The Mass Spec Source

VOL. XVIII, NO. 2 MARCH 1994

For Users of Mass Spectrometers and Gas Chromatographs

FEATURES

CONTENTS

PITTCON '94 *A Preview*

Scientific Instrument Services will once again be exhibiting at the Pittsburgh Conference. This year's conference will be held in Chicago the week of February 28 through March 3, 1994. We invite you to stop by our booth # 1749 in the East Hall of the convention center. We would be happy to discuss any of our products with you. If you have any suggestions or concerns about our supplies and services, we are always happy to hear from you. We will be presenting 5 poster presentations at this meeting in addition to the new and existing products we supply to the mass spec and GC communities. Please stop by and see us.

Scientific Instrument Services at PITTCON '94
Booth #1749 in East Hall

S.I.S. Presentations at PITTCON '94

S.I.S. will be presenting a number of presentations at this meeting describing mass spec probes, the GC Cryo-Trap as well as applications of the Thermal Desorption System. The poster presentation on the Tenax Resins will demonstrate the use of breakthrough volume data for a wide range of organic compounds on both Tenax TA and Tenax GR adsorbent resins for use in P&T applications of these resins. We would

Presentation Schedule

Sunday, - February 27 (12:00 noon to 5:00 p.m.) **East Hall - Level 2**
(040P) A New High Temperature Probe for a Mass Spectrometer - C. Baker, S.I.S.

(042P) Automated GC Cryo-Trap/Heater for On-column Trapping of Organics in GC Thermal Desorption and Head Space Analysis - J. Manura, S.I.S.

Monday, February 28 (9:30 - 11:00 a.m.) **East Hall - Level 2**
(040P) A New High Temperature Probe for a Mass Spectrometer - C. Baker, S.I.S.

(042P) Automated GC Cryo-Trap/Heater for On-Column Trapping of Organics in GC Thermal Desorption and Head Space Analysis - J. Manura, S.I.S.

(089P) Analysis of Black Electrical Tapes by Short Path Thermal Desorption and GC/MS - T.A. Brettell, N.J. State Police

Monday, February 28 (9:30 - 11:00 a.m.) **North Hall - Level 2**
(178P) Adsorption and Desorption Characteristics of Tenax Adsorbent Resins for Thermal Desorption Applications - J. Manura, S.I.S.

Monday, February 28 (1:30 - 5:00 p.m.) **North Hall - Room M3**
(221P) Identification and Quantification of Residual Solvents in Pharmaceuticals Via Direct Thermal Extraction using Thermal Desorption GC/MS techniques - J. Manura, S.I.S.

Tuesday, March 1 (1:30 - 5:00 p.m.) **North Hall - Level 2**
(396P) Evaluation of Cooking Oils by Thermal Desorption - S.V. Overton, S.I.S.

be glad to meet and talk with you at the following presentations:

New Products-*GC Cryo-Trap*

We will be introducing our new GC Cryo-Trap at this year's conference. The GC Cryo-Trap permits the trapping of volatiles and semi-volatiles at the head of the GC column utilizing a liquid CO_2 cooled trap. The trap also contains a heater to rapidly elute the trapped volatiles off the trap when ready for chromatography. This Cryo-Trap has a wide range of applications including Thermal Desorption Trapping, GC head space trapping, trapping of samples via direct injection and also multidimensional GC systems. The GC Cryo-Trap permits the trapping of volatiles and semi-volatiles at the front of the GC column utilizing less than 10% of the CO_2 which would be required to cool the entire GC oven. It also permits the cryo trapping of volatiles at temperatures down to -70°C using liquid CO_2. A dual temperature controller permits the setting of cooling temperatures down to -70°C for cryo cooling and heating temperatures up to 400°C for the release of trapped volatiles. Our poster on Sunday and Monday at the manufacturers poster sessions will demonstrate the usefulness of the GC Cryo-Trap in both thermal desorption and GC head space applications.

New Thermal Desorption System - *Model TD3*

Also being introduced at this year's show will be the new Model TD-3 of the Short Path Thermal Desorption System. Several changes and additions were made to our previous model TD-2 Thermal Desorption System. These include the control of the GC Cryo-Trap cooling and heating from the desorption electonics control and the addition of a programmable temperature ramp for the desorption system blocks. A new four line display panel shows the complete system status and permits the user to change all system parameters including desorption and sampling timers and both cryo trapping and thermal desorption temperatures. Both the new model TD-3 and the model TD-2 will be on display at our booth.

Mass Spectrometer Probes

Over the last few years, S.I.S. has developed a large variety of probes for mass spectrometers. These include direct probes, direct exposure probes (DEP or DCI), high temperature probes, pyrolysis probes and thermospray probes. Our new high temperature direct probes were developed as a result of several customer inquiries from last year's show. This high temperature direct probe permits the analysis of samples up to 800°C. As always if you have suggestions as to types of probes that are required or if you have a need for a custom designed mass spec probe, we would be glad to help you. Also on display will be our temperature controllers for all these mass spec probes. A new microprocessor controller has just been introduced for the Direct Exposure Probes. This controller permits the heating of the DEP filament wire with a programmable ramp of the wire current.

FOR SALE

Finnigan Incos 50 MS with Varian 3400 GC and Tekmar LSC2000 Purge & Trap. Data General Data System, Printronix Printer. Many spare parts. Excellent condition. Price Negotiable. Contact: Connie Stier, RCP, Inc. 405 N Hamilton Rd, Columbus Ohio (614) 864-6123

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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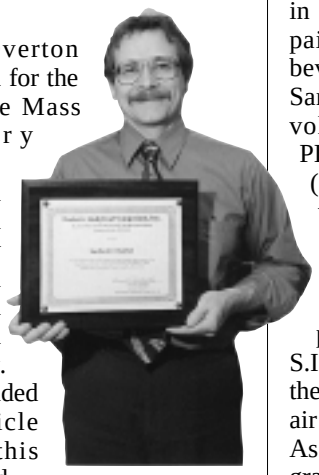
FEATURE

Posters at Eastern Analytical Symposium

Best Poster Presentation in Both Gas Chromatography and Mass Spectrometry

by John J. Manura
Scientific Instrument Services, Inc., 1027 Old York Road, Ringoes, NJ 08551

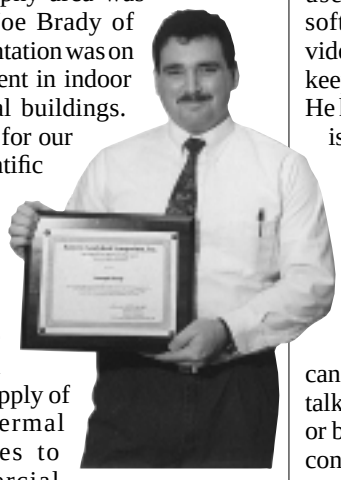
This year's Eastern Analytical Symposium in Somerset, N.J. in November of 1993 announced awards in 5 categories for outstanding poster presentations. The purpose of these awards was to recognize outstanding contributions to the Symposium Poster Sessions as well as improve the quality and professionalism of the presentations. The five categories for the awards were (1) Mass Spectrometry, (2) Gas Chromatography, (3) Infrared Spectroscopy, (4) Liquid Chromatography and (5) NMR Spectroscopy.



Santford V. Overton

Sandy Overton received the award for the best poster in the Mass Spectrometry category. Sandy's presentation was on the volatiles in mushrooms. Sandy is interested in mushrooms both professionally and as a personal hobby. His poster is expanded upon in the article located in this newsletter. Sandy has been looking at the volatiles present in edible mushrooms. This work was completed using the S.I.S. Purge and Trap system to purge the volatiles from the heated mushrooms and collect the volatiles onto a Tenax Trap. The volatiles were then thermally desorbed into the GC for analysis using the Short Path Thermal Desorption System. An additional presentation is presently being developed on the mushroom studies for the Fifth International Mycological meeting in Vancouver, British Columbia, Canada scheduled for the week of August 14 - 21, 1994.

