytical Symposium

November 15-19, 1993 Somerset, NJ

Scientific Instrument Services is chairing two workshops at the 1993 Eastern Analytical Symposium in Somerset, NJ on November 15-19, 1993. We have put together a group of speakers for these workshops to develop a program that will be both informative and educational for all who attend. If you are interested in attending either one of these half day workshops, please register through the Eastern Analytical Symposium registration or contact Terry at S.I.S. (908)-788-5550.

Mass Spectrometer Inlet Techniques: Recent Advances

Chairman: **Christopher Baker, Scientific Instrument Services**

Time: **Wednesday Afternoon, November 17, 1993**

This symposium is designed to be an informative series of presentations on the latest advancements on mass spectrometer inlet techniques particularly in the areas of Liquid Chromatography (LC), Capillary Electrophoresis (CE) and Fast Atom Bombardment (FAB). Lectures are being presented by the companies and individuals responsible for recent advances in these techniques.

1. ***"Recent Advances of LC/MS and CE/MS on a Benchtop Ion Trap Using Atmospheric Pressure Ionization"*** Jack Henion, A.Morehai, H.K. Lim, and J.Cai, Drug Testing and Toxicology, NYS School of Veterinary Medicine, Cornell University, Ithaca, NY.
2. ***"Routine High Flow LC/MS Using New Atmospheric Pressure Ionization Technology",*** C.S. Campbell, Finnigan MAT,San Jose, CA.
3. ***"Continuous Flow Fast Atom Bombardment Mass Spectrometry (CF-FAB/MS) With and Without the Addition of Matrix"***, Robert Dilliplane, Rohm and Haas Company, Spring House, PA.
4. ***"A Review of Ionization Mechanisms in Thermospray-Mass Spectrometry"*** Richard D. Hiserodt and Robert T. Rosen, Center for Advanced Food Technology, Rutgers University, New Brunswick, NJ
5. ***"Simplified Automated Sample Introduction into the Mass Spectrometer"*** Hewlett Packard Co., Paramus, NJ
6. ***"LC/MS Techniques: Expanding Every Day"***, Brian Musselman, JEOL USA, Inc., Peabody, MA.

GC Introduction Techniques - Thermal Desorption

Chairman: **John J. Manura, Scientific Instrument Services**

Time: **Thursday Morning, November 18, 1993**

This series of lectures is designed to be an instructional and educational program to aid the GC or GC/MS user with the application of Thermal Desorption Techniques. Topics will be covered such as the use and selection of adsorbent resins; environmental, food science, forensic and other applications; and techniques such as purge and trap, air testing and direct thermal extraction.

1. ***"Introduction to Thermal Desorption - Selection and Use of Adsorbent Resins for Thermal Desorption Applications"***, John J. Manura, Scientific Instrument Services, Inc., Ringoes, NJ.
2. ***"Use of Multibed Adsorbent Traps" and GC System Considerations for Thermal Desorption"*** Scott Hazard, Supelco, Bellefonte, PA.
3. ***"Methods and Techniques for Quantification Using Thermal Desorption"***, Thomas Hartman, CAFT, Rutgers University, New Brunswick, NJ.
4. ***"Optimization of Methods TO-1 and TO-2 for Ambient Air Analysis"***, Valerie Naughton, Tekmar, Cincinnati, OH.
5. ***"Thermal Desorption Applications and Techniques Using Adsorbent Resins"***, Santford Overton, Scientific Instrument Services, Inc., Ringoes, NJ.
6. ***"Direct Thermal Extraction Using Thermal Desorption - Extraction and Analysis Without the Use of Solvents"***, John J. Manura, Scientific Instrument Services, Inc., Ringoes, NJ.

FEATURE

How to Upgrade a Conventional HPLC for On-line Capillary LC/MS

Jean-Pierre Salzmann
Tech Support LC Packings

Capillary LC/MS has become a standard for many LC/MS applications interfacing with Electrospray or Continuous Flow FAB. Whenever the available sample amount is limited and/or the concentration of the sample is low, Capillary LC/MS can substantially increase the sensitivity.

Due to the low flow rates—typically between 1 and 10 µl/min—no post-column splitting is required: all of the column eluent is introduced into the mass spectrometer without any loss of sample. When combined with large volume injections—10 or 20 µl—the gain in sensitivity can be quite dramatic.

What is required

Almost any modern HPLC system—reciprocating or syringe—can be upgraded to cutting-edge Capillary LC.

LC Packings developed an upgrade kit that converts conventional HPLC pumps and UV detectors into reliable micro pumps and micro detectors, dedicated and perfectly suited for Capillary LC work.

