



**VARIAN**

Varian, Inc.  
2700 Mitchell Drive  
Walnut Creek, CA 94598-1675/usa

---

## **4000 GC/MS**

# **Hardware Operation Manual**



All rights reserved including the right of reproduction in whole or in part in any form. This document may be electronically reproduced, distributed, or printed in its entirety provided this copyright and statement are attached. Any modification or any other reproduction, distribution, or use of this document or portions hereof is strictly prohibited without the express written permission of Varian, Inc.

COPYRIGHT 2007. All rights reserved.



# ***Declaration of Conformity***

We hereby Declare that the equipment listed below complies with the requirements of:

The Low Voltage Directive 73/23/EEC (93/68/EEC)  
The EMC Directive 89/336/EEC (92/31/EEC and 93/68/EEC)

## **Applicable Standards**

**LVD** EN 61010-1:2001

**EMC** EN 61326+A1

**Type of Equipment:** **Model:** 4000 MS

## **Authorized Representative in the EU**

**Print Name:** G. A. Wassink

**Signed:**

**Position:** Quality Manager

**Date:** March 23, 2005

**Company Name:** Varian B.V.

**Address:** Herculesweg 8

P.O. Box 8033

4330 EA Middelburg

The Netherlands

**Telephone:** +31(0) 118 671 000

**Fax:** +31(0) 118 633 118

## **Manufacturer**

**Print Name:** Seamus Flanagan

**Signed:**

**Position:** General Manager

**Date:** March 23, 2005

**Company Name:** Varian, Inc.

**Address:** 2700 Mitchell Drive

Walnut Creek, California 94598

USA

**Telephone:** 925-939-2400

**Fax:** 925-945-2168



**VARIAN**





**VARIAN**

## *Quality Systems At Varian, Inc.*

The ISO 9000 series standards were created in Geneva in 1987 to cut through a morass of conflicting quality definitions. These standards define a model for quality assurance systems in product design, development, manufacturing, installation, service, and customer support. They are now the worldwide quality assurance benchmark used to gauge the strength of a company's commitment to quality, and the value of its quality systems.

Various organizations around the world, such as the British Standards Institution (BSI), provide certified, objective auditors to scrutinize quality procedures, product development, manufacturing processes, and customer satisfaction programs. No company can claim ISO 9000 series registration unless it receives a stamp of approval from the demanding quality assessors of BSI or similar accredited examining body. ISO 9000 series registration constitutes an objective third-party report to determine the level of a supplier's commitment to quality.

In 1992, Varian, Inc., Analytical Instruments became registered to the most comprehensive of the ISO 9000 series standards — ISO 9001. ISO 9001 registration means that every stage of our quality system, including product development, manufacturing, final test, shipping, and parts and supplies has been rigorously examined against the most exacting set of internationally recognized standards. It means we live up to a standard of quality that you can count on today, and into the future. Our Quality System has received ISO 9001 certification number FM21797.

The quality systems that earned us ISO 9001 registration have direct benefits for our customers:

- ◆ We can speed instruments to you faster than ever before. Emergency orders can be processed even faster.
- ◆ We fill your orders promptly and completely.
- ◆ We have implemented a system of continuous feedback from our customers — we are aware of your needs today and tomorrow.
- ◆ We have improved your productivity by cutting systems failure rates in half and speeding service response time.
- ◆ We have embedded continuous improvement into the fabric of our organization so that we can achieve even higher levels of quality in the future.
- ◆ We are embedding GLP requirements into our products and services to help you meet your regulatory compliance requirements.

ISO 9001 registration is not enough. For us, quality is defined by our customers. We are not satisfied unless you are satisfied. We are striving to understand customer needs, using independent surveys, user groups, customer advisory boards, and our "Hallmark of Quality" response program, in addition to individual face-to-face customer contact. Our products and our processes are configured to meet those needs.

We know that you are seeking more than the most advanced processes and top-notch applications expertise. You want to join forces with a partner committed to delivering world-class quality, reliability, and value — on time, every time.

Our overriding aim is to be that partner.





**VARIAN**

## *Qualitätssysteme bei Varian, Inc.*

Die Standards der ISO 9000 Serien wurden 1987 in Genf mit dem Ziel geschaffen, das Durcheinander gegensätzlicher Qualitätsbestimmungen zu entwirren. Diese Standards legen ein Modell für Qualitätssicherungssysteme hinsichtlich Produktdesign, Entwicklung, Herstellung, Installation, Service und Kundenbetreuung fest. Sie sind nun die weltweiten Maßstäbe der Qualitätssicherung, die die Anstrengungen eines Unternehmens bezüglich der Qualität und der Bedeutung seiner Qualitätssysteme messen.

Verschiedene Organisationen in der ganzen Welt, wie die British Standards Institution (BSI), stellen ausgebildete, objektive Prüfer zur Begutachtung von Qualitätsmaßnahmen, Produktentwicklung, Herstellungsprozessen und von Programmen zur Erforschung der Kundenzufriedenheit zur Verfügung. Kein Unternehmen kann die ISO 9000 Registrierung beantragen, ohne die Genehmigung von den beauftragten Qualitätsgutachtern der BSI oder einer ähnlichen akkreditierten Stelle erhalten zu haben. Die ISO 9000 Registrierung bildet einen objektiven Bericht von dritter Seite, um den Grad der Qualitätsanstrengung eines Lieferanten zu bestimmen.

1992 wurden die Varian, Inc., Analytical Instruments nach den umfassendsten Standards der ISO 9000 Serie registriert — ISO 9001. Die ISO 9001 Registrierung bedeutet, daß jedes Stadium unseres Qualitätssystems, einschließlich Produktentwicklung, Herstellung, Endkontrolle, Versand, sowie Teile und Zubehör rigoros gegen die anspruchsvollste Serie international anerkannter Standards geprüft worden ist. Das bedeutet, daß wir einen Qualitätsstandard bieten, auf den Sie heute und in Zukunft rechnen können. Unser Qualitätssystem hat die ISO 9001 Zertifikatnummer FM21797 erhalten.

Die Qualitätssysteme der ISO 9001 Registrierung haben für unsere Kunden direkte Vorteile:

- ◆ Wir können Instrumente schneller denn je zu Ihnen schicken. Eilbestellungen werden noch schneller durchgeführt.
- ◆ Wir erfüllen Ihre Bestellungen pünktlich und vollständig.
- ◆ Wir haben ein System kontinuierlichen Informationsrückflusses von unseren Kunden aufgebaut—wir kennen Ihre Anforderungen von heute und von morgen.
- ◆ Wir haben Ihre Produktivität durch Halbierung der Systemfehlerraten und durch Verkürzung unserer Reaktionszeit im Service verbessert.
- ◆ Wir haben kontinuierliche Verbesserungen in unserer Organisationsstruktur verankert, so daß wir künftig eine noch höhere Qualität erreichen können.
- ◆ Wir haben die GLP Anforderungen in unsere Produkte und Dienstleistungen eingeführt, um Ihnen bei der Erfüllung Ihres behördlichen Abnahmeprotokolls zu helfen.

Die ISO 9001 Registrierung ist nicht genug. Für uns wird Qualität durch unsere Kunden definiert. Wir sind nicht zufrieden, wenn Sie es nicht auch sind. Wir bemühen uns, die Anforderungen unserer Kunden durch unabhängige Untersuchungen, Anwendergruppen, Kundenberatungsgremien und unser Antwortprogramm "Gütesiegel der Qualität" zu verstehen, zusätzlich zu persönlichen Kundenkontakten. Unsere Produkte und unsere Prozesse sind so gestaltet, daß sie diese Anforderungen erfüllen.

Wir wissen, daß Sie mehr als fortschrittliche Prozesse und ausgezeichnetes Anwendungswissen suchen. Sie suchen einen Partner, der Qualität von Weltklasse, Verlässlichkeit und Nutzen für Sie liefert—pünktlich und jederzeit.

Unser oberstes Ziel ist, für Sie dieser Partner zu sein.

QUALITY SYSTEM  
**ISO 9001**  
CERTIFIED



**VARIAN**

## *Systèmes de qualité chez Varian, Inc.*

Les normes ISO série 9000 ont été créées à Genève, en 1987, pour remédier à la confusion dans la définition des normes de qualité. Ces normes définissent un modèle de contrôle de qualité dans le domaine de la conception produit, du développement, de la production, des installations, des services et du support client. Elles constituent à présent la référence mondiale en matière de contrôle de qualité utilisée aux fins d'évaluation du niveau d'engagement d'une entreprise dans ce domaine et la valeur de ses systèmes de qualité.

Plusieurs organisations de par le monde, telle la British Standards Institution (BSI) offrent les services d'auditeurs qualifiés et objectifs, chargés d'examiner les procédures de qualité, le développement de produit, les procédés de fabrication et les programmes de satisfaction du client.

Aucune société ne peut se prévaloir de l'homologation ISO 9000, sans avoir reçu l'approbation des évaluateurs rigoureux de la BSI ou d'un organisme accréditif similaire. L'homologation ISO 9000 constitue une évaluation objective d'un tiers afin de déterminer le niveau d'engagement d'un fournisseur dans le domaine de la qualité.

En 1992, Varian, Analytical Instruments a reçu l'homologation ISO 9001, normes des plus complètes de la série ISO 9000. En d'autres termes, chaque étape du processus de qualité, notamment le développement produit, la fabrication, le test final, l'expédition et les fournitures de pièces a été soumis à un contrôle rigoureux par rapport à des normes extrêmement strictes, reconnues au niveau international. Nous sommes donc à même de vous garantir et de maintenir un niveau de qualité. Lesdites procédures ont reçu l'homologation ISO 9001 numéro FM21797.

Les systèmes de qualité qui ont reçu l'homologation ISO 9001 présentent des avantages directs pour nos clients :

- ◆ Nous sommes en mesure de vous livrer les instruments et de traiter les commandes en urgence dans des délais record.
- ◆ Nous répondons pleinement et de manière rapide à vos commandes.
- ◆ Nous avons mis en place un système de feedback continu de la part de nos clients et sommes conscients de vos attentes présentes et futures.
- ◆ Nous avons amélioré votre productivité en réduisant de moitié les Temps de panne et en accélérant les temps de réponse.
- ◆ Nous avons apporté des améliorations constantes au sein de notre structure, afin d'atteindre des niveaux de qualité optima, à l'avenir.
- ◆ Nos produits et services reflètent les exigences BPL pour vous permettre de répondre aux impératifs de respect de la réglementation.

Toutefois, nous ne nous contentons pas de l'homologation ISO 9001. Pour nous, la qualité est définie par nos clients. Nous ne sommes satisfaits que lorsque nos clients le sont. Nous nous efforçons de comprendre vos besoins, à l'aide d'évaluations externes, de groupes d'utilisateurs, de comités de conseil clients, et de notre programme "Hallmark of Quality", outre les contacts directs que nous établissons avec chacun de nos clients. Nos produits et nos procédés sont conçus pour répondre à vos attentes.

Nous n'ignorons pas que vous recherchez plus que des processus évolués et un savoir-faire d'exception dans le domaine des applications. Vous souhaitez conjuguer vos forces avec un partenaire s'étant engagé à offrir une qualité, une fiabilité et une valeur optimales, au moment où il faut et quand il faut.

Notre principal objectif : devenir votre partenaire !





**VARIAN**

## *I sistemi di qualità della Varian, Inc.*

La serie degli standard ISO 9000 è stata presentata nel 1987 a Ginevra con lo scopo di mettere ordine in un groviglio di definizioni contrastanti sulla qualità. Tali standard definiscono un modello che assicura la qualità nella progettazione, nello sviluppo, nella fabbricazione, nell'installazione e nella manutenzione dei prodotti nonché nel servizio assistenza clienti. Oggi come oggi essi costituiscono il punto di riferimento, a livello mondiale, ai fini della valutazione dell'impegno delle diverse aziende sul fronte della qualità e della validità dei sistemi di qualità da esse adottati.

Diverse organizzazioni internazionali, come la British Standard Institution (BSI), dispongono d'ispettori certificati e imparziali per la valutazione delle procedure di qualità, dello sviluppo dei prodotti, dei processi di fabbricazione e dei programmi di soddisfazione del cliente. Nessuna azienda può asserire d'essere in possesso della certificazione ISO 9000 finché non dispone del marchio d'approvazione concesso dai rigorosi ispettori di qualità della BSI o di altri enti di controllo riconosciuti. La certificazione di conformità agli standard ISO 9000 costituisce un'attestazione imparziale di terzi del grado d'impegno di una determinata azienda nei confronti della qualità.

Nel 1992 la Varian, Inc., Analytical Instruments ha ottenuto l'omologazione allo standard più completo della serie ISO 9000, l'ISO 9001. L'omologazione ISO 9001 significa che ogni singola fase del nostro sistema di qualità - compresi lo sviluppo del prodotto, la fabbricazione, le prove finali, la spedizione, i componenti e le forniture - è stata rigorosamente esaminata a fronte della serie più esigente di standard riconosciuti a livello mondiale, il che significa che rispondiamo pienamente ad uno standard qualitativo sul quale il cliente può contare oggi come nel futuro. Il nostro Sistema di Qualità ha ottenuto la certificazione ISO 9001 col numero FM21797.