Sandy has been with S.I.S. for almost 5 years in the position of Technical Sales Manager. His main responsibilities include conducting research utilizing our Short Path Thermal Desorption and Mass Spectrometer Systems as well as customer support and applications development for customers using the products we manufacture and market. Sandy received his Ph.D. in Plant Pathology from Virginia Polytechnic Institute in Blacksburg, Virginia,and is presently working on a collaborative program with them on volatiles in honey. Sandy is conducting research in a number of areas, which are mainly guided by customer ideas and problems. These include volatiles in honey, cooking oils, mushrooms, latex paints, synthetic polymers, commercial beverages, colognes, perfumes and fruit. Sandy is preparing a presentation on the volatiles in cooking oils for this years PITTCON meeting and two presentations (one on volatiles in paints and one on volatiles in colognes) for this years ASMS meeting.



Joseph Brady

The award in the Gas Chromatography area was presented to Joe Brady of S.I.S. Joe's presentation was on the volatiles present in indoor air in commercial buildings. As our Salesman for our graphic and scientific product lines, Joe spends a good deal of time traveling and at trade shows. During this time he took a personal air pump and a supply of conditioned thermal desorption tubes to various commercial locations including a variety of ethnic restaurants, convention centers, hotels, airplanes and even our hospitality suite in San Francisco last year. He then worked in conjunction with Sandy to analyze these samples to determine the nature and relative amounts of volatiles present in these air samples. An article on this data is scheduled for a future newsletter. Some interesting results were readily apparent. The compounds limonene and BHT (Butylated Hydroxy Toluene) were very prevalent in many of these samples. Limonene is widely used in most cleaning type products and BHT is a widely used antioxidant in plastics.

Joe has his B.S. degree from Rutgers University in Chemistry. Joe has been with S.I.S. for ten years. Since he has a background in Chemistry and his hobbies included photography and art, he was originally hired to direct the production of our catalogs and advertising. Joe was the reason that you saw a major improvement in our catalogs eight years ago. He is presently a salesman for S.I.S. on the graphic art line that we market to the local professional market. These products include color printers, scanners and slide making systems. He is involved in marketing and training users in the use of both hardware and software for the production of computer video presentations. In addition Joe does keep his hand in the scientific product line. He helps us out at our trade shows. Since Joe is fluent in the German language, he will represent S.I.S. at Analyticia in Germany this year. He will be helping Peter Weibel of MS Wil, who is our dealer in Switzerland, with the Short Path Thermal Desorption System and our other products at this show.

As always, if any member of our staff can be of service to you, we would be glad to talk to you either at the trade shows we attend or by phone or letter. We thank you for your continued support.

FEATURE

Determination of Volatile Organic Compounds in Mushrooms

by Santford V. Overton
Scientific Instrument Services, Inc., 1027 Old York Road, Ringoes, NJ 08551

Introduction

Certain kinds of mushrooms are considered a delicacy throughout the world. The flavor qualities are greatly dependent on the numerous volatile and semi-volatile compounds (VOC's) contained within the mushroom complex. However, others are poisonous and can be very detrimental to one's health. In addition, mushrooms have been, and are currently being investigated as sources of natural products for new and better flavors as well as those which may be used by the pharmaceutical industry in the development of life saving drugs. Analytical techniques are needed to identify and quantitate VOC's present in mushrooms so that these techniques can be used to analyze the components which are responsible for flavor and off-flavors as well as identify compounds which may be toxic or unique to the species of mushroom. Volatile organic compounds were collected from several kinds of edible mushrooms and analyzed by using a purge and trap technique (P&T), followed by trapping on an adsorbent resin and subsequent analysis by thermal desorption-gas chromatography-mass spectrometry (TD-GC-MS).

Instrumentation

Samples were collected using a Scientific Instrument Services Purge & Trap System. This apparatus **(Fig. 1)** consists of a purge gas inlet connected to a stainless steel purging needle that is inserted through an adapter fitting into a 10 ml test tube. A dry purge gas inlet is located at a right angle to the purge gas inlet at the top of the apparatus **(Fig. 1)**. The purpose of the dry purge is to reduce the water vapor condensation on the adsorbent trap. This problem can be especially troublesome when isolating volatiles from high moisture samples at high temperatures. Although the adsorbent traps packed with Tenax have a low affinity for water it is inevitable that some water condensation will occur in the trap due to the high relative humidity of the purge gas as it exits the apparatus. When moisture condenses on the adsorbent resin it can block the pores of the resin matrix and thereby drastically reducing the trapping of volatile organics. Opposite the dry purge inlet is the connector for the glass-lined stainless steel (GLT) desorption tube containing the adsorbent resin **(Fig. 1)**. The Purge & Trap System also contains two ball rotameters with adjustable needle valves mounted on a stationary base and permits the visual indication and independent adjustment of the gas flow to the purge gas and dry purge inlets.

All experiments were conducted using a Scientific Instrument Services model TD-2 Short Path Thermal Desorption System accessory **(1&2)** connected to the injection port of an HP 5890 Series II GC interfaced to an HP 5971 Mass Selective Detector. The mass spectrometer was operated in the electron impact mode (EI) at 70eV and scanned from 35 to 400 daltons during the GC run for the total ion chromatogram.

A short 0.5 meter by 0.53 mm diameter deactivated fused silica precolumn was attached to the injection port end of a 60 meter x 0.25 mm i.d. DB-5MS capillary column containing a 0.25 µm film