Superior separations can be achieved within a few hours without the need of purchasing new hardware! The upgrade can be easily removed if required. The upgrade kit consists of the following parts:

- Acurate™, micro flow processor
- Fusica™, packed capillary column
- U–Z View™, capillary flow cell

Optionally a micro switching device, µ-Dumper™, can be installed on-line after the UV-detector and before the MS interface.

Acurate Microflow Processor

Until now, one of the major limitations in Capillary LC was the lack of reproducible microgradients.

Although pumps that allow micro gradient elution are commercially available, the reproducibility and accuracy when operating at capillary flow rates are not satisfactory.

Fig 1: Acurate microflow processor with outlet for connection to injector and outlet to waste in gradient mode or for recycling in isocratic mode

Mixing flow rates as small as a few µl/min or less are not only a necessity for running microgradients in Capillary LC but also extremely difficult. The mixing chamber should have a volume in the sub-microliter range in order to reduce the delay volume (dwell-volume) and to minimize gradient dispersion.

Further, the mixing device should provide an homogeneous mixing for any type of solvent and solvent composition used. Mixing chambers which fulfill these requirements simply do not exist. Another drawback of using syringe pumps for the delivery of microgradients is flow equilibration: A time consuming process strongly affected by the solvents' compressibility, viscosity and variations in temperature.

An easy and proven solution

Today, with the advent of reliable pulse-free reciprocating pumps, the use of pre-injection split flow techniques is an attractive alternative for the delivery of microflows.

Acurate, developed and manufactured by LC Packings, is the first flow rate processor capable of generating highly accurate and reproducible microflows in the range of 1-10 µl/min under isocratic and gradient conditions. The device consists of a static micromixer and microsplitter. With a dead volume of ≤ 4.8 µl for the micromixer, delay volume and gradient dispersion can be almost neglected. For conventional pump(s) the split ratio is fixed to ca. 1:70 and for syringe pumps to ca. 1:30.

Furthermore, Acurate compensates for most of the viscosity changes during gradient elution and, hence, stabilizes the baseline noise (streamlining), and keeps the split ratio perfectly linear independent of the mobile phase composition. Built-in high pressure in-line filters protect the Acurate from particles generated by the pump(s).

Fusica Packed Capillary Columns

LC Packings provides an extensive series of packed capillary columns with inner diameters of 180 µm, 320 µm and 800 µm in lengths of 5, 15 and 25 cm. and with a wide selection of stationary phases. Efficiencies of more than 100,000 plates can be obtained.

Besides the standard reversed phase and many specialty chemistries, LC Packings offers in cooperation with PerSeptive Biosystems of Cambridge, MA, Capillary Perfusion Chromatography® columns. Advantages offered by these Fusica™-Poros® columns have been presented at the recent 41st annual ASMS meeting in San Francisco and in a publication in "Techniques in Protein Chemistry IV," D. Kassel et al. Academic Press, R. Angeletti Editor, 1993, pages 55-64.

All Fusica columns are made of fused silica and are shielded by a stainless steel tubing to guarantee robustness and ease of use. The O.D. is 1/16 inch and allows for direct connection of the column into the injection valve, thereby minimizing the risk of extra column dispersion caused by

connecting tubings.

The reduction in the amount of column packing material is probably one of the most attractive features using Capillary LC . Column-to-column reproducibility, which has often been a problem due to batch–to–batch variations in silica gel or in the bonding synthesis, can now be avoided. Recovery is higher than in conventional columns due to the reduced surface of the sorbent.

Custom packing to guarantee identical selectivity

The scale down of a conventional HPLC method can often be somewhat challenging if the type of stationary phase packed into the micro column does not provide the same selectivity. For crucial separations the use of identical packings in capillary columns is recommended to avoid time-consuming method adaptation. To solve this problem, LC Packings provides custom packing for all of its Fusica columns.