I sistemi di qualità per i quali abbiamo ottenuto l'omologazione ISO 9001 comportano dei vantaggi diretti per i nostri clienti, ovvero:

- ◆ Siamo in grado di consegnare gli strumenti più rapidamente rispetto al passato, con la possibilità di evadere le richieste d'emergenza con una rapidità ancora maggiore.
- ◆ Gli ordini vengono evasi tempestivamente ed in modo completo.
- ◆ Abbiamo messo a punto un sistema di riscontro costante con la clientela, in modo da poter essere sempre perfettamente informati sulle esigenze attuali e future del cliente.
- ◆ Abbiamo migliorato la produttività del cliente riducendo della metà il tasso di guasti dei sistemi e velocizzando i tempi d'intervento della manutenzione.
- ◆ Abbiamo introdotto un costante miglioramento nella nostra struttura organizzativa in modo da poter conseguire in futuro livelli qualitativi ancor più elevati.
- ◆ Stiamo adeguando i nostri prodotti e servizi agli standard GLP per poter aiutare i clienti a soddisfare i requisiti di conformità posti loro dagli enti normativi.

Ma l'omologazione ISO 9001 non è tutto. Per quanto ci riguarda, la qualità viene definita dai nostri clienti: noi siamo soddisfatti solo se lo è il cliente. Ci adoperiamo al massimo per comprendere le esigenze del cliente, ricorrendo ad indagini di società private, gruppi di utenti, associazioni di consumatori e con il nostro programma di risposta Hallmark of Quality - il marchio di garanzia di qualità - oltre che col contatto diretto coi singoli clienti. I nostri prodotti ed i nostri processi sono configurati per rispondere a tali esigenze.

Sappiamo che a Voi i processi più avanzati e l'esperienza delle applicazioni di prim'ordine non bastano. Sappiamo che intendete unire le vostre forze con quelle d'un partner impegnato a fornire livelli qualitativi internazionali, affidabilità e valore, in modo tempestivo e costante.

Quel partner vogliamo essere noi.





**VARIAN**

## *Sistemas de calidad en Varian, Inc.*

Las normas ISO 9000 fueron creadas en Ginebra en 1987 para acabar con una multitud de definiciones de calidad contradictorias. Estas normas constituyen un modelo de sistemas de garantía de calidad en el diseño, desarrollo, fabricación, instalación, mantenimiento y asistencia técnica de productos. Se han convertido en el banco de pruebas de garantía de calidad a nivel mundial y miden el grado de compromiso de una empresa con la calidad, así como el alcance de sus sistemas de calidad.

Diversas organizaciones mundiales, como la British Standards Institution (BSI), proporcionan expertos titulados de probada objetividad para investigar procedimientos de calidad, desarrollo de productos, procesos de fabricación y programas de servicio al cliente.

Varian, Inc., Analytical Instruments fue registrada en 1992 con la norma más exhaustiva de la serie ISO 9000: la ISO 9001. La certificación por la norma ISO 9001 significa que todas las etapas de nuestro sistema de calidad, como el desarrollo del producto, la fabricación, las pruebas finales, la expedición, así como los suministros y recambios, han sido examinados rigurosamente respecto a las normas más exigentes reconocidas internacionalmente. Significa que nos comprometemos a mantener un nivel de calidad con el que podrá siempre contar, hoy y en el futuro. Il nostro Sistema di Qualità ha ottenuto la certificazione ISO 9001 col numero FM21797.

Los sistemas de calidad que nos valieron la certificación ISO 9001 representan beneficios directos para nuestros clientes:

- ◆ haremos llegar nuestros aparatos más rápidamente que nunca. Podemos cumplir con pedidos urgentes aún más deprisa.
- ◆ Atenderemos sus pedidos de forma rápida y completa.
- ◆ Aplicamos un sistema de retorno de información permanente con nuestros clientes: siempre somos conscientes de sus necesidades, actuales o futuras.
- ◆ Hemos mejorado la productividad de nuestros clientes, disminuyendo el índice de defectos a la mitad y acortando el tiempo de respuesta del servicio de mantenimiento.
- ◆ Hemos integrado sistemas de mejora continua en nuestra organización, de forma que podremos obtener niveles de calidad aún superiores en un futuro.
- ◆ Estamos integrando los requerimientos GLP en nuestros productos y servicios para ayudarle a cumplir con requerimientos de conformidad obligatorios.

La conformidad con ISO 9001 no nos basta. Para nosotros, los criterios de calidad los definen nuestros clientes. No estaremos satisfechos hasta que usted lo esté. Intentamos comprender las necesidades de nuestros clientes, a través de entidades independientes, grupos de usuarios, oficinas de asesoramiento a usuarios y nuestro programa de respuesta "Hallmark of Quality", además de los contactos directos con nuestros clientes. Nuestros productos y procedimientos están diseñados para poder corresponder a sus necesidades.

Sabemos que nuestros clientes buscan más que experiencia en procesos avanzados y aplicaciones punteras. Se trata de unir fuerzas con un socio que se compromete a entregar calidad reconocida a nivel mundial, fiabilidad y valor, a tiempo, siempre.

Nuestra meta principal es ser ese socio.



# Varian, Inc. Analytical Instrument Warranty

## **Hardware Products**

All analytical instruments sold by Varian, Inc. are warranted to be free from defects in material and workmanship for the periods specified and in accordance with the terms on the face of Varian's quotation or as otherwise agreed upon in writing between Varian and the Customer. The warranty period begins on the date of **shipment** from Varian to the original Customer. However, where installation is paid for by the Customer or included in the purchase price, the warranty period begins upon completion of installation. If the Customer schedules **installation** to start later than 30 days after delivery or if such delay is caused through the Customer's inability to provide adequate facilities or utilities or through failure to comply with Varian's reasonable pre-installation instructions or through other omissions by Customer, then the warranty period starts on the 31st day from date of shipment. Moreover Varian will charge the Customer for labor and other expenses involved in making multiple or follow-up installation service calls.

## **Software Products**

Where software is provided within the frame of a license agreement concluded between the Customer and Varian, any warranty shall be strictly in accordance with the terms of such agreement.

In the absence of a license agreement and unless an alternate warranty period is agreed upon in writing between Varian and the Customer, the warranty period is as specified on the face of Varian's quotation. Varian warrants such software products, if used with and properly installed on Varian hardware or other hardware as specified by Varian to perform as described in the accompanying Operator's Manual and to be substantially free of those defects which cause failure to execute respective programming instructions; however, Varian does not warrant uninterrupted or error-free operation.

## **Remedies**

The sole and exclusive remedy under hardware warranty shall be **repair** of instrument malfunctions which in Varian's opinion are due or traceable to defects in original materials or workmanship or, at Varian's option, **replacement** of the respective defective parts, provided that Varian may as an alternative elect to **refund** an equitable portion of the purchase price of the instrument or accessory.

Repair or replacement under warranty does not extend the original warranty period.

Repair or replacement under warranty claims shall be made in Varian's sole discretion either by sending a Customer Support Representative to the site or by authorizing the Customer to return the defective accessory or instrument to Varian or to send it to a designated service facility. The Customer shall be responsible for loss or damage in transit and shall prepay shipping cost. Varian will return the accessory or instrument to the Customer prepaid and insured. Claims for loss or damage in transit shall be filed by the Customer. To correct software operation anomalies, Varian will issue software revisions where such revisions exist and where, in Varian's opinion, this is the most efficient remedy.

## **Limitation of Warranty**

This **warranty does not cover** software supplied by the Customer, equipment and software warranted by another manufacturer or replacement of expendable items and those of limited life, such as but not limited to: Filters, glassware, instrument status lamps, source lamps, septa, columns, fuses, chart paper and ink, nebulizers, flow cells, pistons, seals, fittings, valves, burners, sample tubes, probe inserts, print heads, glass lined tubing, pipe and tube fittings, variable temperature dewars, transfer lines, flexible discs, magnetic tape cassettes, electron multipliers, filaments, vacuum gaskets, seats and all parts exposed to samples and mobile phases.

This **warranty shall be void** in the event of accident, abuse, alteration, misuse, neglect, breakage, improper operation or maintenance, unauthorized or improper modifications or tampering, use in an unsuitable physical environment, use with a marginal power supply or use with other inadequate facilities or utilities. Reasonable care must be used to avoid hazards.

**This warranty is expressly in lieu of and excludes all other express or implied warranties, including but not limited to warranties of merchantability and of fitness for particular purpose, use or application, and all other obligations or liabilities on the part of Varian, unless such other warranties, obligations or liabilities are expressly agreed to in writing by Varian.**

## **Limitation of Remedies and Liability**

The remedies provided herein are the sole and exclusive remedies of the Customer. In no case will Varian be liable for incidental or consequential damages, loss of use, loss of production or any other loss incurred.

# Varian, Inc. Analytical Instrument Garantie

## Hardwareprodukte

Es wird garantiert, daß alle von Varian, Inc. verkauften analytischen Instrumente für die angegebene Zeitdauer und in Übereinstimmung mit den „Allgemeinen Lieferbedingungen“ oder anderen schriftlichen Zusagen zwischen Varian und dem Kunden frei von Material- und Herstellungsfehlern sind. Die Garantiezeit beginnt mit dem **Versanddatum** von Varian zum Originalkunden. Wenn die Installation vom Kunden bezahlt oder im Verkaufspreis eingeschlossen ist, beginnt die Garantiezeit nach der abgeschlossenen Installation. Wenn der Kunde den **Installationsbeginn** später als 30 Tage nach erfolgter Lieferung ansetzt, oder wenn die Verzögerung dadurch verursacht wird, daß der Kunde nicht den ausreichenden Platz oder die Versorgungseinrichtungen beschafft oder Varian's berechtigte Anweisungen zur Installationsvorbereitung nicht einhält oder andere Versäumnisse des Kunden vorliegen, dann beginnt die Garantiezeit am 31. Tag nach dem Versanddatum. Darüber hinaus wird Varian dem Kunden den Arbeitsaufwand und andere Unkosten durch mehrfache oder fortgesetzte Installationsanforderungen berechnen.

## Softwareprodukte

Wo Software innerhalb des Rahmens eines Lizenzabkommens zwischen dem Kunden und Varian geliefert wird, wird die Garantie genau entsprechend der zeitlichen Abmachung eingehalten.

Besteht kein Lizenzabkommen und ist keine alternative Garantiezeit schriftlich zwischen Varian und dem Kunden festgelegt, gilt die Garantiezeit der „Allgemeinen Lieferbedingungen“. Varian garantiert für solche Softwareprodukte, die mit Varian's Hardware benutzt und richtig installiert sind oder zur Ausführung mit anderer von Varian angegebener Hardware, wie sie in der beigefügten Bedienungsanleitung beschrieben ist, daß sie im wesentlichen frei von solchen Defekten sind, die Fehler bei der Ausführung der jeweiligen Programmieranweisungen verursachen; Varian garantiert jedoch keine ununterbrochene oder fehlerfreie Arbeitsweise.

## Abhilfen

Die einzige und ausschließliche Abhilfe in der Hardwaregarantie wird die **Reparatur** der Instrumentstörungen sein, die sich nach Varian's Ansicht auf Defekte in den Originalteilen oder bei der Herstellung zurückführen läßt oder, nach Varian's Wahl, der **Austausch** der entsprechenden defekten Teile oder die **Erstattung** eines fairen Teils des Kaufpreises des Instruments oder Zubehörs, vorausgesetzt, daß sich Varian alternativ dafür entscheidet.

Reparatur oder Austausch unter Garantie verlängert nicht die ursprüngliche Garantiezeit.

Reparatur oder Austausch unter Garantieansprüchen soll in Varian's ausschließlich Ermessen entweder durch einen Serviceingenieur beim Kunden oder durch Ermächtigung des Kunden zum Einschicken des defekten Zubehörs oder Instruments an Varian oder einen Servicestützpunkt erfolgen. Der Kunde übernimmt die Verantwortung für Verlust oder Beschädigung im Transit und hat die Versandkosten im voraus zu bezahlen. Varian wird das Zubehör oder Instrument vorausbezahlt und versichert zum Kunden zurückschicken. Ansprüche für Verlust oder Beschädigung im Transit hat der Kunde zu erheben. Zur Korrektur von Anomalien des Softwarebetriebs wird Varian Software-Neuausgaben ausgeben, sofern Revisionen existieren und dies die beste Abhilfe ist.

## Garantieeinschränkungen

Diese **Garantie erfaßt nicht** vom Kunden bereitgestellte Software, Ausrüstungen und Software, die von anderen Herstellern garantiert werden oder den Austausch entbehrlicher Teile und solcher von begrenzter Lebensdauer wie diese, aber nicht darauf beschränkt: Filter, Glaswaren, Instrument Statuslampen, Lampenquellen, Septen, Säulen, Sicherungen, Schreiberpapier und Tinte, Zerstäuber, Flußzellen, Kolben, Dichtungen, Fittings, Ventile, Brenner, Probenröhren, Sondeneinsätze, Druckköpfe, glasausgekleidetes Rohr, Leitungs- und Rohrfittings, Dewars für variable Temperaturen, Transferleitungen, flexible Disketten, Magnetbandkassetten, elektronische Vervielfacher, Hitzdrähte, Vakuum Gaskets, Sitzflächen und alle Teile, die den Proben und mobilen Phasen ausgesetzt sind.