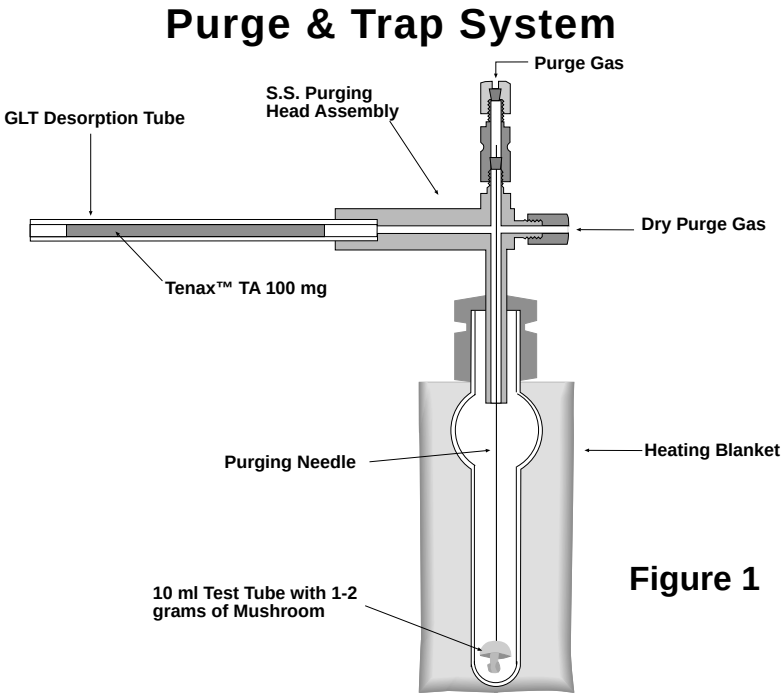


Figure 1

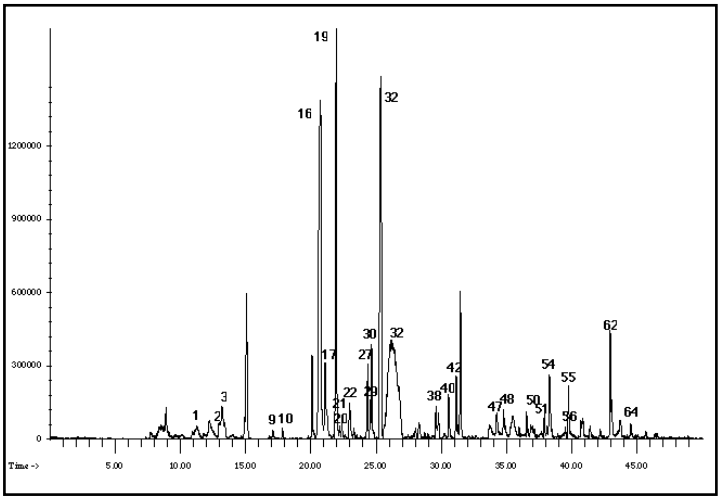


Figure 2. Shiitake Mushroom - 1.169 g. Collected for 35 min at 90°C at 20 ml/min with 25 ml/min dry purge. Thermally desorbed at 250°C for 5 min.

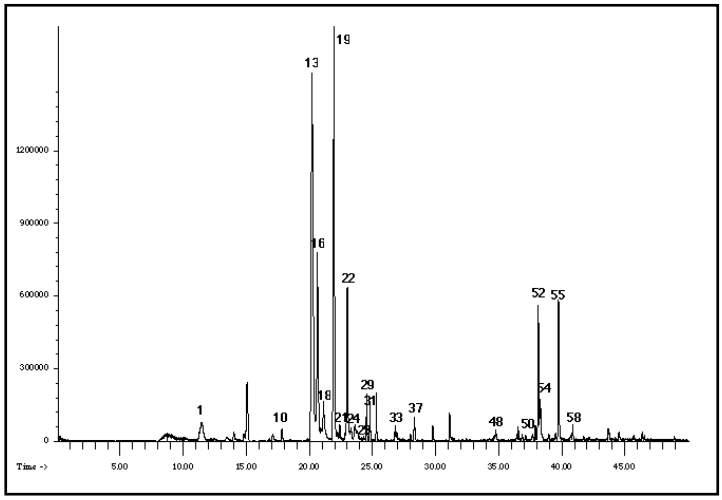


Figure 3. Portabella - 1.539 g. Collected for 35 min at 90°C at 20 ml/min with 25 ml/min dry purge. Thermally desorbed at 250°C for 5 min.

thickness. The GC injection port was set to 250°C and direct splitless analysis was used. The head of the column was maintained at -70°C using an S.I.S. Cryotrap model 951 during the desorption process in order to trap the volatiles at the front of the precolumn. The trap was then ballistically heated to 200°C at the end of the desorption process and the GC oven was temperature programmed from 35°C (hold for 5 minutes) to 80°C at a rate of 10°/min and to 280°C at 4°/min.

Experimental

Six different kinds of edible mushrooms (Shiitake, Portabella, Agaricus bisporus, Agaricus campestris, Lactarius & puffball Calvatia) were analyzed to compare and quantify the volatile organics contained within the mushroom complex. For quantification, a deuterated cymene internal standard was spiked into the adsorbent traps after the sample had been isolated. No correction for extraction efficiency of recovery is achieved using this technique; however, it serves as a useful means of quantifying the relative levels of components from the mushroom samples. **(3)**.

Samples sizes of 1-2 g of mushroom (pileus & stipe) were transferred into a 10 ml purge & trap tube and heated to 90°C. Samples were purged with high purity helium at 20 ml/min with an additional 25 ml/min dry purge using the S.I.S. Purge & Trap System. Volatile analytes were gas extracted and carried to a preconditioned 4.0 mm i.d. glass-lined stainless steel desorption tube packed with 100 mg of Tenax™TA. Once the samples were collected they were spiked with 200 ng of d-14 cymene internal standard by injecting 1 µl of a 200 ng/µl of a d-14 cymene stock solution in methanol by syringe injection into the Tenax matrix.

The desorption tube with sample and internal standard were then attached to the Short Path Thermal Desorption System and a syringe needle attached. The desorption tube was injected into the GC injection port and desorbed at a desorption block temperature of 250°C for 5 minutes. The desorbed volatiles were trapped at the front of the GC column and subsequently eluted through the GC column for detection and identification by the mass spectrometer.

Results and Discussion

Three cultivated mushrooms (Shiitake, Portabella, Agaricus bisporus) and three edible wild mushrooms (Agaricus campestris, Lactarius sp., Calvatia sp.) were analyzed to identify, compare and quantify the volatile organics present. **Table I** shows the VOC's detected and the relative amounts of these compounds in each mushroom. The mushrooms were found to contain numerous middle-chain aliphatic alcohols, aldehydes and ketones, which are believed to be the degradation products of fatty acids **(4)**. The compounds 3-methyl-butyraldehyde and 2,6 bis (1,1-dimethyl ethyl)-phenol were identified in each of the mushrooms **(Fig. 2-6)** with the exception of the giant puffball Calvatia. These compounds are thought to contribute to the characteristic aroma of the mushroom. Phenylacetaldehyde which appears to be an important flavor compound was present in each of the mushrooms except for A. bisporus **(Figs 2,3,5-7)**. The flavor compound benzaldehyde was also detected in Portabella as well as A. bisporus **(Figs. 3&4)**. Lactarius sp. contained a high concentration of the aliphatic compound 1-octen-3-ol **(Fig. 6)**. 3-Octanone was also found in the Shiitake, Portabella and A. bisporus mushrooms **(Figs. 3&4)**. The presence of 1-octen-3-ol and 3-octanone suggests that the activity of lipoxygenase and hydroperoxide lyase producing C_8 compounds from linoleic acid was stronger in these mushrooms **(4)**. 1-Methyl-1H-pyrrole which was detected in Shiitake was the only nitrogenous compound identified **(Fig. 2)**. 2-formylpyrrole has also been found in the pentane extract of dried Boletus edulis **(5)** and in liquid cultures of Polyporous tuberasters **(4)**. This nitrogen compound has been identified in cocoa and bread and may be produced by nonenzymatic browning reactions. In addition, the sulfur compounds 1,2,4-trithiolane, 1,2,4,6-tetrathiepane and dimethyl disulfide were identified in Shiitake **(Fig. 2)**. This suggests that they were produced by chemical reactions in which one reactant was an amino acid containing sulfur. The meadow mushroom A. campestris and the giant puffball Calvatia were found to contain high concentrations of phenol **(Fig. 5)** and methoxybenzene **(Fig. 7)**, respectively. Although similar to the button mushroom, A. bisporus, found at the grocery store, the meadow mushroom is far richer in flavor. It

Table I - Relative Amounts of Volatile Organics in Mushrooms (ng/g)