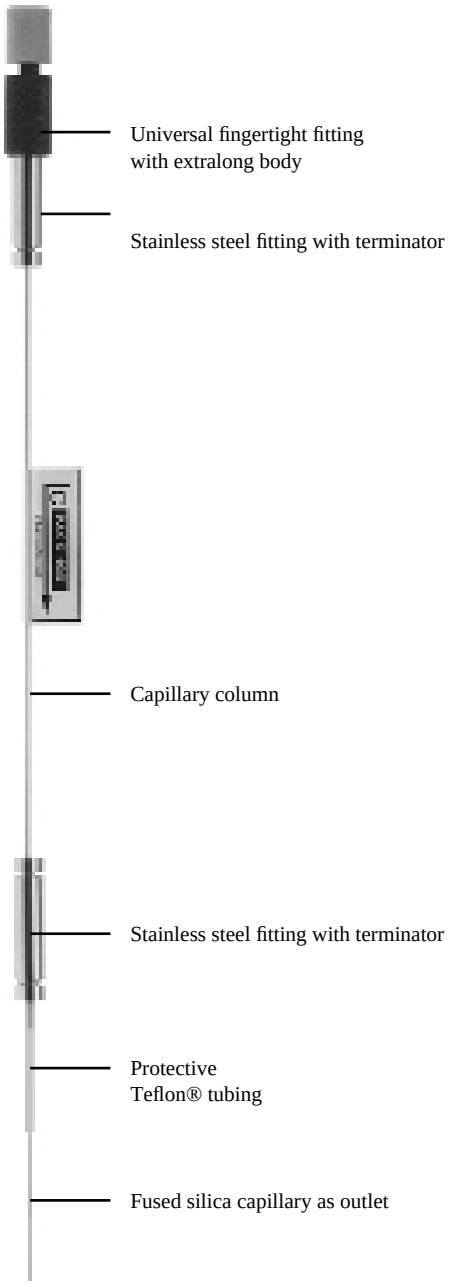


Fig.2: Fusica II column— all parts customized for optimum performance and longevity

U-Z View Capillary Flow Cell

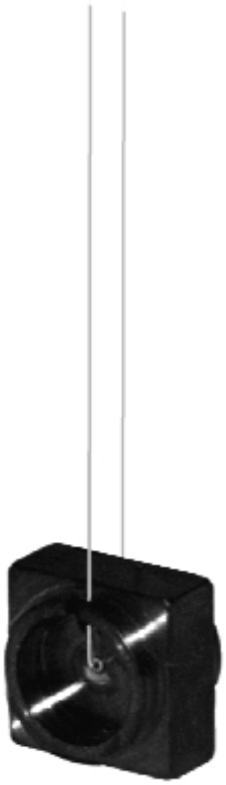


Fig. 3: U-Z View capillary flow cell with inlet and outlet capillary with U shape optical path

One of the excuses to legitimate post-column splitting of scarce sample was the idea that the split-off eluent can be directed to UV detection. With the LC Packings upgrade kit it is possible to perform on-line UV-detection directly prior to the MS interface. The UV detector almost functions as a window for monitoring the progress of the separation. This can be very helpful for trouble shooting.

Z-shape capillary flow cells with volumes ≤ 35 nl and path length of 8 mm are available for many of the most popular UV-detectors (see list). In contrast to on-column detection with its very short optical path length—typically the I.D. of the fused silica capillary—the bending of the capillary in a U or Z shape yields a path length of approximately 8 mm. According to Beer-Lambert's law this increases sensitivity S/N by a factor of 20 or more depending on the type of detector.

Large Volume Injections

Large volume injections are routine procedures in Capillary LC and result in dramatic increases in sensitivity. Large volume injections (volume overload) onto micro columns can be easily accomplished without penetration of the sample into the column bed and loss in efficiency. This technique consists of dissolving the sample in a weaker solvent than the mobile phase.

For 10 to 20 microliter injections, a difference of 20% in organic modifier is often sufficient to obtain compression and enrichment of the solutes at the top of the column - even under isocratic elution conditions.

For aqueous samples (e.g., tryptic digests of proteins and peptides), several hundred microliters can be injected onto the micro column under gradient elution conditions. The mobile phase gradient is maintained at a highly aqueous composition until the sample loop is flushed and the sample loaded onto the column. Once the sample is trapped on top of the column, the gradient is started.

Installation without modification

The U-Z View capillary flow cells, made of fused silica, can be installed within seconds, without coating removal or capillary alignment. The tubing has the same inner and outer diameter as the outlet capillary of the Fusica column, so the connection can be made butt-to-butt with a Teflon® connector. The outlet capillary can be directly connected to the MS interface. When used with a post-column switching device for directing the microflows the connection is made with another Teflon connector.

µ-Dumper Micro Switching Device

µ -

Dumper is a miniaturized two way switching device that allows for selectively directing microflows of 1-10 µl/min without any peak dispersion or loss in chromatographic performance.

The unit is typically installed after the capillary column. Through the two ports of the µ-Dumper, the direction of the microflow can be switched either to the mass spectrometer or to waste. A second important function in LC/MS is heart cutting to allow for better spectra on peaks of interest which otherwise might be masked by a preceding peak.