Diese **Garantie erlischt** bei eingetretenem Unfall, falscher Benutzung, Umbau, Mißbrauch, Vernachlässigung, Bruch, falscher Benutzung oder falscher Wartung, unbefugten oder falschen Modifikationen oder Basteleien, Benutzung in ungeeigneter physikalischer Umgebung, Benutzung mit marginaler Stromversorgung oder Benutzung mit anderen ungenügenden Einrichtungen oder Versorgungen. Mit vernünftiger Sorgfalt müssen Gefahren vermieden werden.

Diese Garantie steht ausdrücklich anstelle von allen anderen angedeuteten Garantien und schließt sie aus, einschließlich, aber nicht beschränkt auf Garantien der Verkäuflichkeit und Eignung für einen besonderen Zweck, Gebrauch oder Anwendung und allen anderen Verpflichtungen oder Haftungen von Varian's Seite, wenn nicht solche Garantien, Verpflichtungen oder Haftungen ausdrücklich schriftlich mit Varian vereinbart wurden.

## Beschränkung der Hilfen und Haftung

Die hier gegebenen Hilfen sind einzig und allein Sache des Kunden. In keinem Fall wird Varian für versehentliche oder sich ergebende Schäden wie Nutzungsverlust, Produktionsverlust oder jeden anderen Verlust haften.

# Garantie des instruments d'analyse Varian, Inc.

## **Matériel**

Les instruments d'analyse vendus par Varian, Inc. sont garantis exempts de défauts de matière et de fabrication, pour les périodes spécifiées et conformément aux conditions mentionnées sur le recto du devis ou aux termes de tout autre accord écrit intervenu entre Varian et le client. La période de garantie commence à compter de la date de **livraison** de Varian au client d'origine. Cependant, lorsque le client a acquitté les frais d'installation ou que celle-ci est inclue dans le prix d'achat, la période de garantie commence à compter de l'achèvement de l'installation.

Si le client prévoit le début de **l'installation** au-delà de 30 jours après la livraison ou si ledit retard est dû à l'inaptitude du client à mettre à disposition les installations ou services ou au non respect des instructions de pré-installation de Varian ou à la suite desdites négligences du client, la période de garantie commence le 31ème jour à compter de la date de livraison. De plus, Varian fera supporter au client tout frais de main d'oeuvre et autres coûts résultant de multiples appels téléphoniques aux fins de suivi de l'installation.

## **Logiciel**

Pour tout logiciel faisant l'objet d'un accord de licence conclu entre le client et Varian, la garantie sera strictement limitée aux termes dudit accord.

En l'absence d'accord de licence et sauf accord écrit sur tout autre période de garantie entre Varian et le client, la période de garantie est telle que spécifiée sur le recto du devis de Varian. Sous réserve de leur installation et de leur utilisation correcte sur le matériel Varian ou tout autre matériel, tel que spécifié, Varian garantie le fonctionnement tel que décrit dans le manuel d'utilisation fourni avec le matériel et l'absence de défauts entraînant l'impossibilité d'exécuter des instructions de programmation respectives. Toutefois, Varian ne garantit pas un fonctionnement sans interruption et sans erreurs.

## **Recours**

Le seul et unique recours relatif à la garantie du matériel se limite à la **réparation** suite à un mauvais fonctionnement de l'instrument, qui, de l'avis de Varian, est dû à des défauts des pièces d'origine ou de la fabrication, ou, à la discréction de Varian, au **remplacement** des pièces défectueuses en question, sous réserve du choix de Varian de **rembourser** une part raisonnable du prix d'achat de l'instrument ou de l'accessoire.

La répaation ou le remplacement sous garantie n'étend pas la période de garantie originale.

La réparation ou le remplacement, aux termes d'un recours, est laissé à l'entière discréction de Varian, soit par l'envoi d'un technicien de maintenance sur le site du client, soit en autorisant le client à retourner l'accessoire ou l'instrument défectueux à Varian, voire à l'envoyer à un service de maintenance désigné.

Le client assumera la responsabilité de toute perte ou sinistre lors du transport et réglera à l'avance les frais de transport. Varian renverra l'accessoire ou l'instrument au client en port payé et assuré. Toute réclamation résultant d'une perte ou d'un sinistre intervenu lors du transport devra être faite par le client. Aux fins de correction des anomalies de fonctionnement du logiciel, Varian diffusera des mises à jour des logiciels, le cas échéant, et si de l'avis de Varian, elles constituent la mesure corrective la plus appropriée en la matière.

## **Limitation de garantie**

Cette **garantie ne couvre pas** le logiciel fourni par le Client, les équipements ou logiciels garantis par un autre fabricant ni le remplacement des pièces consommables ou présentant une durée de vie limitée, notamment : filtres, verres, indicateurs d'état de l'instrument, lampes source, septa, colonnes, fusibles, papier graphique et encre, nébuliseurs cellules, pistons, joints, raccords, vannes, brûleurs, tubes d'échantillonnage, inserts de sonde, têtes d'impression, tubes à garniture de verre, dewars, lignes de transfert, disquettes, cassettes magnétiques, multiplicateurs d'électron, filaments, joints hermétiques, isolant et toutes les pièces en contact avec des échantillons et des phases mobiles.

**Ladite garantie est nulle** en cas d'accident, de mauvaise utilisation, d'altération, de négligence, de bris, d'utilisation, maintenance voire de modifications inappropriées, d'utilisation dans un environnement inadapté, d'utilisation avec une alimentation marginale ou d'autres installations ou services inappropriés. Un certain nombre de précautions doivent être prises pour éviter tout accident.

**Ladite garantie se substitue et exclue expressément toute garantie expresse ou tacite, y compris mais ne se limitant pas aux garanties relatives à la qualité marchande du programme et la garantie de son aptitude à une utilisation ou une application particulière, ainsi que toutes les autres obligations ou engagements de la part de Varian, à moins que lesdites garanties, obligations ou engagements aient fait expressément l'objet d'un accord écrit deVarian.**

## **Limitations de garantie et de la responsabilité :**

**Les recours exclusifs du client sont expressément énoncés aux présentes. En aucun cas, Varian ne sera tenu pour responsable de tout dommage provenant de l'utilisation ou en découlant, de toute impossibilité d'utilisation ou de déficit de production ou de tout autre perte y afférent.**

# Garanzia sugli strumenti analitici Varian, Inc.

## Prodotti hardware

Tutti gli strumenti analitici commercializzati dalla Varian, Inc. sono garantiti da eventuali difetti di materiali e di costruzione per i periodi ed alle condizioni indicati sull'offerta Varian o comunque concordati per iscritto tra la Varian ed il Cliente. Il periodo di garanzia decorre dalla data di spedizione dalla Varian al Cliente. Se l'installazione è a carico del Cliente o compresa nel prezzo d'acquisto, il periodo di garanzia decorre dalla fine dell'installazione. Se il Cliente prevede di procedere all'installazione oltre i 30 giorni dalla consegna o se tale ritardo è imputabile alla mancata messa a disposizione, da parte del Cliente, di locali o strumenti idonei o al mancato rispetto delle ragionevoli istruzioni di preinstallazione della Varian o comunque a fatti imputabili al Cliente, il periodo di garanzia decorre dal 31° giorno dalla data di spedizione. Inoltre, la Varian addebiterà al Cliente le spese di manodopera e d'altro tipo sostenute per interventi d'installazione multipli o di verifica.

## Prodotti software

Se il software viene fornito nell'ambito d'un contratto di licenza stipulato tra la Varian e il Cliente, trovano applicazione in via esclusiva le garanzie previste dal contratto.

In assenza d'un contratto di licenza e salvo diverso accordo scritto tra la Varian e il Cliente, vale il periodo di garanzia indicato nell'offerta della Varian. La Varian garantisce che i prodotti software, purché regolarmente utilizzati ed installati su hardware Varian o d'altri marchi da essa indicate, hanno le prestazioni descritte nel Manuale d'uso fornito a corredo del software e che sono sostanzialmente esenti da difetti che impediscono l'esecuzione delle rispettive istruzioni di programma. La Varian non garantisce alcun funzionamento ininterrotto o senza errori.

## Interventi Tecnici

Gli unici interventi previsti dalla garanzia sull'hardware sono o la riparazione dei malfunzionamenti dello strumento che, a giudizio della Varian, siano dovuti o riconducibili a difetti di costruzione dei materiali originali o, a discrezione della Varian, la sostituzione dei componenti difettosi, fermo restando che la Varian potrà, in alternativa, optare per il rimborso di una congrua parte del prezzo d'acquisto dello strumento o dell'accessorio difettoso.

La riparazione o la sostituzione in garanzia non valgono a prorogare in alcun modo il periodo di garanzia originariamente previsto.

Le riparazioni o le sostituzioni in garanzia verranno effettuate, ad esclusiva discrezione della Varian, inviando sul posto un tecnico o autorizzando la resa dello strumento o dell'accessorio difettoso alla Varian o al centro d'assistenza indicato dalla Varian. Il Cliente sarà responsabile di eventuali danni o perdite subiti durante il trasporto dallo strumento o dall'accessorio reso e dovrà pagare le spese di spedizione in via anticipata. La Varian restituirà al Cliente lo strumento o l'accessorio in porto franco con assicurazione a proprio carico. Sono a cura del Cliente gli eventuali reclami per perdite o danni di trasporto. Per eliminare eventuali anomalie di funzionamento del software, la Varian fornirà le eventuali revisioni del software disponibili qualora a suo giudizio siano il rimedio migliore.

## Limitazioni della garanzia

La presente garanzia non copre il software fornito dal Cliente, le attrezzature e il software garantiti da altre case né la sostituzione del materiale di consumo o di durata limitata, quali, senza intento limitativo, filtri, provette, spie di stato dello strumento, voltmetri, setti, colonne, fusibili, carta ed inchiostro, nebulizzatori, celle a flusso, pistoni, garnizioni, pezzi speciali, valvole, bruciatori, tubi di campionamento, inserti per sonde, testine di stampa, tubazioni rivestite in vetro, raccordi per tubi, dewars a temperatura variabile, linee di trasferimento, dischi flessibili, cassette a nastro magnetico, fotomoltiplicatori, filamenti, garnizioni per vuoto, e tutte le parti esposte all'azione dei campioni o delle fasi mobili.

La presente garanzia decade in caso d'incidente, abuso, modifica, uso improprio, incuria, rottura, funzionamento o manutenzione impropri, modifiche non autorizzate od improprie o manomissioni, impiego in ambiente fisico non idoneo, impiego con alimentazione ai limiti o con altri mezzi o dispositivi inadeguati. Devono inoltre essere adottate tutte le misure ragionevoli atte ad evitare ogni e qualsiasi rischio.

La presente garanzia sostituisce ed esclude espressamente ogni altra garanzia espressa o implicita, comprese - senz'intento limitativo - le garanzie di commerciabilità ed idoneità a scopi, impieghi od applicazioni specifici nonché tutti gli altri obblighi o responsabilità della Varian, a meno che le altre garanzie, obblighi o responsabilità in parola non siano stati accettati per iscritto dalla Varian.

## Limitazione degli interventi e delle responsabilità

Quelli qui contemplati sono gli unici ed esclusivi interventi cui ha diritto il Cliente. In nessun caso la Varian sarà responsabile per danni indiretti o consequenziali, mancata disponibilità, perdita di produzione o altre perdite subite.

# Instrumentos analíticos Varian, Inc. Garantía

## Productos hardware

Todos los instrumentos analíticos vendidos por Varian, Inc. están garantizados contra defectos de materiales y de fabricación por la duración especificada y de acuerdo con los términos establecidos en las ofertas de Varian, o según lo especificado en el acuerdo escrito entre Varian y el cliente. El plazo de garantía comienza a partir de la fecha de **envío** del material de Varian al cliente original. Sin embargo, si la instalación ha sido pagada por el cliente o incluida en el precio de compra, el plazo de garantía comenzará a partir de la fecha de conclusión de la instalación. Si el cliente especifica que la **instalación** comenzará 30 días después de la entrega, o si este plazo se genera por la imposibilidad por parte del cliente de proveer los medios necesarios o la falta de cumplimiento de las directrices de preinstalación de Varian, o cualquier otra omisión por parte del cliente, el plazo de garantía comenzará el trigésimo primer día a partir del envío. Además, Varian cobrará al cliente por trabajos y otros gastos relacionados con intervenciones de servicio de instalación múltiples o tardías.

## Productos de software

Cuando el software se suministra dentro del marco de una licencia de utilización acordada entre Varian y el cliente, cualquier garantía estará estrictamente limitada a los términos del citado acuerdo. En ausencia de una licencia de utilización y a no ser que exista un acuerdo de período de garantía por escrito entre Varian y el cliente, el período de garantía será el fijado de acuerdo con los términos de Varian que se citan. Varian garantiza estos productos de software si se instalan y usan con hardware Varian, u otro tipo de hardware en el que Varian certifique que funcionan según lo descrito en Manual de instrucciones, y que esté libre de defectos que impidan la ejecución de instrucciones de programación. Sin embargo, Varian no garantiza la utilización ininterrumpida o libre de errores.