		Shiitake	*Portabella*	*A. bisporus*	*A. campestris*	*Lactarius*	*Calvatia*
1	3-methyl-Butyraldehyde	24.3	13.8	29.9	0.5	30.2	-
2	1-methyl-1H-Pyrrole	12.4	-	-	-	-	-
3	dimethyl - Disulfide	35.3	-	-	-	-	-
4	3-methyl-1-Butanol (impure)	-	-	-	13.7	-	-
5	Toluene	-	-	-	14.8	-	2.9
6	2,4-dimethyl - Heptane	-	-	-	-	-	3.5
7	Hexanal	-	-	-	-	32.0	-
8	1,3-Octadiene	-	-	31.9	-	-	-
9	ethyl benzene	6.9	-	-	-	-	-
10	Nonane	4.5	4.3	-	-	-	-
11	methoxybenzene	-	-	-	-	-	307.2
12	(E)-2- Heptenal	-	-	-	-	-	5.1
13	Benzaldehyde	-	192.6	59.4	-	-	-
14	Phenol	-	-	-	208.3	-	-
15	1-Octen-3-ol	-	-	-	-	265.2	-
16	3- Octanone	321.6	68.7	323.4	-	-	-
17	3- Octanol	69.9	-	45.2	11.7	12.5	-
18	Decane	-	9.2	-	-	-	-
19	d-Cymene			(Internal Standard)			
20	1-methyl-4-(1-methylethyl)-benzene	7.9	-	-	-	-	-
21	Limonene	10.2	3.7	-	5.2	-	4.5
22	Phenylacetaldehyde	24.5	48.5	-	13.1	12.7	49.4
23	(E)-2-Octenal	-	-	-	-	14.6	-
24	(E)-2-Octen-1-ol	-	10.0	-	5.0	145.7	-
25	1-phenyl-Ethanone	-	6.3	14.9	5.1	-	-
26	1 - Octanol	-	-	4.8	-	-	-
27	2-(1,1-dimethylethyl)-Phenol	33.2	-	-	-	-	-
28	2,4-dimethyl-Heptane	-	-	-	-	-	4.2
29	Undecane	12.6	10.3	-	-	-	-
30	3-(4-methyl-3-pentenyl)-Furan	35.3	-	-	-	-	-
31	Nonanal	-	9.3	-	-	5.8	10.0
32	1,2,4-Trithiolane	264.2	-	-	-	-	-
33	1-Dodecanol	-	4.6	-	-	-	-
34	Nonadecane	-	-	-	1.8	-	-
35	1-methylene-1H-Indene	-	-	-	2.2	-	-
36	Naphthalene	-	-	-	-	5.9	-
37	Decanal	-	8.5	-	-	-	-
38	Z-Citral	18.0	-	-	-	-	-
39	(E)-2-Decenal	-	-	-	-	-	6.5
40	Citral	25.6	-	-	-	-	-
41	3 - methyl-, butyl ester -Butanoic Acid	-	-	-	-	-	9.3
42	2-Undecanone	6.8	-	-	-	-	-
43	Tridecane	62.1	-	-	-	-	-
44	(E,E)-2,4-Octadienal	-	-	-	-	4.1	-
45	1,3-bis(1-methylethenyl)-Benzene	-	-	-	-	2.7	-
46	Trans, trans-nona-2,4-Dienal	-	-	-	-	20.7	19.0
47	2-ethyl-3-hydroxyhexyl Ester	15.0	-	-	-	-	-
48	Tetradecane	7.7	4.0	-	3.8	-	-
49	5,7-dimethyl-Undecane	-	-	-	-	-	5.4
50	Pentadecane	9.0	3.9	-	3.5	-	13.8
51	Farnesol	6.4	-	-	-	-	-
52	(Z,E)-alpha-Farnesene	-	33.1	-	-	-	-
53	alpha-Bisabolene	-	-	-	-	-	13.6
54	2,6-bis(1,1-dimethylethyl)-Phenol	37.7	16.9	20.3	10.3	5.7	-
55	Ionol 2	21.5	37.0	52.6	-	-	-
56	1,2,4,6-Tetrathiepane	10.7	-	-	-	-	-
57	3-methyl-1,4-Heptadiene	-	-	-	-	87.5	-
58	Hexadecane	-	4.8	-	6.0	-	10.1
59	1-chloro-6-Heptadecyne	-	-	-	-	23.4	-
60	3-Octadecyne	40.4	-	-	-	-	-
61	1,1'-methylenebis [4-methyl]-benzene	-	-	-	-	9.3	-
62	Pentatriacontane	12.4	-	-	-	-	-
63	Heptacosane	-	-	-	-	-	3.6
64	2,2'-diethyl-1,1'-Biphenyl	5.8	-	-	-	-	-
65	alpha - Ginensal	-	-	-	-	8.3	-
66	cis-Farnesol	-	-	-	-	-	3.7
67	4,4'-(1,2-diethyl-1,2-etha-Phenol	-	-	-	-	19.0	-
68	1-methyl-3-[(4-methylphenyl)]-Benzene	-	-	-	-	12.4	-

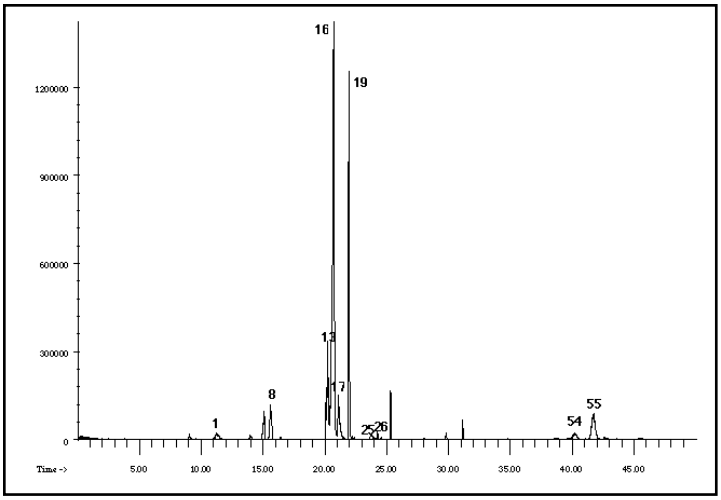


Figure 4. A. bisporus - 1.577 g. Collected for 35 min at 90°C at 20 ml/min with 25 ml/min dry purge. Thermally desorbed at 250°C for 5 min.

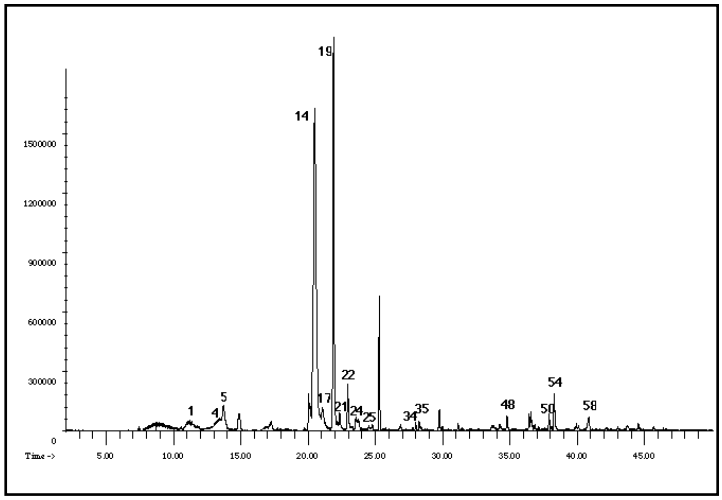


Figure 5. Lactarius - 1.903 g. Collected for 35 min at 90°C at 20 ml/min with 25 ml/min dry purge. Thermally desorbed at 250°C for 5 min.