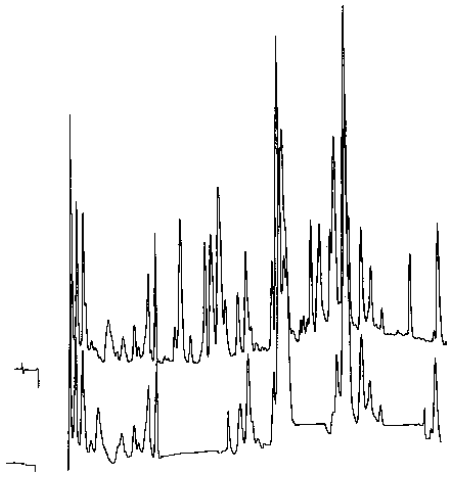


Fig. 4: Gradient separations of tryptic digest of hGH at 2 µl/min: Upper chromatogram without, lower chromatogram with use of µ-Dumperfor heart cuts

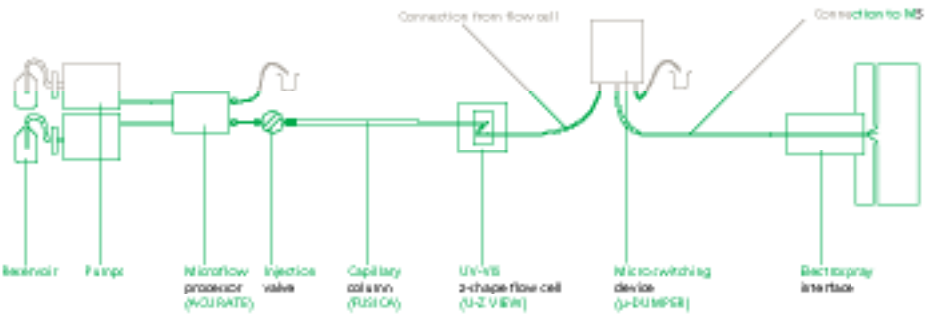


Fig. 5: Set-up of Capillary LC (high pressure mixing gradient) using upgrade kit including M-Dumper

Beyond LC/MS interfaces, other applications are coupling with secondary chromatographic systems (two-dimensional chromatography, micro fractionation, micro blotting, etc.)

The extremely small dead volume of the µ-Dumper perfectly maintains the integrity of the micro separation even at flow rates < 0.5 µl/min.

The use of the device is fully automated and the flow switching can be controlled manually or automatically via a data system contact closure.

Flow diagram of micro switching valve set to MS

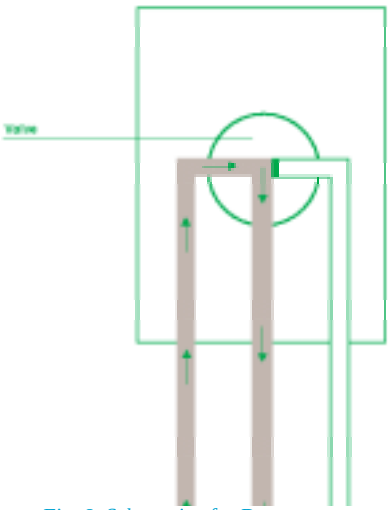


Fig. 6: Schematic of µ-Dumper set to waste

Installation

The upgrade kit is installed within minutes and fully preserves the integrity of the conventional HPLC. As most users will work with large volume injections conventional injection valves or autosamplers can be used without any modification. When used with a high pressure mixing gradient system it is recommended to bypass the dynamic or static mixer of the system. Instead the pumps are directly connected to the 1/16" Valco ports of the built-in high pressure filters of the Acurate and the microflow processor is directly connected to the injector (lines provided with device).

Conclusion

The upgrade of a conventional HPLC is readily feasible and results in performance that surpasses in accuracy, reproducibility, baseline stability, sensitivity and linearity any commercially available Micro or Capillary LC system. The cost for the upgrade depending on the system, ranges between $4,565 and $9,665 for an upgrade kit with a micro switching device.

HPLC Capillary Upgrade Accessories

Fusica™-Packed Capillary Columns by LC Packings

Fusica™-packed capillary columns are offered in three different diameters (180, 320 and 800µm) and in lengths of 5, 15, and 25 cm with a wide variety of stationary phases.

Listed by ID the following 3 column types are available:

180µm I.D. Pico Series: ideal for Electrospray

320µm I.D. Analytical: the workhorse standard in capillary LC (2-10ul/min). Available in high-pressure configurations for LC or SFC. A coiled 1 meter column, is also available when highest resolution is required.

800um I.D. Microbore/Microprep: Flow rates 10-100µl/min. Ideal for micro HPLC.

Efficiencies of more than 100,000 plates can be obtained.

For complete listing of columns see SIS catalog pages 344 - 351.

µ-Dumper™ Micro Switching Interface

Two-way switching device for directing microflows or for heart cutting, dead volume <5.0 nl. Can be installed on line after the UV-detector and before the MS Interface

Part #	Description	Price ea.
DP-20	µ-Dumper	$3850.00

Accurate Microflow Processor by LC Packings

- Convert ml/min to µl/min flow rates
- Highly reproducible micro flows (±0.2%CV)
- Virtually no dead volume (<4.8 ul)
- Highly reproducible micro gradients

Accurate™ - Microflow processor with built-in static mixer and flow splitter. Converts flows of conventional pumps into highly reproducible microflows (1-10ul/min.) There are gradient and isocratic versions.