## Recursos

El único y exclusivo recurso en cuanto a hardware bajo garantía será **reparar** los defectos del aparato, que, en opinión de Varian, sean claramente imputables a defectos de los materiales originales o de fabricación, o **sustituir** los componentes defectuosos, pudiendo Varian optar por **reembolsar** una parte equitativa del precio de compra del aparato o componente.

Las reparaciones o sustituciones en período de garantía no prolongan el período de garantía original.

Las reparaciones o sustituciones en período de garantía se efectuarán, a criterio exclusivo de Varian, enviando un representante de servicio posventa a la instalación, o autorizando al cliente a reexpedir el componente o aparato defectuoso a Varian o a un servicio de reparación designado. El cliente será responsable sobre pérdidas o daños de transporte, y pagará los costes de dicho transporte. Varian reexpedirá el componente o aparato a portes pagados y con seguro de transporte. Las demandas por daños o pérdidas deberán ser gestionadas por el cliente. Para corregir anomalías de funcionamiento de software, Varian editará revisiones de software, siempre y cuando éstas estén disponibles, y cuando, en opinión de Varian, este sea el remedio más eficaz.

## Limitación de garantía

Esta garantía no cubre software provisto por el cliente, equipos y software garantizados por otros fabricantes, consumibles o artículos de duración de vida limitada, como son, entre otros: filtros, elementos de vidrio, pilotos, lámparas, diafragmas, columnas, fusibles, papel y tinta de gráficos, nebulizadores, células de flujo, pistones, cierres, juntas, válvulas, quemadores, tubos de muestras, inserciones de sondas, cabezales de impresión, tubos de vidrio, juntas de tubo, dispositivos de temperatura variable, líneas de transferencia, discuetes, cintas magnéticas, multiplicadores de electrones, filamentos, juntas de vacío, soportes y todos los componentes en contacto con muestras y partes móviles.

Esta garantía no tendrá efecto en los casos de accidente, abuso, alteración, utilización incorrecta, negligencia, rotura, mantenimiento o uso inadecuados, modificaciones inadecuadas o no autorizadas, uso de la fuerza, uso en un entorno inadecuado, funcionamiento con una alimentación defectuosa o el uso con medios inadecuados. Es necesario tomar las precauciones adecuadas para evitar riesgos.

**Las garantías de los productos de software de Varian sustituyen y excluyen cualquier otra garantía, implícita o explícita, incluidas pero sin limitación, las garantías de comerciabilidad, adecuación a un fin, uso o aplicación en particular, y todas las demás obligaciones y responsabilidades por parte de Varian, a no ser que estas garantías, obligaciones y responsabilidades sean otorgadas expresamente y por escrito por Varian.**

## Limitaciones de recursos y responsabilidades

Los recursos provistos en lo citado son única y exclusivamente los del cliente. Varian no podrá ser responsable en ningún caso por daños imprevistos o consecuencias, pérdida de uso, pérdida de producción o cualquier otra pérdida incurrida.

# Safety Information

## Operating Instructions

This instruction manual is provided to help you establish operating conditions which will permit safe and efficient use of your equipment. Special considerations and precautions are also described in the manual, which appear in the form of **NOTES**, **CAUTIONS**, and **WARNINGS** as described below. It is important that you operate your equipment in accordance with this instruction manual and any additional information which may be provided by Varian. Address any questions regarding the safe and proper use of your equipment to your local Varian office.

### NOTE

Information to aid you in obtaining optimal performance from your instrument.

### ! CAUTION

Alerts you to situations that may cause moderate injury and/or equipment damage, and how to avoid these situations.

### ! WARNING

Alerts you to potentially hazardous situations that could result in serious injury, and how to avoid these situations.

#### Warning Symbol



##### WARNING: SHOCK HAZARD



##### WARNING: CHEMICAL HAZARD



##### WARNING: BURN HAZARD



##### WARNING: EYE HAZARD



##### WARNING: FIRE HAZARD



##### WARNING: EXPLOSION HAZARD



##### WARNING: RADIATION SOURCE



##### WARNING: MOVING PARTS

#### Warning Description

Hazardous voltages are present inside instrument. Disconnect from main power before removing screw-attached panels.

Hazardous chemicals may be present. Avoid contact, especially when replenishing reservoirs. Use proper eye and skin protection.

Very hot or cryogenically cold surfaces may be exposed. Use proper skin protection.

Eye damage could occur either from flying particles, chemicals, or UV radiation. Use proper eye and face protection.

The potential for fire may be present. Follow manual instructions for safe operation.

The potential for explosion may exist because of type of gas or liquid used.

Ionizing radiation source is present. Follow manual instructions for safe operation.

Keep hands and fingers away.



VARIAN

## General Safety Precautions

Follow these safety practices to ensure safe equipment operation.

- Perform periodic leak checks on all supply lines and pneumatic plumbing.
- Do not allow gas lines to become kinked or punctured. Place lines away from foot traffic and extreme heat or cold.
- Store organic solvents in fireproof, vented and clearly labeled cabinets so they are easily identified as toxic and/or flammable materials.
- Do not accumulate waste solvents. Dispose of such materials through a regulated disposal program and not through municipal sewage lines.

**NOTICE:** This instrument has been tested per applicable requirements of EMC Directive as required to carry the European Union CE Mark. As such, this equipment may be susceptible to radiation/interference levels or frequencies which are not within the tested limits.



### WARNING

This instrument is designed for chromatographic analysis of appropriately prepared samples. It must be operated using appropriate gases and/or solvents and within specified maximum ranges for pressure, flows, and temperatures as described in this manual. If the equipment is used in a manner not specified by the manufacturer, the protection provided by the equipment may be impaired.



### WARNING

It is the responsibility of the Customer to inform Varian Customer Support Representatives if the instrument has been used for the analysis of hazardous biological, radioactive, or toxic samples, prior to any instrument service being performed or when an instrument is being returned to the Service Center for repair.

## Electrical Hazards

- Disconnect the instrument from all power sources before removing protective panels to avoid exposure to potentially dangerous voltages.
- When it is necessary to use a non-original power cord plug, make sure the replacement cord adheres to the color coding and polarity described in the manual and all local building safety codes.
- Replace blown fuses with fuses of the size and rating stipulated on the fuse panel or in the manual.
- Replace faulty or frayed power cords immediately with the same type and rating.
- Make sure that voltage sources and line voltage match the value for which the instrument is wired.

## Compressed Gas Cylinders

- Store and handle compressed gases carefully and in strict adherence to safety codes.
- Secure cylinders to an immovable structure or wall.
- Store and move cylinders in an upright, vertical position. Before transport, remove regulators and install cylinder cap.
- Store cylinders in a well-ventilated area away from heat, direct sunshine, freezing temperatures, and ignition sources.
- Mark cylinders clearly so there is no doubt as to their contents.
- Use only approved regulators and connections.
- Use only connector tubing that is chromatographically clean (Varian Part Number 03-918326-00) and has a pressure rating significantly greater than the highest outlet pressure from the regulator.

## GC Safety Practices

### Exhaust System

No special exhaust ducting is necessary for GC detectors installed in a well-ventilated room except when the detectors are used to test hazardous chemicals. If you do install ducting:

- Use only fireproof ducting.
- Install a blower at the duct outlet.
- Locate duct intakes such that their vibration or air movement does not effect detector operation.
- Check periodically for proper operation of the duct.
- Ensure proper ventilation in lab area.

### Radioactive Source Detectors

- Read carefully and comply with all NOTES, CAUTIONS, and WARNINGS in the Ni<sup>63</sup> ECD manual.
- Perform the tests for removable radioactive contamination described in the Ni<sup>63</sup> ECD manual.
- Comply with leak test schedules and procedures.

### Burn Hazard

Heated or cryogenically cooled zones of gas chromatographs can remain hot or cold for a considerable time after instrument power is turned off. To prevent painful burns, ensure that all heated or cooled areas have returned to room temperature or wear adequate hand protection before you touch potentially hot or cold surfaces.

## LC Safety Practices

### High Pressure Hazard

- If a line ruptures, a relief device opens, or a valve opens accidentally under pressure, potentially hazardous high liquid pressures can be generated by the pump causing a high velocity stream of volatile and/or toxic liquids.
- Wear face protection when you inject samples or perform routine maintenance.
- Never open a solvent line or valve under pressure. Stop the pump first and let the pressure drop to zero.
- Use shatter-proof reservoirs capable of operating at 50-60 psi.
- Keep the reservoir enclosure closed when the reservoir is under pressure.
- Read and adhere to all NOTES, CAUTIONS, and WARNINGS in the manual.

### Flash Chromatography

The operator should be familiar with the physico-chemical properties of the components of the mobile phase.

Keep solvents from direct contact with the polyurethane supply tubing as certain solvents will cause weakening and leaks with possible bursting.

All components of the system should be connected to a common power supply and common ground. This ground must be a true ground rather than a floating ground.

Non-polar solvents can develop a static charge when pumped through the system. All vessels that contain mobile phase (including tubing and collection vessels) must be grounded to dissipate static electricity.

Employ static measuring and static discharge devices (e.g., air ionizers) to safeguard against the buildup of static electricity.

### Ultraviolet Radiation

Liquid chromatograph detectors that use an ultraviolet light source have shielding to prevent radiation exposure to personnel.

For continued protection:

- Ensure that protective lamp covers of variable and fixed wavelength detectors are in place during operation.
- Do not look directly into detector fluid cells or at the UV light source. When inspecting the light source or fluid cell, always use protective eye covering such as borosilicate glass or polystyrene.

**The following is a Federal Communications Commission advisory:** This equipment has been tested and found to comply with the limits of a Class A computing device, pursuant to part 15 of the FCC Rules. These limits are designed to provide reasonable protection against harmful interference when the equipment is operated in a commercial environment. This equipment generates, uses, and can radiate radio frequency energy and, if not installed and used in accordance with the instruction manual, may cause harmful interference to radio communications. Operation of this equipment in a residential area is likely to cause harmful interference in which case the user will be required to correct the interference at his own expense.

## Spare Parts Availability

It is the policy of Varian to provide operational spare parts for any instrument and major accessory for a period of five (5) years after shipment of the final production run of that instrument. Spare parts will be available after this five (5) year period but on an *as available* basis. Operational spare parts are defined as those individual electrical or mechanical parts that are susceptible to failure during their normal operation. Examples include relays, lamps, temperature probes, detector elements, motors, etc. Sheet metal parts, structural members or assemblies and castings, printed circuit boards, and functional modules are normally capable of being rebuilt to like-new condition throughout their useful life and therefore will be supplied only on an *as available* basis after the final production run of the instrument.

## Service Availability

Varian provides a variety of services to support its customers after warranty expiration. Repair service can be provided by attractively priced service contracts or on a time and material basis. Technical support and training can be provided by qualified personnel on both a contractual or as-needed basis.

## Varian, Inc. Analytical Instruments Sales Offices

For Sales or Service assistance and to order Parts and Supplies, contact your local Varian office.

### Argentina

Buenos Aires  
Tel. +54.11.4.783.5306

### Australia

Mulgrave, Victoria  
Tel. +61.3.9566.1134

### Austria

Vösendorf bei Wien  
Tel. +43.1.699.9669

### Benelux

Bergen Op Zoom  
Tel. +31.164.282.800

### Brazil and Latin America (S)

São Paulo  
Tel. +55.11.820.0444

### Canada

Mississauga, Ontario  
Tel. 800.387.2216

### China

Beijing  
Tel. +86.106209.1727

### Europe

Middelburg, The Netherlands  
Tel. +31.118.671.000

### France

Les Ulis Cédex  
Tel. +33.1.6986.3838

### Germany

Darmstadt  
Tel. +49.6151.7030

### India

Mumbai  
Tel. +91.22.857.0787/88/89

### Italy

Torino  
Tel. +39.011.997.9111

### Japan

Tokyo  
Tel. +81.3.5232.1211

### Korea

Seoul  
Tel. +82.2.345.22452

### Mexico and Latin America (N)

Mexico City  
Tel. +52.5.523.9465

### Russian Federation

Moscow  
Tel. +7.095.937.4280

### Spain

Madrid  
Tel. +34.91.472.7612

### Sweden

Solna  
Tel. +46.8.445.1620

### Switzerland

Varian AG  
Tel. +41.848.803.800

### Taiwan

Taipei Hsien  
Tel. +886.2.698.9555

### United Kingdom and Ireland

Walton-on-Thames  
Tel. +44.1932.898000

### Venezuela

Valencia  
Tel. +58.41.257.608

### United States

Walnut Creek, California, USA  
Tel. +1.800.926.3000

(GC and GC/MS)

Tel. +1.800.367.4752  
(LC)



**VARIAN**

[www.varianinc.com](http://www.varianinc.com)

# Sicherheitsinformationen

## Arbeitsanleitungen

Diese Arbeitsanleitung will Ihnen bei der Aufstellung solcher Arbeitsbedingungen helfen, die einen sicheren und wirkungsvollen Gebrauch Ihrer Geräte ermöglichen. Besondere Überlegungen und Vorsichtsmaßnahmen erscheinen in diesem Handbuch in Form von **HINWEIS**, **ACHTUNG** und **WARNUNG**, wie unten beschrieben. Es ist wichtig, daß Sie Ihr Gerät in Übereinstimmung mit dieser Arbeitsanleitung und allen möglichen zusätzlichen Informationen von Varian betreiben. Alle Fragen bezüglich Sicherheit und Handhabung Ihres Gerätes richten Sie an Ihr Varian Büro.