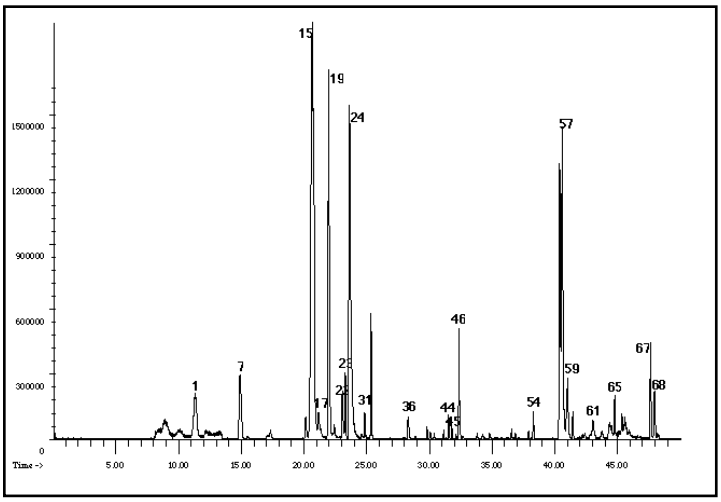


Figure 6. A. campestris - 1.906 g. Collected for 35 min at 90°C at 20 ml/min with 25 ml/min dry purge. Thermally desorbed at 250°C for 5 min.

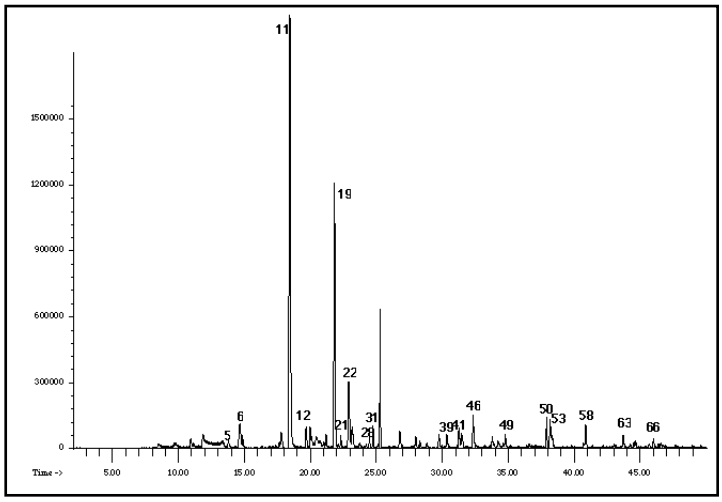


Figure 7. Calvatia - 1.417 g. Collected for 35 min at 90°C at 20 ml/min with 25 ml/min dry purge. Thermally desorbed at 250°C for 5 min.

is superior in any recipe that calls for its relatively bland cultivated brother. Both cultivated and edible wild mushrooms possessed many common compounds although each had its own fingerprint chromatograph. However, trace amounts of the compound Ionol 2 were detected only in the cultivated mushrooms **(Figs. 2-4)**. The presence of this compound may be due to increased pigment degradation over time in the cultivated mushrooms compared to the freshly harvested edible wild mushrooms.

Conclusion

Many kinds of flavors are used in the food industry, and there is a demand for new and improved ones, especially natural ones. Such a source are mushrooms which can produce a variety of flavors. In addition, mushrooms are currently being investigated as sources of natural products which can be used by the pharmaceutical industry in the development of life saving drugs. The Short Path Thermal Desorption System used in conjunction with the Purge & Trap System permits the identification and quantification of trace levels of volatile organics responsible for flavors in mushrooms as well as those which are unique to the species of mushroom. These techniques present a tremendous improvement over the time-consuming solvent extraction techniques normally used in the laboratory, and can be easily incorporated in flavor studies, general QA/QC testing or to a lesser extent for screening of mushrooms for natural products for use in the pharmaceutical industry.

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2. Manura, J.J. and T.G. Hartman. 1992. Applications of a Short Path Thermal Desorption GC Accessory. American Laboratory. May: 46-52.

3. Methodologies for the Quantification of Purge and Trap Thermal Desorption and Direct Thermal Desorption Analyses. S.I.S. Application Note No. 9, September 1991.

4. Kawabe, T. and H. Morita. 1993. Volatile Components in Culture Fluid of Polyporus tuberaster. J. Agric. Food Chem. Vol. 41 (4): 637-640.

5. Thomas, A.F. 1973. An Analysis of the Flavor of the Dried Mushroom, Boletus edulis. J. Agric. Food. Chem. 21: 955-958.

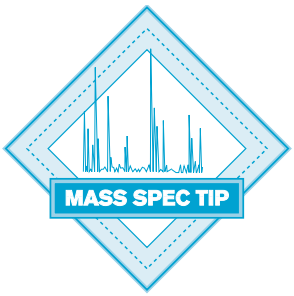
MASS SPEC TIPS

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)
Troubleshooting Finnigan 5100 GC/MS Systems

Author: Dennis Beauchamp
Affiliation: Roy F. Weston

In the lab where I work, we have many GC/MS systems but the majority of the systems are Finnigan 5100's. Many things can go wrong with mass spectrometers but the most common problem is when an analyst injects something and sees nothing. I call this "No Peaks".

No Peaks

This can be caused by many different things. Troubleshoot the problem with the following procedure.

Is the Reset Light lit? YES-Don't press the button before you check for the problem. This light being lit turns off all the analyzer voltages. It could be a vacuum problem. Open the right door and look at the power controller. The foreline pressure should be below .25 torr, the Ion source pressure should be below 1.25 torr.

Are any of the Red LED's lit? Which one? These little lights can tell you a lot if you let them. The Turbo Speed light means the turbo is not running. This could be caused by a high foreline pressure above .25 torr, bad turbo or bad turbo controller. High foreline pressure could be caused by a broken column or a loose fitting in the interface/separator oven or a bad foreline pump. A high foreline pressure should also cause the Foreline Pressure light to light. The Turbo Coolant light lit could be caused by a Water cooler problem if so equipped. The System Vented light lites only when the PUMP/Off/VENT switch on the front panel is switched to vent position.

If there are no errors indicated push the Reset light button. If it goes out just continue to troubleshoot the instrument.

Reset Light Off

This tells you at least the Vacuum system is operational and also that your analyzer voltages should be on. Now its time to check some voltages. Open the left door. This is called the "CARD CAGE". This is where all the circuit boards for the Mass Spec are located. Starting at the left of the card cage they are:

Digital I/O Board
GC Interface Board
Ion source Board
DC Rod Driver Board
Temperature Controller Board
Autosampler Board
RF Generator

Each board has test points on it that allow you an easy and safe way to check most of the important voltages that are needed for the GC/MS to operate.

The first voltage to check for is 5 Volts DC, this is the voltage that almost all the logic circuits use. The best place to check this is on the Ion Source Board, Test Points (TP) 1 and 2. If you have 5 volts here it should be everywhere else, but there are always exceptions so you should also check it on the digital I/O Board TP 1 and 2 and TP 3 and 2. If the Voltage is between 4.75 and 5.25 and free from AC noise it is okay.

The next voltage I usually check is the 12 volt dc Filament Power supply. This is the voltage that is needed to run the filament. The place to check for this is on the Ion Source Board at TP 15 and 18. With the filament off, the meter should read around 14 volts dc. When you turn the filament on it should drop down to between 10 and 12 volts dc. If you see this 2 to 4 volt voltage drop, your filament is not blown. If you don't see this 2 to 4 volt drop and have the 14 volts at TP 15 and 18 then your filament is probably blown and replacement is necessary. If the 14 volts is not at TP 15 and 18 it can mean a blown fuse. This fuse is in the power controller and it is not easy to get to. It is on the MS power supply, labeled F9, and it is a 5 amp slow blow fuse.