Part #	Description	Price ea
AC-30*	Gradient version for syringe pumps, splitting ratio ca 1:30, with accessories	$2975.00
AC-70*	Gradient version for piston pumps, splitting ratio ca 1:70, with accessories	$2975.00
AC-00*	Gradient version with custom splitting ratio please specify	$3250.00
IC30*	Isocratic version for syringe pumps, splitting ratio ca 1:30, with accessories	$1975.00
IC-70*	Isocratic version for piston pumps, splitting ratio ca 1:70, with accessories	$1975.00
IC-00*	Isocratic version with custom splitting ratio, please specify	$2250.00

**Please specify fitting by adding the applicable suffix for the injector to the cat.no (e.g, AC-30V): V for Valco, R for Rheodyne, O for other, please specify*

U-Z View Capillary Flow Cells

- Illuminated volume ≤ 90nl
- Path Length up to 20 mm
- Ultra-sensitive: up to 100 times higher S/N
- Virtually no dead volume
- Pressure rating up to 500 bar (7000 psi)

The new capillary flow cells of LC Packings have been specifically designed to achieve the highest UV sensitivity in microseparation techniques such as Capillary LC and Capillary SFC.

The unique "U" or "Z" shape of the flow cell and the extremely small cell Volume result in virtually no dead volume and guarantee the maintenance of excellent chromatographic resolution.

Design and Sensitivity

In contrast to on-column detection, with its very short path length-typically determined by the I.D. of the fused silica capillary, the bending of the capillary into a "U" or "Z" configuration (longitudinal alignment) yields path lengths of up to 20 millimeters. This significant increase boosts the sensitivity in accordance with Beer-Lambert's law by a factor of nearly 100.

Because the entire flow cell consists of one single piece of fused silica capillary, dead volumes are minimized and are comparable to those of a 3 nl capillary flow cell (on-column). For low wavelength detection, e.g. proteins/peptides (<215nm), flow cells with 3mm path length are recommended to compensate UV cutoff.

Part No.	Description	Price ea.
UZ-AB	Flow cell for ABI/Kratos UV-detectors, e.g. 757, 783, 785, path length 8mm	$1850.00
UZ-LI	Flow cell for Linear 200 series UV-detectors and OEM products path length 8mm	$1975.00
UZ-HI	Flow cell for Hitachi 4200, 4250 UV-detectors, path length 14mm	$1975.00
UZ-WA	Flow cell for Waters 440 series UV-detectors, path length 8mm	$1975.00
CE-AB	CE column I.D. 75µm with built-in flow cell for ABI systems, path length 3.5mm	$785.00
CE-WA	CE column I.D. 75µm with built-in flow cell for Waters Quanta, path length 3.5mm	$785.00

Scientific Instrument Services, Inc. 908-788-5550

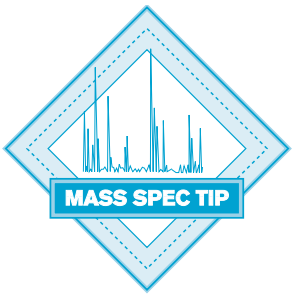
MASS SPEC TIPS

FROM SCIENTIFIC INSTRUMENT SERVICES

Mass Spec Tips is a forum for the exchange of ideas on the operation and maintenance of mass spectrometers, methods and techniques for sample handling, and ideas for unique problem solving. The use of mass spectrometer computer software as well as its modification can be included and Macros that you have written for particular applications can also be included. Over the many years that mass spectrometers have been utilized, many problems have been encountered and solved by numerous operators only to have the same problem reoccur for another operator. Now is your chance to share your ideas and suggestions, with other users.

If you have any ideas, tips or suggestions please give us a call or drop us a note to have your input included in this new forum. In order for "Mass Spec Tips" to flourish, we need your input, so please give us a call. Authors names and affiliations are listed at your discretion. S.I.S. reserves the right to select or reject ideas for publication in this section.

All Authors will be compensated $50.00 for each "Mass Spec Tip" published in this newsletter. In addition we will be selecting one person each year at the ASMS meeting from the contributors to both "Mass Spec Tips" and S.I.S. feature articles to receive a free 17" color TV or gift certificate. For each tip or article published in this newsletter between consecutive ASMS meetings, the author will receive one chance at this yearly drawing.