### HINWEIS

Eine Information, um einen optimalen Wirkungsgrad Ihres Instruments zu erzielen.

### ! ACHTUNG

Weist auf Situationen, die zu mäßiger Beeinträchtigung und/oder zu Geräteschäden führen und auf die Vermeidung dieser Situationen hin.

### ! WARNUNG

Weist auf mögliche Gefahrensituationen, die zu ernsthaften Verletzungen führen können und auf die Vermeidung dieser Situationen hin.

#### Warnungssymbol



#### WARNUNG ELEKTRISCHER SCHLAG



#### WARNUNG CHEMISCHE GEFAHR



#### WARNUNG VERBRENNUNGSGEFAHR



#### WARNUNG AUGENVERLETZUNG



#### WARNUNG FEUERGEFAHR



#### WARNUNG EXPLOSIONSGEFAHR



#### WARNUNG STRAHLUNGSQUELLE



#### WARNUNG BEWEGTE TEILE

#### Warnungsbeschreibung

Gefährliche Spannungen bestehen innerhalb des Instruments. Trennen Sie das Gerät vom Netz, bevor Sie abschraubbare Paneele entfernen.

Gefährliche Chemikalien können vorhanden sein. Vermeiden Sie jeden Kontakt, besonders beim Auffüllen der Reservoirs. Benutzen Sie wirksamen Augen und Hautschutz.

Sehr heiße oder tiefstgekühlte Oberflächen können freigelegt sein. Benutzen Sie einen wirksamen Hautschutz.

Herumfliegende Partikel, Chemikalien oder UV-Strahlung können Augenschäden verursachen. Tragen Sie deshalb einen geeigneten Schutz für Augen und Gesicht.

Es besteht eine mögliche Feuergefahr. Beachten Sie die Vorschriften im Handbuch für eine gefahrlose Benutzung.

Eine mögliche Explosionsgefahr besteht infolge der benutzten Gas- oder Flüssigkeitsart.

Es besteht eine ionisierende Strahlungsquelle. Beachten Sie die Vorschriften im Handbuch für eine gefahrlose Benutzung.

Bleiben Sie mit Ihren Händen und Fingern weg.



## Allgemeine Sicherheitsmaßnahmen

Befolgen Sie diese Sicherheitspraktiken für eine gefahrlose Gerätebenutzung.

- Prüfen Sie regelmäßig alle Versorgungs und Pneumatikleitungen auf Lecks.
- Gasleitungen dürfen nicht geknickt oder angestochen werden. Verlegen Sie die Leitungen außerhalb von Laufwegen und abseits von extremer Hitze oder Kälte.
- Lagern Sie organische Lösungsmittel in feuerfesten, belüfteten und eindeutig bezeichneten Schränken, damit sie leicht als toxische und/oder brennbare Materialien erkannt werden.
- Sammeln Sie keine Lösungsmittelabfälle. Entsorgen Sie solche Materialien über ein geregeltes Entsorgungsprogramm und nicht über die öffentlichen Abwasserleitungen.

**HINWEIS:** Dies Instrument wurde nach den zutreffenden Vorschriften der EMC Direktive getestet, die zum Führen des CE Zeichens der Europäischen Union berechtigen. Dieses Gerät kann an sich auf Strahlungs-/Störpegel oder Frequenzen außerhalb der getesteten Grenzen reagieren.



### WARNUNG

Dies Instrument ist für chromatographische Analysen entsprechend präparierter Proben gedacht. Es muß mit geeigneten Gasen und/oder Lösungsmitteln und innerhalb der im Handbuch spezifizierten maximalen Werte für Druck, Flüsse und Temperaturen betrieben werden.



### WARNUNG

Der Kunde ist vor der Durchführung irgendeines Geräteservices verpflichtet den Varian Kundendienstvertreter zu informieren, wenn das Instrument für Analysen gefährlicher biologischer, radioaktiver oder toxischer Proben benutzt worden ist.

## Elektrische Gefahren

- Lösen Sie das Instrument von allen Stromquellen, bevor Sie Schutzpaneele entfernen, damit Sie nicht mit potentiell gefährlichen Spannungen in Berührung kommen.
- Wenn ein Nicht-Original Netzkabelstecker benutzt werden muß, muß das Austauschkabel die im Handbuch beschriebene Farbcodierung und Polarität beibehalten und alle örtlichen Sicherheitsvorschriften erfüllen.
- Ersetzen Sie durchgebrannte Sicherungen nur mit Sicherungen der Werte, die am Sicherungspaneel oder im Handbuch angegeben sind.
- Ersetzen Sie fehlerhafte oder durchgescheuerte Netzkabel sofort durch Kabel gleicher Art.
- Sorgen Sie dafür, daß Spannungsquellen und die Netzspannung den gleichen Wert haben, für den das Instrument verdrahtet ist.

## Gasdruckflaschen

- Lagern und handhaben Sie komprimierte Gase vorsichtig und in strikter Einhaltung der Sicherheitsvorschriften.
- Befestigen Sie die Gasflaschen an feststehenden Aufbauten oder an Wänden.
- Lagern und transportieren Sie Gasflaschen in aufrechter Stellung. Druckregler zuvor abnehmen.
- Lagern Sie Gasflaschen in gut durchlüfteten Räumen, weit genug weg von Heizungen, direktem Sonnenschein, Frosttemperaturen und Entzündungszonen.
- Kennzeichnen Sie die Flaschen so eindeutig, daß kein Zweifel über deren Inhalt bestehen kann.
- Benutzen Sie nur geprüfte Druckminderer und Verbindungsstücke.
- Benutzen Sie nur chromatographisch reines Verbindungsrohr (Varian Part Number 03-918326-00), das wesentlich höheren Druck als den höchsten Ausgangsdruck des Druckminderers aushält.

## GC Sicherheitspraktiken

### Abgassystem

Für GC Detektoren, die in einem gut durchlüfteten Raum installiert sind, ist keine spezielle Abgasführung erforderlich, außer wenn die Detektoren zum Testen gefährlicher Chemikalien benutzt werden. Wenn Sie eine Abgasführung installieren:

- Benutzen Sie nur feuerfeste Führungen.
- Installieren Sie ein Gebläse am Ausgang.
- Ordnen Sie die Ansaugöffnung so an, daß ihre Er-schütterungen oder Luftströmungen nicht die De-tektorfunktion beeinträchtigen.
- Prüfen Sie regelmäßig die einwandfreie Arbeits-weise der Abgasführung.
- Sorgen Sie für gute Entlüftung im Laborbereich.

### Radioaktive Detektoren

- Lesen Sie sorgfältig und befolgen Sie alle **HINWEISE, ACHTUNGEN** und **WARNUNGEN** im Ni<sup>63</sup> ECD Handbuch.
- Führen Sie die Tests für zu beseitigende radioak-tive Kontamination durch, die im Ni<sup>63</sup> ECD Hand-buch beschrieben sind.
- Erfüllen Sie die Zeitpläne und Verfahren zur Di- chtigkeitsprüfung.

### Verbrennungsgefahr

Beheizte oder tieftemperaturgekühlte Zonen des Gas-chromatographen können beträchtlich lange heiß oder kalt bleiben, nachdem das Instrument bereits abgeschal-tet ist. Zur Vermeidung schmerzhafter Verbrennungen müssen Sie darauf achten, daß alle beheizten oder gekühlten Zonen auf Raumtemperatur zurückgegangen sind oder Sie müssen ausreichenden Handschutz be-nutzen, bevor Sie möglicherweise heiße oder kalte Oberflächen berühren.

## LC Sicherheitspraktiken

### Gefahr durch hohen Druck

Wenn eine Leitung bricht, eine Entlüftungseinheit sich öffnet oder ein Ventil sich unbeabsichtigt unter Druck öffnet, kann durch die Pumpe möglicherweise ein gefährlich hoher Flüssigkeitsdruck entstehen, der einen Strahl flüchtiger und/oder toxischer Flüssigkeiten von hoher Störmungsgeschwindigkeit verursacht.

- Tragen Sie einen Gesichtsschutz, wenn Sie Proben injizieren oder Routinewartungen durchführen.

- Öffnen Sie niemals eine unter Druck stehende Lösungsmittelleitung oder ein Ventil. Halten Sie zuerst die Pumpe an und lassen Sie den Druck auf Null abfallen.
- Benutzen Sie splittersichere Reservoirs, die für einen Druck von 3,4 bis 4,1 bar ausgelegt sind.
- Halten Sie die Reservoirverkleidung geschlossen, wenn die Reservoirs unter Druck stehen.
- Lesen Sie und befolgen Sie alle **HINWEISE, ACHTUNGEN** und **WARNUNGEN** im Handbuch.

### Blitzlicht-Chromatographie

Der Bediener sollte mit den physikalisch-chemischen Eigenschaften der Komponenten vertraut sein, aus denen sich die mobile Phase zusammensetzt.

Vermeiden Sie direkten Kontakt der Lösungsmittel mit den Zuführungsleitungen aus Polyurethan, da einige Lösungsmittel das Material der Leitungen schwächen und damit Undichtigkeiten oder Brüche hervorrufen können.

Alle Systemkomponenten sollten an der gleichen Netzstromquelle und einer gemeinsamen Erdung angeschlossen sein. Dabei muss es sich um eine echte, nicht um eine schwebende Erdung handeln.

Nicht-polare Lösungsmittel können sich beim Pumpen durch das System statisch aufladen. Alle Gefäße, die mobile Phase enthalten (einschließlich Leitungen und Sammelgefäß), müssen zur Ableitung elektro-statischer Aufladungen geerdet sein.

Setzen Sie Geräte zur Messung und Ableitung elektrostatischer Aufladungen (z.B. Geräte zur Luftpionisierung) als Maßnahmen gegen den Aufbau statischer Elektrizität ein.

### Ultraviolette Strahlung

Detektoren in Liquidchromatographen, die eine ultraviolette Lichtquelle benutzen, besitzen eine Abschirmung, die das Bedienungspersonal gegen Abstrahlungen schützt. Zum ständigen Schutz:

- Achten Sie darauf, daß die schützende Lampenab-deckung der Detektoren mit variablen und festen Wellenlängen während des Betriebs an ihrem Platz ist.
- Schauen Sie nicht direkt in die Flüssigkeitszellen im Detektor oder in die UV Lampe. Zum In-spizieren der Lichtquelle oder der Flüssigkeitszelle benutzen Sie immer einen wirksamen Augenschutz, wie er durch Borsilikatglas oder Polystyrol gewähr-leistet wird.

## **Verfügbarkeit von Ersatzteilen**

Es ist Varian's Grundsatz, Ersatzteile für alle Instrumente und die wichtigsten Zubehöre für einen Zeitraum von fünf (5) Jahren nach dem Fertigungs auslauf dieser Geräteserie verfügbar zu haben. Nach diesem Zeitraum von fünf (5) Jahren können Ersatzteile auf der Basis *solange vorhanden* bezogen werden. Als Ersatzteil werden hier solche elektrischen und mechanischen Einzelteile verstanden, die unter normalen Bedingungen ausfallen können. Beispiele sind Relais, Lampen, Temperaturfühler, Detektorelemente, Motoren usw. Metallbleche, Formteile oder Baugruppen und Gußteile, PC Boards und Funktionsmodule können normalerweise neuwertähnlich für eine brauchbare Lebensdauer instandgesetzt werden und werden deshalb nur auf der Basis *solange vorhanden* nach dem Produktionsauslauf des Instruments geliefert werden.

## **Serviceverfügbarkeit**

Varian bietet seinen Kunden auch nach dem Auslaufen der Garantie eine Vielfalt von Serviceleistungen an. Reparaturservice kann zu attraktiven Preisen über eine Wartungsvereinbarung oder nach Zeit- und Materialaufwand zur Verfügung gestellt werden. Technische Unterstützung und Training bieten wir Ihnen durch qualifizierte Chemiker sowohl auf einer Kontraktbasis als auch nach Ihren Erfordernissen an.

## **Varian Analytical Instruments Verkaufsbüros**

Für Verkaufs oder Servicehilfe und zum Bestellen von Teilen und Zubehören setzen Sie sich bitte mit Ihrem Varian Büro in Verbindung.