After checking the Filament supply, next check the Emission Current Voltage. You measure this also on the Ion Source Board, at TP 8 and 10. It should be between 50 and 100 mv dc depending on the initial adjustment (mv=millivolt) as long as it is a stable voltage. The next voltage to check is the Collector Current. This is also measured on the Ion Source, TP 6 and 7. It should

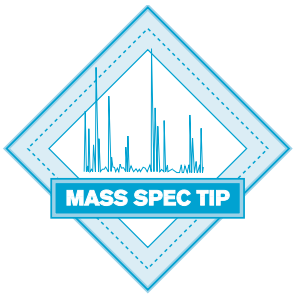
be about 70% of the emission current. Any lower than 15mv and this could indicate a bowed filament.

The next voltage to check also on the Ion Source Board is the -125 Volts dc for the RF Generator. You measure this at TP 21 and 22. It should be -125 volts dc +/-.5. If this voltage is below 1 volt dc check TP 11 and 22. This voltage should be around 150 to 175 volts dc. If you have this voltage but are below 125 vdc at TP 21 and 22 this means your RF Generator is "LOCKED UP". To correct this situation you have to do the following: First put your meter leads in TP 21 and 22, then on the Power Controller (behind right door), switch the Standby/Enable Switch to Standby, the Reset light should light. Carefully slide the RF Generator out approximately 3 to 4 inches. (to do this you have to grab the black handles on each side and lift up on them. This will allow the RF Generator out the 3 inches, switch the Standby/Enable switch to Enable. Then push the Reset button. Your meter should now read 125 volts dc, if it doesn't, this indicates a fuse is blown in the power controller. This fuse is labeled A-F3 and it is a 1/2 amp fuse. If it does read 125 volts, great. Now switch the Standby/Enable Switch back to Standby and then slowly, carefully slide the RF Generator back into it's slot. You have to lift up slightly on the black handles to allow it to seat itself back in the grooves. When you get it back in, switch the switch back to Enable and push the Reset button. If it reads 125 volts again, all systems go!! If not, repeat these steps, sometimes you need to do this a few times to get the RF Generator going again. You can't get peaks without RF so it's important that you get the 125 volts dc with the Generator installed.

The next voltages to check are on the DC Rod Driver Board. At TP 1 and 8 you should read approximately -255 volts dc. This is the minus DC voltage that goes to the rods. Now leave one lead in TP 8 and put the other one in TP 4, you should read +255 volts dc. If this is good then leave one lead in TP 8 and put the other one in TP 6, this should read +220 volts dc. This is the plus DC voltage that goes to the rods. If you have the + and -255 volts but one or both of the 220 volts are low or not there, that means the board is probably bad. If you don't have the + or theq -255 volts then that probably means the problem is a fuse on the MS power supply board in the power controller. The fuses are labeled F6 and F7 and they are 3/16 amp fuses.

The other TP to check on the Rod Driver Board are TP 11 and 14 which is -15 volt dc and TP 12 and 14 which is +15 volt dc. These seldom are bad. If you were to find these bad, the fuses are again on the MS power supply labeled F4 and F5 and they are 1 amp fuses. Also on this board are the Hardware Resolution Adjustments. They are at the top of the board, R13 Res Cal (low res.), R36 High Mass Res, R45 Corrector Mag, and R52 Corrector Form. If the instrument has been tuning up to now, I would advise not adjusting these pots. Make any resolution adjustments thru the MTune adjustments. The next voltage to check is the RF Generator voltage at TP 3 and 4 on the RF Generator. To check this voltage correctly, you first have to go into MTune and turn the sweep on, set the first mass to 800 and the mass range to 0, then with the meter at TP 3 and 4 adjust the black knob to give the lowest possible dc voltage. It should be between .5 and 1 volt dc. If you are unable to get a value below 1 volt or if it is over 10 volts, then you need to change your MTune settings. Set the first mass to 300 and the mass range to 0 and try to adjust the voltage again. If you are able to get a lower dc voltage, probably around .25vdc then in MTune set first mass to 500 and adjust the knob again. The voltage will be higher but still should be lower than 1 volt. Then set the first mass to 800 and adjust the knob again. Hopefully it will be lower than 1 volt dc. There is a factory recommended procedure for RF Generator Adjustment in the 5100 service manual. It is in the Installation Section on page 25.

If after going thru all the above checks and you still have "No Peaks", it's probably time for a service call. Hopefully though during one of the previous steps you will have found a problem you can fix yourself. Good luck!



(2)
Leak Checking Mass Spectrometers

Author: Richard A. Berger
Affiliation: Washington University School of Medicine

The November, 1993 issue of THE MASS SPEC SOURCE contained a Mass Spec Tip concerning the use of Freon for leak checking Mass Spectrometers. We had used Freon-12 (Dichlorodifluoromethane) for this purpose until it was implicated in the ozone layer depletion phenomena. While Argon (MW 40) works well for leak detection in electron ionization (EI) mode, it does not work well in positive chemical ionization (PCI) mode using methane as the reagent gas since it is overshadowed by one of the methane complex ions ($C_3H_5^+$, M/E 41). Argon does not show up at all in negative chemical ionization (NCI) mode.

We searched for some time for a gas which was:

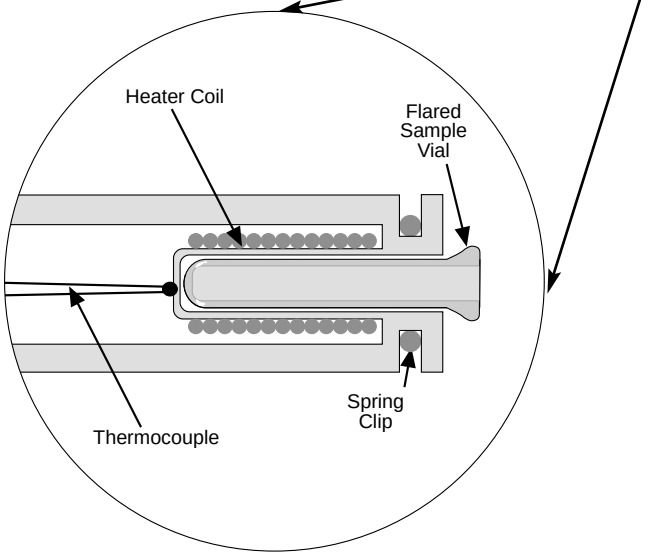
1. Non-flammable, non-toxic, and non-reactive,
2. Inexpensive and readily available, and
3. could be used in EI, PCI, and NCI modes.

We found that sulfur hexafluoride (SF_6 MW 146) satisfies most of these requirements. It is non-flammable and non-reactive; the TLV-TWA is 1000 ppm. It is available as a pure gas, usually shipped at its vapor pressure of 320 PSIG, or as an uncertified mixture with air, nitrogen, helium, or argon. Eighteen pounds of SF_6 in a “G” size cylinder cost less than $200. This included the cost of a control valve and cart to move the cylinder (about 50 lbs gross weight) from machine to machine. In EI and methane PCI mode, the sulfur hexafluoride spectra has M/E 127 $[M-F]^+$ as its base peak. In NCI mode M/E 146 (M^-) is the base peak.

HIGH TEMPERATURE DIRECT PROBE

For Hewlett-Packard and Finnigan MAT Mass Spectrometers

- **Maximum Temperature 800°C**
- **High Ramp Rates**
- **Direct Heating of Sample**
- **Strong, Easy to Use Flared Sample Vials**



Scientific Instrument Services has developed a new High Temperature Direct Insertion Probe (HT-DIP). HT-DIP models are available for use on the Hewlett-Packard 5989 MS Engine and Finnigan MAT SSQ/TSQ mass spectrometers. The probes can be used from ambient temperature up to 800°C. The probes are controlled via a stand alone digital temperature controller. Temperature ramp rates of up to 200°C/minute can used with controller. The controller includes a remote start button to remotely start the mass spectrometer data system. Applications include polymers, pharmaceuticals, natural products as well as other solid or liquid samples.