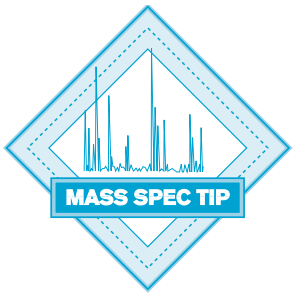
(1)
Freon for use in Mass Spectrometer Leak Checking

Author: John J. Manura
Affiliation: Scientific Instrument Services

Many mass spectroscopists have routinely utilized Freon (Dichlorodifluormethane) for locating mass spectrometer vacuum leaks around flanges, feedthroughs, probe inlet seals, diffusion pumps and other seals on the mass spectrometer vacuum system. The product was convenient to use since it was supplied in a small compressed air can with a nozzle (DustOff by Falcon Products). The gas could easily be sprayed around a suspected leaking fitting while the mass spectrometer was scanned for the predominant ion in the Dichlorodifluoromethane spectra. If a fitting was leaking, the Freon would produce a mass spec ion of 85 M/e in the EI mode. In the last two years, the availability of Dichlorodifluoromethane has been discontinued by all manufacturers in small compressed gas cans due to its ozone depleting properties. As a result other products will need to be utilized for mass spectrometer leak checking.

A similar product, Monochlorodifluoromethane, sold under the same name of Dust-Off is available from Scientific Instrument Services (part # FGB). This product can be used in the same manner as the previous product, however the mass spectrometer should be scanned for the 51 M/e ion in the spectrum. Monochlorodifluoromethane (M.W. = 86) produces a major peak at 51 (100%) with minor peaks at 67 (15%) and 31 (17%) when the mass spectrometer is scanned in the EI mode. However it is expected that sometime soon this product will also be eliminated because it too is a fluorocarbon and may have ozone depleting properties much like its predecessor Dichlorodifluoromethane.

Perhaps the best product to use for leak checking the flanges on the mass spectrometer is Argon gas. Argon (M.W. = 40) produces a predominant molecular ion at M/e 40 when scanned in the EI mode. Argon can be purchased in larger gas cylinders from your local gas supplier. To this tank you will need a two stage regulator and a length of flexible 1/8" tubing for directing the gas flow to the area to be leak checked. This product will not have the harmful environmental effects that the previous fluorocarbons have on the environment.



(2)
Mass Spectrometer Probe Cooling.

Author: John J. Manura
Affiliation: Scientific Instrument Services

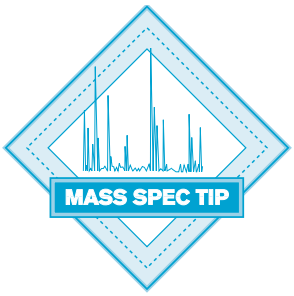
The probe shafts on many mass spec probes can become excessively hot when the probes are used. Many probe tips reach temperatures in excess of 500° C. In addition the probe tip and shaft can be further heated via thermal conductivity from the mass spectrometer source to which it is mated. When the probe is removed from the source, if the probe shaft is in excess of 200°C the seals of the probe inlet system can be damaged. Many mass spectroscopists have used the same Freon products for cooling the direct probes on mass spectrometers. By inverting the Dust-Off can upside down, the liquid Freon is projected from the compressed gas canister and is directed into the cooling line of the direct probe. The new Dust-Off can be used in the same manner as the old product for this purpose. The Monochlorodifluoromethane has similar cooling properties as the Dichlorodifluoromethane product when used in this manner. However this new Dust-Off may have some undesirable environmental consequences, and its availability may be limited in the future. Other, safer, products should be found and used to provide cooling of the mass spectrometer probes.

In our laboratory, we normally use compressed air for cooling all of our

probes that we manufacture and use in our applications laboratory. The compressed air source can be set to 20 to 40 psi and the air line from this compressed air source attached via leak tight fittings (such as Swagelok fittings) to the gas fitting on the mass spectrometer probe. In the probes that we design at S.I.S. we have incorporated a heat exchanger chamber in the tip of the probe which works very well using compressed air for cooling. We even use compressed air to cool our new High Temperature Probe. In this probe the probe tip can reach 800°C. Compressed air is passed through the probe during the entire heating cycle which maintains the shaft temperature to less than 100°C.

If faster cooling is required, Liquid CO_2 could be used but is not normally recommended for cooling the direct probes. The liquid CO_2 should be attached to the probe inlet via the same type of leak tight fittings mentioned above. In addition, the user may wish to vent the exhaust of the probe via a second line out of the laboratory to eliminate the possibility of excessive levels of the gas into the laboratory atmosphere. Extreme care must be exercised when using CO_2 in this manner for probe cooling due to the low temperatures and pressures involved. Flexible plastic or nylon gas lines cannot be used. Stainless steel lines are recommended and all fittings must be securely fastened. The design of the probe may not permit the use of liquid CO_2 . If plastic interconnecting tubing is used inside the probe handle, these lines could easily come undone when CO_2 is delivered to the probe. Due to these problems and hazards, liquid CO_2 is not normally used for cooling mass spectrometer probes.