### **Argentina**

Buenos Aires  
Tel. +54.11.4.783.5306

### **Australia**

Mulgrave, Victoria  
Tel. +61.3.9566.1134

### **Austria**

Vösendorf bei Wien  
Tel. +43.1.699.9669

### **Benelux**

Bergen Op Zoom  
Tel. +31.164.282.800

### **Brazil and Latin America (S)**

São Paulo  
Tel. +55.11.820.0444

### **Canada**

Mississauga, Ontario  
Tel. 800.387.2216

### **China**

Beijing  
Tel. +86.106209.1727

### **Europe**

Middelburg, The Netherlands  
Tel. +31.118.671.000

### **France**

Les Ulis Cédex  
Tel. +33.1.6986.3838

### **Germany**

Darmstadt  
Tel. +49.6151.7030

### **India**

Mumbai  
Tel. +91.22.857.0787/88/89

### **Italy**

Torino  
Tel. +39.011.997.9111

### **Japan**

Tokyo  
Tel. +81.3.5232.1211

### **Korea**

Seoul  
Tel. +82.2.345.22452

### **Mexico and Latin America (N)**

Mexico City  
Tel. +52.5.523.9465

### **Russian Federation**

Moscow  
Tel. +7.095.937.4280

### **Spain**

Madrid  
Tel. +34.91.472.7612

### **Sweden**

Solna  
Tel. +46.8.445.1620

### **Switzerland**

Varian AG  
Tel. +41.848.803.800

### **Taiwan**

Taipei Hsien  
Tel. +886.2.698.9555

### **United Kingdom and Ireland**

Walton-on-Thames  
Tel. +44.1932.898000

### **Venezuela**

Valencia  
Tel. +58.41.257.608

### **United States**

Walnut Creek, California, USA  
Tel. +1.800.926.3000

(GC and GC/MS)  
Tel. +1.800.367.4752  
(LC)



**VARIAN**

[www.varianinc.com](http://www.varianinc.com)

# Informations et mesures de sécurité

## Instructions de fonctionnement

Ce manuel d'instruction est conçu pour aider l'utilisateur à créer des conditions opératoires lui permettant de faire fonctionner le matériel efficacement et en toute sécurité. Il contient entre autres certaines observations spéciales présentées sous forme de **NOTES**, **MISES EN GARDE** et **AVERTISSEMENTS**. Il est important de faire fonctionner ce matériel conformément aux instructions du présent manuel et à toute autre information émanant de Varian. S'adresser au bureau régional Varian pour toute question relative à la sécurité ou à l'utilisation correcte du matériel.

### NOTE

Information destinée à tirer le meilleur parti du matériel sur le plan des performances



### MISE EN GARDE

Attire l'attention sur une situation pouvant occasionner des dommages corporels légers et/ou des dégâts mineurs à l'appareil et indique comment remédier à cette situation



### AVERTISSEMENT

Attire l'attention sur une situation potentiellement dangereuse pouvant occasionner des dommages corporels importants et indique comment remédier à cette situation

## Symboles d'avertissement

## Description



### ATTENTION RISQUE D'ELECTROCUSSION

Exposition à des tensions dangereuses. Débrancher le matériel du secteur avant de dévisser les panneaux protecteurs.



### ATTENTION SUBSTANCES CHIMIQUES DANGER

Présence éventuelle de substances chimiques dangereuses. Eviter tout contact, en particulier lors du remplissage des réservoirs. Prendre les mesures de protection adéquates pour les yeux et la peau.



### ATTENTION RISQUE DE BRÛLURES

Exposition à des surfaces chaudes ou traitées cryogéniquement. Prendre les mesures de protection adéquates pour la peau.



### ATTENTION DANGER POUR LES YEUX

Les dommages causés aux yeux sont de deux natures différentes : jet de particules et de produits chimiques ou radiations UV. Utiliser des protections du visage et des yeux appropriées.



### ATTENTION RISQUE D'INCENDIE

Risque potentiel d'incendie. Se conformer aux instructions du manuel pour faire fonctionner le matériel en toute sécurité.



### ATTENTION RISQUE D'EXPLOSION

Risque potentiel d'explosion en raison du type de gaz ou de liquide utilisé.



### ATTENTION SOURCE DE RADIATION

Présence d'une source de radiation ionisante. Se conformer aux instructions du manuel pour faire fonctionner le matériel en toute sécurité.



### ATTENTION PIÈCES EN MOUVEMENT

Garder les mains et les doigts hors de portée.



## Précautions générales en matière de sécurité

Les pratiques suivantes garantissent une utilisation sans risques du matériel:

- Effectuer régulièrement des essais d'étanchéité de tous les conduits d'alimentation et de tous les tuyaux du système pneumatique.
- Ne pas travailler avec des conduits de gaz déformés ou percés. Installer les conduits de gaz à l'écart des allées et venues et à l'abri du chaud ou du froid.
- Conserver les solvants organiques dans des récipients à l'épreuve du feu, bien ventilés et portant mention de la nature de leur contenu, en particulier lorsque lesdits solvants sont toxiques et/ou inflammables.
- Ne pas accumuler les solvants de rebut. Les éliminer conformément à un programme agréé d'élimination des déchets et non via les égouts municipaux.

**NOTE:** Ce matériel a été testé conformément aux dispositions de la directive CME afin de pouvoir porter le sigle CE de l'Union européenne. Il en résulte qu'il peut être sensible à des niveaux de radiation/d'interférence ou à des fréquences se situant hors des limites testées.



Ce matériel est conçu pour effectuer des analyses chromatographiques d'échantillons préparés selon des méthodes appropriées. Il convient de le faire fonctionner avec les gaz et/ou les solvants adéquats et dans les limites des pressions, des débits et des températures maximales spécifiées dans le présent manuel.



Le client est tenu d'informer le service Varian d'assistance à la clientèle que son matériel a été utilisé pour l'analyse d'échantillons biologiques dangereux, radioactifs ou toxiques avant que n'en soit effectué la maintenance.

## Risques de chocs électriques

- Déconnecter le matériel de toute source d'alimentation avant d'en démonter les panneaux de protection, sous peine de s'exposer à des tensions dangereuses.
- En cas d'utilisation d'un cordon d'alimentation n'étant pas d'origine, s'assurer que celui-ci soit conforme à la polarité et au codage des couleurs décrits dans le manuel d'utilisation ainsi qu'à toutes les normes régionales de sécurité régissant le secteur de la construction.
- Remplacer les fusibles sautés par des fusibles de même type que ceux stipulés sur le panneau des fusibles ou dans le manuel d'utilisation.
- Remplacer les cordons d'alimentation défectueux ou dénudés par des cordons d'alimentation de même type.
- S'assurer que les sources de tension et la tension de secteur correspondent à la tension de fonctionnement du matériel.

## Bouteilles à gaz comprimé

- Ranger et manipuler les bouteilles à gaz comprimé avec précaution et conformément aux normes de sécurité.
- Fixer les bouteilles à gaz comprimé à un mur ou à une structure inamovible.
- Ranger et déplacer les bouteilles à gaz comprimé en position verticale. Avant de transporter les bouteilles à gaz comprimé, retirer leur régulateur.
- Ranger les bouteilles dans un endroit bien ventilé et à l'abri de la chaleur, des rayons directs du soleil, du gel ou des sources d'allumage.
- Marquer les bouteilles de manière à n'avoir aucun doute quant à leur contenu.
- N'utiliser que des connexions et régulateurs agréés.
- N'utiliser que des tuyaux de raccordement propres sur le plan chromatographique (Varian P/N 03-918326-00) et pouvant supporter des pressions sensiblement plus élevées que la plus haute pression de sortie du régulateur.

## Mesures de sécurité en CPG

### Système d'échappement

Les détecteurs CPG installés dans une pièce bien ventilée ne nécessitent pas de conduits spéciaux d'échappement excepté lorsqu'ils sont destinés à analyser des substances chimiques dangereuses. Lors de l'installation de tels conduits:

- N'utiliser que des conduits à l'épreuve du feu
- Installer un ventilateur à la sortie du conduit.
- Placer les orifices d'aspiration de manière à ce que les vibrations ou les mouvements d'air n'affectent pas le fonctionnement du détecteur.
- Vérifier périodiquement l'état du conduit.
- S'assurer que le laboratoire est correctement ventilé.

### Détecteurs à source radioactive

- Se conformer au manuel d'utilisation de l'ECD Ni<sup>63</sup>, en particulier à ses **NOTES, MISES EN GARDE ET AVERTISSEMENTS**.
- Effectuer les tests de décontamination radioactive décrits dans le manuel d'utilisation de l'ECD Ni<sup>63</sup>.
- Se conformer aux procédures et au calendrier des essais d'étanchéité.

### Risque de brûlures

Les zones des chromatographes à gaz chauffées ou traitées cryogéniquement peuvent rester très chaudes ou très froides durant une période plus ou moins longue après la mise hors tension du matériel. Pour éviter les brûlures, s'assurer que ces zones sont revenues à température ambiante ou utiliser un dispositif adéquat de protection des mains avant de les toucher.

## Mesures de sécurité en CPL

### Risques liés aux hautes pressions

En cas de rupture d'un tuyau ou en cas d'ouverture accidentelle d'une vanne alors que le système est sous pression, la pompe peut occasionner des dommages en expulsant à grande vitesse des jets de liquides volatiles et/ou toxiques.

- Mettre un masque de protection lors de l'injection des échantillons ou en effectuant les opérations de maintenance de routine.

- Ne jamais déconnecter un conduit de solvant ou une vanne sous pression. Arrêter préalablement la pompe et laisser la pression descendre à zéro.
- Utiliser des réservoirs incassables à 50-60 psi.
- Laisser l'enceinte du réservoir fermée lorsque le réservoir est sous pression.
- Se conformer aux **NOTES, MISES EN GARDE ET AVERTISSEMENTS** du manuel d'utilisation.

### Chromatographie Flash

L'utilisateur aura la connaissance des propriétés physico-chimiques des constituants de la phase mobile.

Eviter le contact direct des solvants avec les tuyaux en polyuréthane : certains solvants sont susceptibles de provoquer des faiblesses et des fuites avec risques d'explosion.

Tous les constituants du système devront être connectés à une source de courant commune et à une prise de terre commune. Cette prise de terre devra être fixe et non mobile.

Les solvants non-polaires peuvent produire de l'électricité statique lorsqu'ils passent au travers du système. Les bouteilles qui contiennent la phase mobile (incluant les tuyaux et les flacons de collecte de fractions) doivent être mises à la terre pour éliminer l'électricité statique.

Utiliser des appareils de mesure et de décharge d'électricité statique (par exemple des ioniseurs d'air) pour combattre la formation d'électricité statique.

### Radiations ultraviolettes

Les détecteurs CPL utilisant une source lumineuse ultraviolette comportent un écran destiné à se prémunir contre les expositions aux rayonnements.

Pour s'assurer une protection permanente:

- Vérifier que le couvercle de protection de la lampe des détecteurs opérant à des longueurs d'onde variables et fixes soit bien en place durant le fonctionnement du matériel.
- Ne pas regarder directement les cellules du détecteur ou la source d'UV. Se protéger systématiquement les yeux lors du contrôle de la source lumineuse ou des cellules, par exemple au moyen de verres borosilicatés ou en polystyrène.

## Disponibilité des pièces de rechange

La politique de Varian consiste à fournir des pièces de rechange pour tous les appareils et accessoires majeurs durant une période de cinq (5) ans après livraison de leur production finale. Les pièces de rechange ne sont fournies au terme de cette période de cinq (5) ans que suivant les disponibilités. Il faut entendre par pièces de rechange les pièces individuelles électriques ou mécaniques susceptibles de défaillance au cours de leur utilisation normale. Par exemple, les relais, les lampes, les sondes thermiques, les éléments de détecteur, les moteurs, etc. Les parties en tôles, les éléments ou assemblages structurels et les pièces de fonderie, les cartes à circuits imprimés et les modules fonctionnels sont normalement susceptibles d'être remis à l'état neuf pendant toute la durée de leur vie utile et ne sont dès lors fournies, au terme de la production finale des appareils, que suivant les disponibilités.

## Service d'assistance à la clientèle

Varian fournit divers services destinés à aider sa clientèle après expiration de la garantie: service de réparation sur base de contrats de maintenance à prix attractifs ou sur base d'accords à durée limitée portant sur du matériel spécifique; support technique et service de formation assurés par des chimistes qualifiés sur base contractuelle ou en fonction des besoins spécifiques.

## Points de vente des instruments analytiques Varian

Contactez votre point de vente régional Varian pour toute question commerciale ou de service d'assistance à la clientèle ou pour passer commande de pièces et de fournitures.

### Argentina

Buenos Aires  
Tel. +54.11.4.783.5306

### Australia

Mulgrave, Victoria  
Tel. +61.3.9566.1134

### Austria

Vösendorf bei Wien  
Tel. +43.1.699.9669

### Benelux

Bergen Op Zoom  
Tel. +31.164.282.800

### Brazil and Latin America (S)

São Paulo  
Tel. +55.11.820.0444

### Canada

Mississauga, Ontario  
Tel. 800.387.2216

### China

Beijing  
Tel. +86.106209.1727

### Europe

Middelburg, The Netherlands  
Tel. +31.118.671.000

### France

Les Ulis Cédex  
Tel. +33.1.6986.3838

### Germany

Darmstadt  
Tel. +49.6151.7030

### India

Mumbai  
Tel. +91.22.857.0787/88/89

### Italy

Torino  
Tel. +39.011.997.9111

### Japan

Tokyo  
Tel. +81.3.5232.1211

### Korea

Seoul  
Tel. +82.2.345.22452

### Mexico and Latin America (N)

Mexico City  
Tel. +52.5.523.9465

### Russian Federation

Moscow  
Tel. +7.095.937.4280

### Spain

Madrid  
Tel. +34.91.472.7612

### Sweden

Solna  
Tel. +46.8.445.1620

### Switzerland

Varian AG  
Tel. +41.848.803.800

### Taiwan

Taipei Hsien  
Tel. +886.2.698.9555

### United Kingdom and Ireland

Walton-on-Thames  
Tel. +44.1932.898000

### Venezuela

Valencia  
Tel. +58.41.257.608

### United States

Walnut Creek, California, USA  
Tel. +1.800.926.3000

(GC and GC/MS)

Tel. +1.800.367.4752  
(LC)



**VARIAN**

[www.varianinc.com](http://www.varianinc.com)

# Informazioni sulla Sicurezza

## Instruzioni per l'Uso

Questo manuale ha lo scopo di aiutare l'operatore ad utilizzare lo strumento in modo sicuro ed efficiente. Le considerazioni e le precauzioni speciali vengono presentate in questo manuale sotto forma di avvisi di **NOTA**, **CAUTELA** e **ATTENZIONE**. E' importante che lo strumento venga utilizzato rispettando le istruzioni fornite in questo manuale o che verranno fornite successivamente dalla Varian. Per ogni eventuale chiarimento sull'uso o sulla sicurezza, si prega di contattare la Varian di Leinì (TO).