HPP4	High temperature DIP for 5989	$4200.00
FP15	High temperature DIP for Finn TSQ	$4200.00
PC2	High temperature DIP Controller	$1400.00

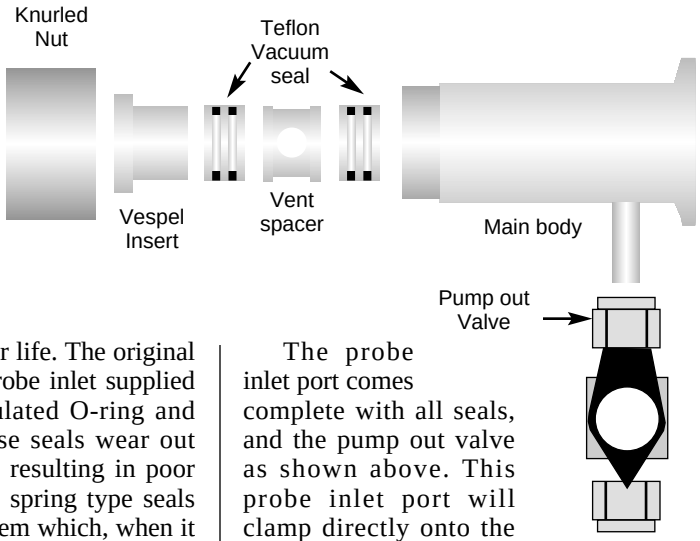
SCIENTIFIC INSTRUMENT SERVICES
908-788-5550
FAX: 908-806-6631

PROBE INLET FOR THE HP 5989

Scientific Instrument Services has redesigned the probe inlet port used on the HP 5989 MS Engine mass spectrometer. This new probe inlet port mounts directly to the mass spectrometer just as the original design, however the new inlet incorporates two design changes which provide major advantages to the user.

The new inlet port has a redesigned Teflon sealing system which provides much better vacuum sealing as well as longer life. The original vacuum seals on the probe inlet supplied by HP are an encapsulated O-ring and two spring seals. These seals wear out quite rapidly with use, resulting in poor vacuum sealing. These spring type seals also have graphite in them which, when it begins to wear, creates small pieces of graphite and dirt in the probe inlet. Our newly designed Teflon seals are very clean, have a long life and provide much better vacuum sealing.

The second design change was to make the inlet slightly longer to allow the use of the SIS direct exposure probe. This probe can be used on the original HP inlet system, however great care must be used to avoid damage to the filament wire at the end of the probe. The SIS probe inlet allows for greater clearance minimizing the potential for damage to the filament when using the direct exposure probe.



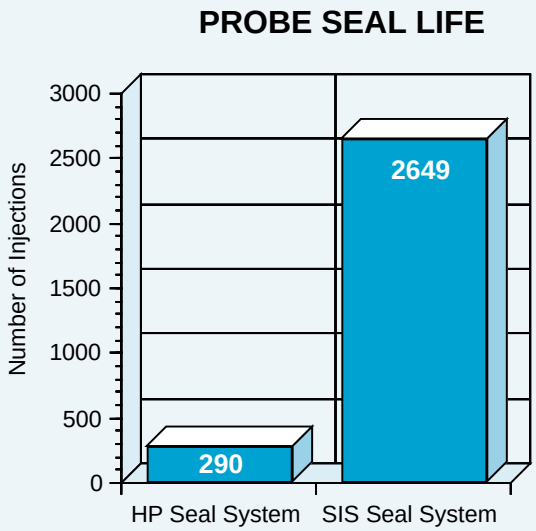
The probe inlet port comes complete with all seals, and the pump out valve as shown above. This probe inlet port will clamp directly onto the HP port valve if this valve is present on your mass spectrometer.

If you currently do not have a probe inlet system on your mass spectrometer, call SIS for more details. We are currently working on a new probe port valve consisting of a 1/4 turn isolation valve, HP Engine adaptor and clamps.

Part #	Description	Price Ea.
PI200	Probe Inlet port for HP 5989	$795.00
PI250	Replacement set of seals for PI200 (Pkg of 2)	$65.00

SIS Probe Inlet vs HP Probe Inlet Study

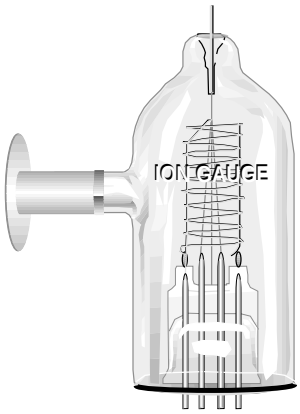
The chart opposite shows the results from a study which was conducted at SIS comparing the HP probe inlet system to the SIS inlet system. The SIS probe inlet system was mounted onto an automatic probe injection system which was fitted to a vacuum chamber where the vacuum was measured with a ionization gauge tube. The vacuum in the chamber was measured after each injection of the probe through the probe inlet. This allowed us to monitor the vacuum levels as the seals in the probe inlet began to wear from repeated use. The probe inlet system was considered to have failed when the ionization gauge would not remain on (i.e. the vacuum was greater than 1x 10^{-4} torr). As can be seen from the chart the SIS probe inlet system provided satisfactory performance for over 2500 injections of the probe until it failed at injection number 2649. The HP probe inlet was then mounted onto the automatic probe injector and the test repeated. The HP probe inlet first failed at injection number 14. The inlet was removed and inspected. There were fine pieces of graphite throughout the probe inlet. The probe inlet was cleaned, the same exact seals re-inserted and the inlet was remounted to the automatic injector. The test procedure was continued. The probe inlet failed again at injection number 29. The cleaning procedure was done a second time and the test continued. This failure and cleaning process was repeated seven times until the probe inlet failed completely at injection number 290.



FEATURED PRODUCTS

FROM SCIENTIFIC INSTRUMENT SERVICES

Ionization Gauge for HP 5989

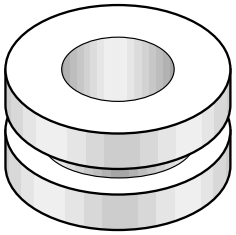


The ionization gauge tubes used on the Hewlett-Packard 5989 MS Engine mass spectrometers are available from SIS. These ionization gauge tubes have the quick flange on the side arm for installation into the mass spec system. They are direct replacements for the HP originals (HP# 0960-0799).

Part #	Description	Price ea.
G100KF25	Ion Gauge Tube for HP 5989	$160.00

Plate Insulator for H.P. 5989

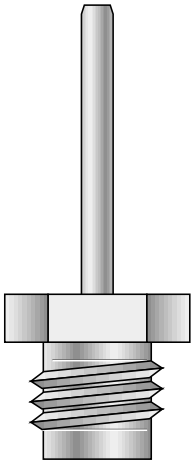
The ceramic plate insulator used in the H.P. Engine source is available from S.I.S. It is made of high purity alumina ceramic.



Part #	H.P. Part #	Description	Price ea.
HP14	05989-20109	Plate Insulator for H.P. Engine.	$25.00

New H.P. 5971 Transfer Line Tip

The transfer line tip for the H.P. 5971 MSD has been redesigned to permit the effluent from the end of the capillary column to purge directly into the MSD source, unlike the original gold plated transfer line tip in which the effluent purged inside this fitting and was diverted through side vents. This new design eliminates any Inlet metal contact with the sample, the capillary column protrudes slightly thru the end of the tip directly into the source. This new design should result in higher sensitivity for compounds which are sensitive to any metal contact.