Liquid cooling circulators have been used by some manufacturers for cooling mass spectrometer probes. This unit consists of a small pump which continually recirculates a cooled fluid through the mass spectrometer probe. They do a good job of cooling the probe, but cannot be used with most manufacturers' probe designs. Most manufacturers' of probes have designed their probes for gas cooling only. Liquids cannot be used because the insulated thermocouple and heater wires would become saturated with the liquid and result in electrical shorts to the probe heater and temperature sensing circuits. We have designed all our cooling probes at S.I.S. with a heat exchanger chamber in the probe tip and a completely isolated cooling system. Therefore these probes can be used with both gases as well as liquids for cooling purposes since none of the electrical wires would be exposed to the cooling liquids. We presently do not have a source for a small liquid cooling circulator. However we are trying to find one, and if not available plan on manufacturing one in the near future.



(3)
Sample Vials for Direct Probes.

Author: Christopher Baker
Affiliation: Scientific Instrument Services

Sample vials for use in direct probes have been made of a variety of materials including aluminum, gold, quartz and Pyrex glass. Quartz, gold and aluminum vials are fairly expensive and therefore many users elect to reuse the vials after cleaning between samples. This is time consuming and care must be taken to insure that the vials are completely clean to eliminate cross contamination.

Glass has been widely used for sample vials because the vials are inexpensive enough to make them disposable. The glass vials, however, can be difficult to use because they are fragile and are susceptible to breaking when inserting or removing the vials from the probe. The small diameters of straight sample vials also makes loading a sample troublesome.

Flared glass sample vials provide advantages over the standard straight glass sample vials. The flared end of the vial makes loading a liquid sample much easier since the flare helps guide the syringe needle into the vial. The flared end also makes the vial much stronger so that vials can easily be inserted and removed from the end of the probe using tweezers without breaking the glass. Flared vials have been used on most Finnigan MAT instruments for many years, however flared vials of various diameters can be manufactured by S.I.S. for use on other instruments providing glass sample vials which are stronger and make sample loading much easier.

If anyone else has any further suggestion on other methods of leak checking or probe cooling, we would like to hear from you. I am sure that there are other methods of solving both of these problems.

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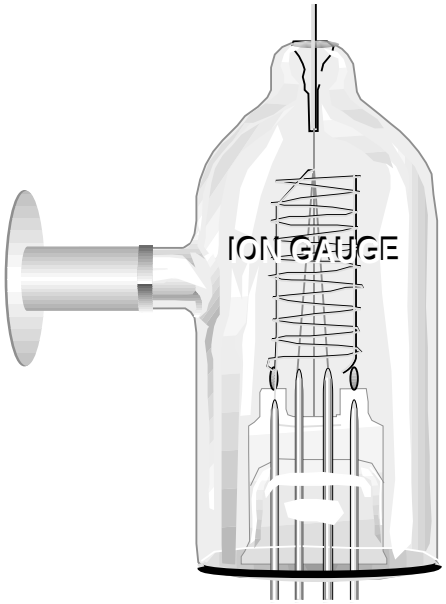
For more information refer to pg 245 of our catalog.

FEATURED PRODUCTS

FROM SCIENTIFIC INSTRUMENT SERVICES

Ionization Gauge

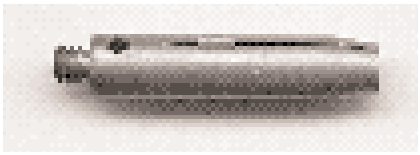


for HP 5989

The ionization gauge tubes used on the Hewlett-Packard 5989 MS Engine mass spectrometers are available from SIS. These ionization gauge tubes have the quick flange on the side arm for installation into the mass spec system. They are direct replacements for the HP originals (HP# 0960-0799).

Part#	Description	Price ea.
G100KF25	Ion gauge tube for HP 5989	$160.00

Replate Gold on Probe Tip

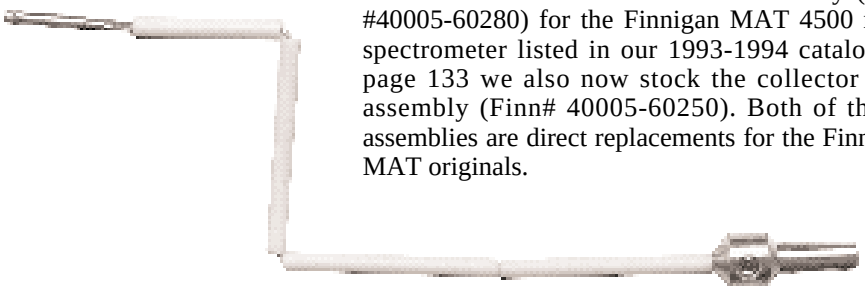


The gold plated probe tips used with the Hewlett-Packard direct probes can be cleaned and replated with gold if they become dirty. The old coat is completely removed and a new coat is applied.