### NOTA

Sono informazioni utili ad ottenere le prestazioni migliori da parte dello strumento.



### ATTENZIONE

Allerta l'operatore su situazioni che potrebbero causare ferite leggere e danni limitati allo strumento ed il modo di evitarle.



### ATTENZIONE

Allerta l'operatore su situazioni potenzialmente pericolose che possono causare danni molto seri ed il modo di evitarle.

#### Segnali di ATTENZIONE



##### ATTENZIONE

Pericolo di folgorazioni



##### ATTENZIONE

ESPOSIZIONE A  
SOSTANZA CHIMICHE



##### ATTENZIONE

Pericolo di scottature



##### ATTENZIONE

PERICOLO PER  
GLI OCCHI



##### ATTENZIONE

Pericolo di incendio



##### ATTENZIONE

Pericolo di esplosioni



##### ATTENZIONE

Pericolo di radiazioni



##### ATTENZIONE

Parti in movimento

#### Descrizione del Pericolo

Nello strumento sono presenti tensioni pericolose. Scollegare il cavo di alimentazione prima di togliere il pannello fissato con le viti.

Possono essere presenti composti chimici pericolosi. Evitare il contatto, specialmente quando si riempiono i contenitori. Usare protezioni opportune per la pelle e per gli occhi.

Pericolo di esposizione a superfici molto calde o raffreddate criogenicamente. Usare protezioni opportune per la pelle.

Particelle volanti, agenti chimici o radiazioni UV possono danneggiare gli occhi. Vanno quindi utilizzate le opportune protezioni per gli occhi e per il volto.

Pericolo potenziale di incendio. Seguire le istruzioni del manuale per lavorare con una maggiore sicurezza.

C'è pericolo di esplosioni a causa del tipo di gas o liquido utilizzato.

E' presente una radiazione ionizzante. Seguire le istruzioni del manuale per lavorare con una maggiore sicurezza.

Non tenere le mani o le dita vicino.



## **Norme di Sicurezza**

Per lavorare in modo sicuro sullo strumento, Vi consigliamo si adottare le seguenti procedure.

- Verificare periodicamente che non ci siano perdite sulle linee e sui raccordi pneumatici.
- Evitare che le linee dei gas vengano piegate o forate. Le linee vanno posizionate in modo tale da non essere calpestate e lontane da sorgenti o troppo calde o troppo fredde.
- I solventi organici vanno conservati in armadi speciali antiincendio, ventilati e con indicazioni chiare sul contenuto di materiali tossici e/o infiammabili.
- Non accumulare i solventi utilizzati. Adottare un programma regolare di smaltimento, ma mai nelle acque di scarico.

**AVVERTENZA:** Questo strumento è stato testato secondo le Direttive EMC allo scopo di poter utilizzare il Marchio CE della Comunità Europea. Questo strumento può essere suscettibile a radiazioni/interferenze o frequenze che non sono entro i limiti collaudati.



### **ATTENZIONE**

Questo strumento è progettato per l'analisi cromatografica di campioni opportunamente preparati. Deve essere utilizzato usando gas e solventi adatti a questo scopo ed entro i limiti massimi di pressione, flusso e temperatura riportati in questo manuale. Se lo strumento non viene utilizzato secondo le modalità specificate dal costruttore, le condizioni di sicurezza previste potranno non essere sufficienti.



### **ATTENZIONE**

E' responsabilità del Cliente informare il Servizio Tecnico Varian, prima di qualsiasi intervento di riparazione, se lo strumento è stato utilizzato per l'analisi di campioni biologicamente pericolosi, radioattivi o tossici.

## **Pericoli Elettrici**

- Prima di togliere i pannelli di protezione, scollegare lo strumento da tutte le alimentazioni elettriche in modo da evitare l'esposizione a voltaggi potenzialmente pericolosi.
- Quando si rende necessario sostituire il cavo di alimentazione, assicurarsi che il nuovo cavo rispetti sia le codifiche di colore e di polarità riportate nel manuale di istruzioni che quelle stabilite dalle norme di sicurezza del laboratorio.
- Sostituire i fusibili bruciati solo con fusibili che abbiano le stesse caratteristiche; queste ultime sono riportate sul pannello dei fusibili e/o nel manuale di istruzioni.
- Sostituire immediatamente i cavi di alimentazione difettosi o consumati con cavi dello stesso tipo e con le stesse caratteristiche.
- Assicurarsi che il voltaggio del pannello di alimentazione corrisponda a quello dello strumento da collegare.

## **Bombole dei Gas**

- Occorre prestare molta attenzione quando si spostano bombole di gas compressi. Rispettare tutte le norme di sicurezza.
- Assicurare le bombole ad una parete o ad una struttura fissa.
- Spostare e conservare le bombole sempre in posizione verticale. Togliere i manometri prima di spostare le bombole.
- Conservare le bombole in un'area ben ventilata, non infiammabile, lontana da sorgenti di calore, non esposta a temperature troppo fredde o alla luce diretta del sole.
- Evidenziare in modo chiaro e che non lasci dubbi il contenuto di ogni bombola.
- Usare solo manometri e raccordi di qualità.
- Usare solo tubazioni cromatograficamente pulite (Numero di Parte Varian 03-918326-00) e calibrate per pressioni superiori a quella massima di uscita dal manometro.

## Procedure di Sicurezza in GC

### Scarico dei Gas

Per i rivelatori GC non è richiesto alcun sistema particolare di scarico dei gas, se lo strumento è installato in una stanza ben ventilata e se non viene utilizzato per l'analisi di sostanze chimiche pericolose. Se si deve installare un sistema di scarico dei gas:

- Usare condutture non infiammabili
- Installare un aspiratore in uscita
- Posizionare la presa d'aria in modo che le vibrazioni e il movimento dell'aria non disturbino il rivelatore.
- Eseguire verifiche periodiche per garantire un funzionamento corretto.
- Garantire una buona ventilazione nel laboratorio.

### Rivelatori a Sorgente Radioattiva

- Leggere e rispettare tutte gli avvisi di **NOTA**, **CAUTELA** e **ATTENZIONE** riportati nel manuale del rivelatore ECD al Ni<sup>63</sup>.
- Eseguire tutti i test di contaminazione radioattiva rimovibile descritti nel manuale dell'ECD al Ni<sup>63</sup>.
- Rispettare tutte le procedure e le scadenze di verifica per eventuali perdite.

### Pericolo di Scottature

Le zone calde o raffreddate criogenicamente del gascromatografo possono mantenere la loro temperatura per parecchio tempo, dopo aver spento lo strumento. Per evitare scottature, assicurarsi che le zone riscaldate o raffreddate siano a temperatura ambiente oppure indossare delle protezioni adeguate prima di toccare tali superfici.

## Procedure di Sicurezza in LC

### Pericolo di Alte Pressioni

In caso di rottura di una linea o di apertura accidentale di una valvola, quando il sistema è sotto pressione, la pompa può liberare liquidi tossici e/o volatili molto pericolosi.

- E' opportuno adottare un sistema di protezione del viso quando si inietta il campione o si esegue una manutenzione routinaria del sistema.

- Non smontare mai una linea del solvente od una valvola quando il sistema è sotto pressione. Fermare prima la pompa ed aspettare che la pressione scenda a zero.
- Usare dei contenitori per solventi infrangibili ed in grado di lavorare a 50-60 psi.
- Quando i contenitori sono sotto pressione, usare una protezione esterna.
- Leggere e rispettare tutti gli avvisi di **NOTA**, **CAUTELA** e **ATTENZIONE**.

### Cromatografia Flash

L'operatore deve conoscere le proprietà fisico-chimiche delle componenti della fase mobile.

I solventi non vanno messi in contatto diretto con il tubo di erogazione in poliuretano, dal momento che alcuni solventi possono causare indebolimento e perdite con possibili scoppi.

Tutte le componenti del sistema vanno collegate ad una fonte di alimentazione e ad una messa a terra comuni. E' meglio che per quest'ultima venga utilizzata una spina con polo di terra.

I solventi non-polari possono sviluppare una carica statica quando vengono pompati attraverso il sistema. Tutti i recipienti che contengono la fase mobile (inclusi i tubi e i recipienti di raccolta) devono avere una messa a terra per disperdere l'elettricità statica.

Vanno utilizzati dispositivi di misurazione e scarico (ad esempio ionizzatori d'aria) per evitare l'aumento di elettricità statica.

### Radiazioni Ultraviolette

I rivelatori di cromatografia liquida che usano sorgenti a luce ultravioletta montano degli schermi di protezione per evitare che gli operatori siano esposti a radiazioni pericolose.

Per una protezione sicura:

- Assicurarsi che i coperchi delle lampade dei rivelatori a lunghezza fissa e variabile siano sempre al loro posto, quando si lavora.
- Non guardare mai direttamente dentro le celle o alla sorgente di luce UV. Quando si vuole ispezionare la lampada o le celle, usare sempre delle protezioni adatte per gli occhi, quali vetro in borosilicato e polistirolo.

## Disponibilità delle Parti di Ricambio

E' politica della Varian il fornire le parti di ricambio per lo strumento ed i suoi accessori per un periodo di cinque (5) anni a partire dalla data di produzione dell'ultima unità della serie. Le parti di ricambio saranno disponibili anche dopo questo periodo di cinque (5) anni ma solo in base alla disponibilità delle stesse. Per parti di ricambio si intendono i componenti elettrici e meccanici soggetti ad usura durante l'uso, in condizioni normali, dello strumento. Come esempio, citiamo i relay, le lampade, i probe di temperatura , i componenti del rivelatore, i motorini, ecc. Le parti strutturali o da fusione, le schede elettroniche ed i moduli funzionali possono essere ricostruiti e rimessi a nuovo durante tutto il loro periodo di vita e perciò sarà possibile acquistarli, dopo la produzione dell'ultima unità delle serie, solo in base alla loro disponibilità.

## Servizi Tecnico

La Varian, alla scadenza del periodo di garanzia, è in grado di fornire ai suoi clienti un'ampia scelta di opzioni. Le riparazioni possono essere effettuate sulla base di contratti di manutenzione particolarmente vantaggiosi od in base ad una tariffa oraria piu' il costo delle parti. A richiesta, si possono avere corsi per operatori sia sotto forma di contratto che a tariffe da concordare.

## Uffici Vendite della Divisione Strumenti Analitici della Varian

Per informazioni relative alla Vendita, al Servizio Tecnico o all'acquisto di Parti di ricambio, si prega di contattare l'ufficio Varian piu' vicino.

### Argentina

Buenos Aires  
Tel. +54.11.4.783.5306

### Australia

Mulgrave, Victoria  
Tel. +61.3.9566.1134

### Austria

Vösendorf bei Wien  
Tel. +43.1.699.9669

### Benelux

Bergen Op Zoom  
Tel. +31.164.282.800

### Brazil and Latin America (S)

São Paulo  
Tel. +55.11.820.0444

### Canada

Mississauga, Ontario  
Tel. 800.387.2216

### China

Beijing  
Tel. +86.106209.1727

### Europe

Middelburg, The Netherlands  
Tel. +31.118.671.000

### France

Les Ulis Cédex  
Tel. +33.1.6986.3838

### Germany

Darmstadt  
Tel. +49.6151.7030

### India

Mumbai  
Tel. +91.22.857.0787/88/89

### Italy

Torino  
Tel. +39.011.997.9111

### Japan

Tokyo  
Tel. +81.3.5232.1211

### Korea

Seoul  
Tel. +82.2.345.22452

### Mexico and Latin America (N)

Mexico City  
Tel. +52.5.523.9465

### Russian Federation

Moscow  
Tel. +7.095.937.4280

### Spain

Madrid  
Tel. +34.91.472.7612

### Sweden

Solna  
Tel. +46.8.445.1620

### Switzerland

Varian AG  
Tel. +41.848.803.800

### Taiwan

Taipei Hsien  
Tel. +886.2.698.9555

### United Kingdom and Ireland

Walton-on-Thames  
Tel. +44.1932.898000

### Venezuela

Valencia  
Tel. +58.41.257.608

### United States

Walnut Creek, California, USA  
Tel. +1.800.926.3000

(GC and GC/MS)

Tel. +1.800.367.4752  
(LC)



**VARIAN**

[www.varianinc.com](http://www.varianinc.com)

# Instrucciones de Seguridad

## Instrucciones de Operación

Este Manual de Instrucciones está diseñado para ayudarle a establecer las condiciones de operación que le permitan operar su instrumento de forma segura y eficaz. Así mismo, se describen consideraciones especiales ó precauciones, que aparecen en forma de **NOTA**, **PRECAUCIÓN**, y **ATENCIÓN** como se indica más abajo. Es importante que utilice el instrumento de acuerdo con este Manual de Operación y cualquier otra información que le proporcione Varian. Remita a la Oficina Local de Varian cualquier cuestión que tenga respecto al correcto uso de su equipo.