Part #	Description	Price ea.
HP99	HP 5971 Transfer Line Tip	$79.00

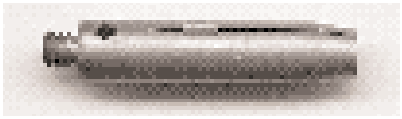
H.P. Engine Probe Port Parts

S.I.S. can now supply a wide variety of parts for the H.P. Engine in addition to the parts listed in our catalog. The following parts replacement for the H.P. Probe Inlet System are available.

Part #	H.P. Part #	Description	Price Ea.
V320	0905-1190	KF25 Viton 'O' Ring	$2.98
V211	0905-0819	Valve Stem Viton O' Ring, Horiz, ea.	1.21
V113	0905-0818	Valve Stem Viton 'O' Ring, Vert., pkg of 10	6.70
HP12	0905-0820	Probe Inlet Seal	12.50
SS4P4T	0101-0606	Pump Out Valve, Nupro 1/4 turn Ball Valve	63.00
NS25025	05989-20724	Hose Adaptor, KF25 to 1/4" Tube	45.00
L-25	6040-0289	Apiezon L Grease, 25 gram tube	68.00

Replate Gold on Probe Tip

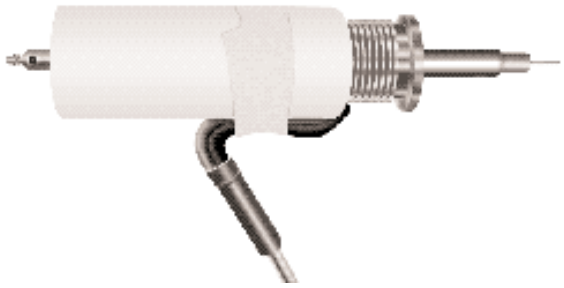
The gold plated probe tips used with the Hewlett-packard direct probes can be cleaned and replaced with gold if they become dirty. The old coat is completely removed and a new coat is applied.



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

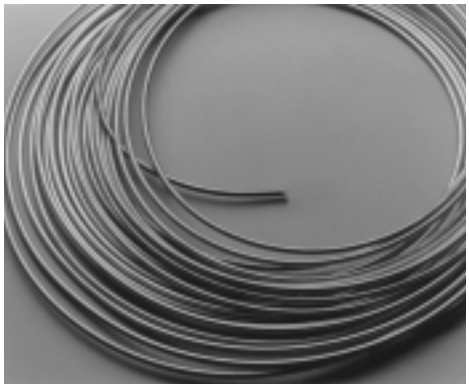
H.P. 5971 GC/MS Interface Repair

In addition to replacing the 1/16" Swagelok fitting on the H.P. 5971 GC/MS Interface, as described in our catalog, S.I.S. can now replace damaged heaters and temperature sensors should they burn out. This new repair includes the replacement of both the heater and the platinum temperature sensor. Other repairs of transfer lines including redesigns of this interface are also available from S.I.S. Parts to perform this repair are also available, if you have the equipment and facilities to perform this repair.



Part #	Description	Price Ea.
REP88	5971 Interface Repair	$130.00
REP89	Interface Heater/Sensor Repair	$185.00
HPC3	Interface Heater	$45.00
PRT 8	Interface Sensor	$80.00

PEEK and Teflon Instrument Tubing



S.I.S. offers both PEEK and Teflon tubing in addition to the copper and stainless steel tubing found in our catalog.

Teflon (PTFE) tubing is very chemically inert and is used in many low pressure applications (500psi.). We use it in our thermal desorption system for example. It is very flexible and easy to work with. It is manufactured to be used with standard Swagelok fittings.

PEEK (Polyetheretherketone) is a very good substitute for stainless steel tubing in many applications and is commonly used in many LC applications. It has a high pressure rating of 5000 psi (1/8" OD PEEK is rated at 3000 psi), is very chemical resistant and very flexible. It is color coded by size as listed below. There is a 10% discount on quantities of 10 feet or more.

PEEK

Part No.	Description	OD	ID	Price/ Ft.
PK005	PEEK Tubing, Red	1/16"	.005"	$4.00
PK007	PEEK Tubing, Yellow	1/16"	.007"	$3.25
PK010	PEEK Tubing, Blue	1/16"	.010"	$2.80
PK020	PEEK Tubing, Orange	1/16"	.020"	$2.80
PK030	PEEK Tubing, Green	1/16"	.030"	$2.80
PK062	PEEK Tubing, Natural	1/8"	.062"	$6.00
PK080	PEEK Tubing, Natural	1/8"	.080"	$6.00

10% Discount on Quantity 10 feet or more

TEFLON

Part No.	Description	OD	ID	Price/ Ft.
T062010	Teflon Tubing	1/16"	.010"	$1.20
T062020	Teflon Tubing	1/16"	.020"	$1.20
T062030	Teflon Tubing	1/16"	.030"	$1.20
T125062	Teflon Tubing	1/8"	1/16"	$2.30

10% Discount on Quantity 10 feet or more

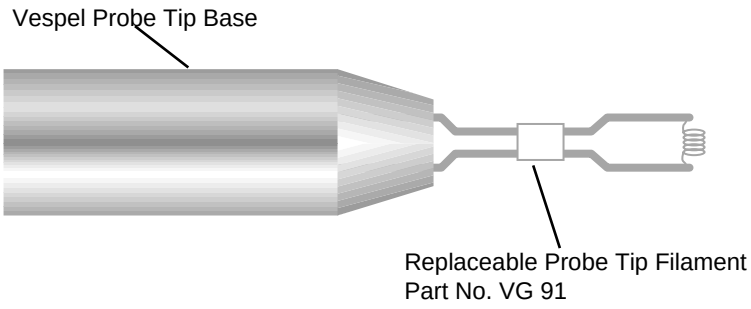
New Ceramics for the VG Mass Spectrometers

The following are new ceramic insulators for the VG TRIO and Autospec instruments which are now available from SIS. These insulators are all manufactured from high purity alumina ceramic for optimum performance. This list is in addition to the insulators listed in our catalog. If there are other insulators you require which are not included please give us a call.

Part No.	VG Part #	Description	Price ea.
VGI32	TSLI051	Source Lens Insulator	$22.00
VGI33	TSLI050	Source Lens Insulator	$22.00
VGI34	TDOW050	Pre-Filter Dowel	$12.00
VGI35	TTIT050	Trap Insulation Tube	$14.00
VGI36	TRIT051	Repeller Insulation Tube	$14.00
VGI37	TSLI053	Source Lens Insulator	$12.00
VGI38	TCSF050	Filter Rod Ceramic	$12.00
VGI39	M702864AD1	Ceramic Rod	$10.00
VGI40	7024364	Ceramic Spacer	$10.00

New DCI Probe Tip Design for the VG Mass Spectrometers

S.I.S. has redesigned the DCI Probe Tip for VG TRIO 1000 and 2000 instruments. The new design incorporates a Vespel DCI Probe Tip Base which plugs into the tip pf your existing probe. Then the replaceable Probe Tip Filaments are plugged into this new Vespel Probe Tip Base. Once the Vespel Probe Tip Base is purchased, the cost of repairing or replacing the probe filaments is much less than those available from the manufacturer. This new DCI probe tip was designed for and tested by several of our customers. The DCI filaments performed the same as those originally used but the replacement cost is much less.



Part No.	Description	Price ea.
VG90	Vespel DCI Probe TIp for VG TRIO 1000, 2000	$300.00
VG91	DCI Probe Tip Filament for VG TRIO 1000	$75.00
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0.32	0.10	123-5531	$435.00	123-5561	$750.00
0.32	0.25	123-5532	$435.00	123-5562	$750.00
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