Part #	Description	Price Ea.
REP120	Recoat Gold Tip	$65.00

Collector Lead for Finnigan MAT 4500

FL1



FL2

In addition to the filament lead assembly (Finn #40005-60280) for the Finnigan MAT 4500 mass spectrometer listed in our 1993-1994 catalog on page 133 we also now stock the collector lead assembly (Finn# 40005-60250). Both of theses assemblies are direct replacements for the Finnigan MAT originals.

Part #	Description	Price ea.
FL1	Filament Lead Assembly	$15.00
FL2	Collector Lead Assembly	$25.00

O'Rings for Varian Saturn GC/MS

Viton O'rings for the Varian Saturn GC/MS are available from stock. Call us if you need a part number or size not listed.

Part #	Varian#	Description	Price ea.
V120	27-402361-00	3/32 x 1.0 O'Ring	$1.05
V239	27-402884-00	1/8 x 1.0 O'Ring	$4.63
V413	27-402883-00	1/4 x 1.0 O'Ring	$12.50

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SIS is pleased to offer individually calibrated and certified syringes on request. Many laboratories are obligated by ISO and GLP protocol to use apparatus, including syringes, which are traceable to International Standards. We can supply syringes which are individually calibrated and are shipped with a certificate listing their displacement volume conformance. You must use the part numbers listed below when ordering a syringe. These part numbers are in addition to the standard syringe part number. This service is available on most of the syringes we supply which are manufactured by SGE. Please allow 4 weeks for delivery of calibrated syringes. If you have any questions or require further tests please give us a call.

Part#	Description	Price ea.
880243	Calibration and certificate for one syringe	$75.00
880244	Calibration and certificates for a six pack	$200.00

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A Compilation of Articles from
"The Mass Spec Source"
1983-1993

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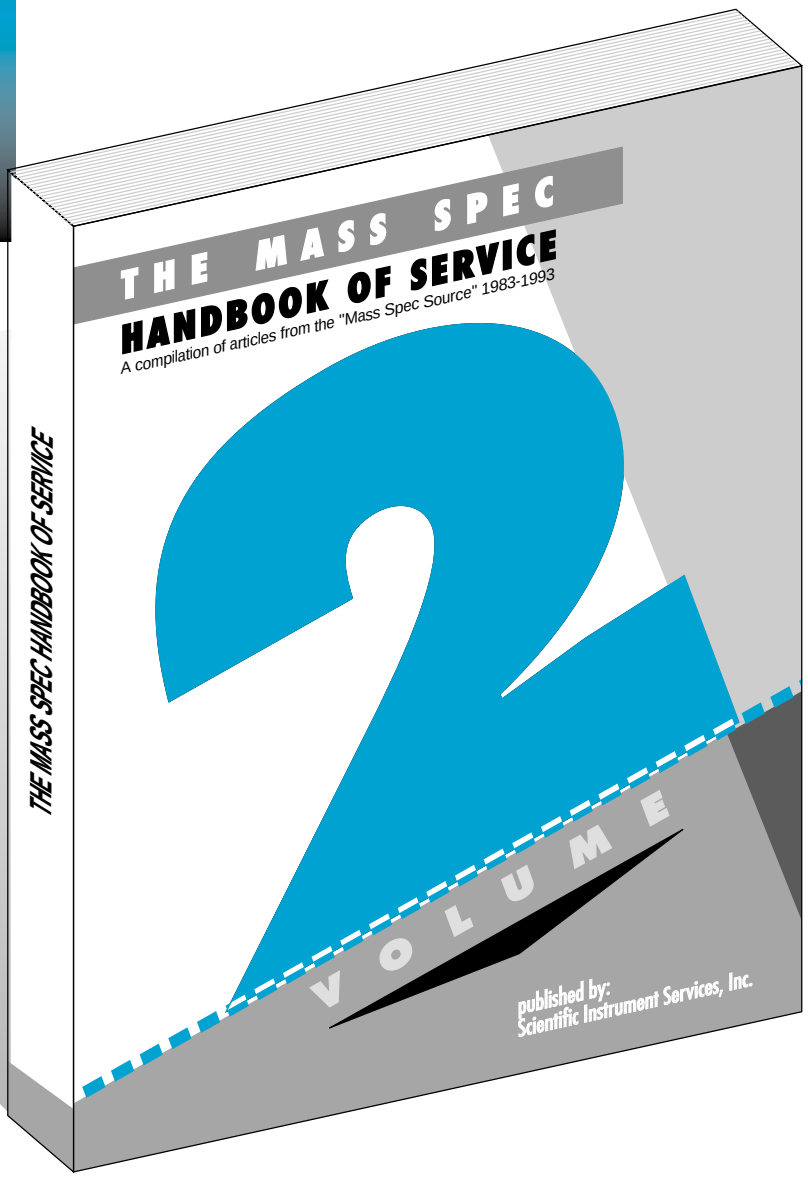

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