### NOTA

Información para ayudarle a obtener unas prestaciones óptimas de su instrumento.

### ! PRECAUCIÓN!

Le alerta de situaciones que pueden causar daños moderados a la salud ó al equipo, y cómo evitar esas situaciones.

### ! ATENCIÓN

Le alerta de potenciales situaciones peligrosas que pueden causar serios daños, y cómo evitar esas situaciones.

#### Símbolo



**ATENCIÓN**  
PELIGRO DE  
DESCARGA ELÉCTRICA



**ATENCIÓN**  
PELIGRO QUÍMICO



**ATENCIÓN**  
PELIGRO DE  
QUEMADURAS



**ATENCIÓN**  
PELIGRO PARA LOS OJOS



**ATENCIÓN**  
PELIGRO DE FUEGO



**ATENCIÓN**  
PELIGRO DE EXPLOSIÓN



**ATENCIÓN**  
PELIGRO DE RADIACIÓN



**ATENCIÓN**  
PARTES EN MOVIMIENTO

#### Descripción

El instrumento utiliza voltajes peligrosos. Desconecte el interruptor general antes de retirar los paneles atornillados.

Peligro de productos químicos. Evite el contacto, especialmente cuando rellene los depósitos. Utilice protección de ojos y piel.

Superficies posiblemente calientes ó frías (criogénico). Utilice protección para la piel.

Las partículas volátiles, productos químicos o radiación UV pueden causar daños en los ojos. Usar las debidas protecciones para la cara y los ojos.

Peligro potencial de fuego. Siga las instrucciones del Manual de Operación para su seguro funcionamiento.

Peligro potencial de explosión debido al tipo de gas ó líquido empleado.

Peligro por Fuente de radiación. Siga las instrucciones del Manual de Operación para su seguro funcionamiento.

Mantenga alejados los dedos y las manos.



## Precauciones Generales de Seguridad

Siga estas indicaciones de seguridad para una correcta operación del equipo.

- Realice verificaciones periódicas de fugas en todas las líneas de suministro y tuberías.
- No permita que las líneas de gas se doblen ó pinchen. Manténgalas alejadas de zonas de paso y del calor ó frío excesivo.
- Guarde los disolventes orgánicos en cabinas ventiladas, a prueba de fuego, y etiquetadas para que puedan ser fácilmente identificadas como material tóxico y/o inflamable.
- No acumule disolventes inservibles. Deseche todo el material inservible a través de un programa especial de desechos y no a través del sistema convencional.

**NOTA:** Este instrumento ha sido testado bajo las normas de la Directiva EMC según requerimientos de la Marca CE de la Unión Europea. Por lo tanto, este equipo puede ser sensible a niveles de radiaciones / interferencias ó frecuencias que no estén incluidas dentro de los límites testados.



Este instrumento está diseñado para análisis cromatográfico de muestras preparadas apropiadamente. Debe ser operado usando gases y/o disolventes apropiados y con unos niveles máximos de presión, flujos y temperaturas, según se describe en este manual.



El Usuario tiene la obligación de informar al Servicio Técnico de Varian cuando el instrumento vaya a ser empleado para análisis de muestras peligrosas de origen biológico, radioactivo ó tóxico, antes de comenzar a realizar cualquier análisis.

## Peligros Eléctricos

- Desconecte el instrumento de todos las conexiones eléctricas a la red antes de retirar los paneles para evitar la posible exposición a peligrosos voltajes.
- Cuando sea necesario emplear una clavija eléctrica no original, asegurese de colocar los cables de acuerdo con el código de colores y polaridades descritos en el manual y los códigos de seguridad de la red eléctrica.
- Sustituya los fusibles fundidos con fusibles del tipo y tamaño estipulados en el panel de fusibles ó en el manual.
- Sustituya los cables deteriorados inmediatamente con cables del mismo tipo y graduación.
- Asegurese de que los valores de las líneas de electricidad se ajustan a los valores para los que el Instrumento ha sido preparado.

## Botellas de Gas Comprimido

- Guarde y maneje las botellas de gas con cuidado y de acuerdo con las normas de seguridad.
- Asegure las botellas a una estructura inmóvil ó a la pared.
- Guarde y mueva las botellas en posición vertical. Retire los reguladores antes de transportarlas.
- Guarde las botellas en un área ventilada, lejos de fuentes de calor, de luz solar directa y de temperaturas extremadamente bajas.
- Identifique las botellas claramente para evitar cualquier duda sobre su contenido.
- Utilice sólamente reguladores y conexiones aprobadas.
- Utilice sólo tubos de conexión cromatográficamente limpios (Varian p/n 03-918326-00) y que tengan una graduación de presión significativamente mayor que la mayor presión del regulador.

# GC Prácticas de Seguridad

## Sistema de Extracción

No se necesita un sistema de extracción para los detectores GC instalados en un laboratorio bien ventilado, excepto cuando se analicen muestras químicas peligrosas. Si instala un sistema de extracción:

- Utilice conductos a prueba de fuego.
- Instale un ventilador al final del sistema.
- Instale entradas de aire cuya vibración no afecte al trabajo del detector.
- Compruebe periódicamente el correcto funcionamiento del sistema.
- Asegurese de una correcta ventilación del laboratorio.

## Detectores con fuentes radioactivas

- Lea con cuidado y cumpla todas las **NOTAS**, **PRECAUCION**, y **ATENCION** del Manual del Detector Ni<sup>63</sup> ECD.
- Realice los test de contaminación radioactiva descritos en el Manual del Detector Ni<sup>63</sup> ECD.
- Cumpla con los plazos y procedimientos de test de fugas.

## Peligro de Quemaduras

Las zonas de calor ó frío (criogénicas) del Cromatógrafo de Gases pueden permanecer calientes ó frías durante bastante tiempo después de apagar el instrumento. Para evitar quemaduras asegúrese de que todas las áreas que se calienten ó enfrién han vuelto a la temperatura ambiente, ó protejase adecuadamente las manos, antes de tocar las superficies potencialmente calientes ó frías.

# LC Prácticas de Seguridad

## Peligro de Alta Presión

Si se rompe una línea de presión, ó se abre una válvula de seguridad accidentalmente bajo presión, la bomba puede generar líquidos a alta presión potencialmente peligrosos, produciendo un chorro a alta velocidad de líquidos volátiles y/o tóxicos.

- Lleve protección facial cuando inyecte muestras ó realice mantenimiento de rutina.

- Nunca abra una línea ó una válvula bajo presión. Apague la bomba antes y deje que la presión baje a cero.
- Utilice depósitos irrompibles que sean capaces de operar a 50-60 psi.
- Mantenga cerrada la junta del depósito cuando se haye bajo presión.
- Lea y cumpla todas las **NOTA**, **PRECAUCION**, y **ATENCION** del manual.

## Cromatografía Flash

El operador debe familiarizarse con las propiedades físico-químicas de los componentes de la fase móvil.

Alejar los disolventes del contacto directo con los tubos de poliuretano ya que ciertos disolventes pueden causar reblandecimiento de los tubos o posibles fugas con riesgo de explosión.

Todos los componentes del sistema deben estar conectados a un enchufe común con toma de tierra común. Esta toma de tierra debe ser una toma de tierra verdadera en lugar de flotante.

Los disolventes no-polares pueden originar carga estática cuando son bombeados por el sistema. Todos los recipientes que contienen fase móvil (incluyendo los tubos y los recipientes de recogida) deben estar conectados a tierra para disipar la electricidad estática.

Utilizar medidores de carga estática y los debidos dispositivos de descarga (por Ej., ionizadores de aire) para salvaguardarse contra la creación de electricidad estática.

## Radiación Ultravioleta

Los detectores del Cromatógrafo de Líquidos que utilizan una fuente de luz ultravioleta disponen de protección para prevenir exposiciones radioactivas al personal.

Para una correcta protección:

- Asegúrese de que las cubiertas de protección de la lámpara de los detectores está correctamente situada durante su funcionamiento.
- No mire directamente a las celdas del detector ó a la fuente de luz UV. Cuando inspeccione la fuente de luz ó la celda, utilice siempre una protección para los ojos como gafas de borosilicato ó poliestireno.

## Disponibilidad de Recambios

Es Política de Varian disponer de Recambios para cualquier instrumento y la mayoría de los accesorios por un periodo de cinco (5) años después del último instrumento fabricado. Los recambios durante esos cinco años estarán disponibles, pero siempre bajo el sistema “*Según disponibilidad*”. Los Recambios están definidos como todas aquellas partes individuales mecánicas ó eléctricas que son susceptibles de fallo durante su normal proceso de operación. Por ejemplo, relés, lámparas, sondas de temperatura, elementos del detector, motores, etc. Las planchas de metal, partes de la estructura, placas de circuitos integrados, y otros módulos funcionales son normalmente susceptibles de reparación y por lo tanto sólo estarán disponibles bajos el sistema “*Según disponibilidad*” después del último instrumento fabricado.

## Disponibilidad de Servicio

Varian ofrece una gran variedad de sistemas de Servicio para mantener el soporte a sus usuarios tras el periodo de garantía. El Soporte de Servicio se ofrece a través de atractivos Contratos de Servicio ó bajo un sistema de facturación de mano de obra y materiales. El mantenimiento y el entrenamiento se realiza por ingenieros cualificados bajo Contrato ó petición.

## Oficinas de Instrumentación Analítica Varian

Para cualquier consulta sobre Instrumentación Analítica, Servicio Técnico ó Recambios y Accesorios, contacte con su oficina local:

### Argentina

Buenos Aires  
Tel. +54.11.4.783.5306

### Australia

Mulgrave, Victoria  
Tel. +61.3.9566.1134

### Austria

Vösendorf bei Wien  
Tel. +43.1.699.9669

### Benelux

Bergen Op Zoom  
Tel. +31.164.282.800

### Brazil and Latin America (S)

São Paulo  
Tel. +55.11.820.0444

### Canada

Mississauga, Ontario  
Tel. 800.387.2216

### China

Beijing  
Tel. +86.106209.1727

### Europe

Middelburg, The Netherlands  
Tel. +31.118.671.000

### France

Les Ulis Cédex  
Tel. +33.1.6986.3838

### Germany

Darmstadt  
Tel. +49.6151.7030

### India

Mumbai  
Tel. +91.22.857.0787/88/89

### Italy

Torino  
Tel. +39.011.997.9111

### Japan

Tokyo  
Tel. +81.3.5232.1211

### Korea

Seoul  
Tel. +82.2.345.22452

### Mexico and Latin America (N)

Mexico City  
Tel. +52.5.523.9465

### Russian Federation

Moscow  
Tel. +7.095.937.4280

### Spain

Madrid  
Tel. +34.91.472.7612

### Sweden

Solna  
Tel. +46.8.445.1620

### Switzerland

Varian AG  
Tel. +41.848.803.800

### Taiwan

Taipei Hsien  
Tel. +886.2.698.9555

### United Kingdom and Ireland

Walton-on-Thames  
Tel. +44.1932.898000

### Venezuela

Valencia  
Tel. +58.41.257.608

### United States

Walnut Creek, California, USA  
Tel. +1.800.926.3000

(GC and GC/MS)

Tel. +1.800.367.4752  
(LC)



**VARIAN**

[www.varianinc.com](http://www.varianinc.com)

# Contents

<b>Introduction.....</b>	<b>5</b>
<b>Functional Description.....</b>	<b>7</b>
Overview.....	7
Transfer Line .....	9
Analyzer.....	10
Internal Ionization Configuration .....	10
External Ionization Configuration .....	12
Hybrid Configuration .....	14
Ion Trap .....	14
Detector.....	16
Vacuum System.....	17
Vacuum Manifold .....	17
Foreline Pump .....	17
Turbomolecular Vacuum Pump .....	18
Ion Gauge.....	18
Thermocouple Gauge .....	19
Pneumatics .....	19
Helium Flow .....	20
Calibration Gas Flow .....	20
The CI Reagent Gas Flow.....	20
Electronics .....	21
Controller.....	22
Power Board .....	23
Manifold Electronics .....	24
RF Generator Assembly.....	24
Ion Detection Board .....	25
Ion Amplifier .....	25
Electronic Flow Control .....	25
Power Input Subsystem and Turbomolecular Pump Controller.....	25
Main Power Circuit .....	26
<b>Periodic Maintenance.....</b>	<b>27</b>
Procedure Interval .....	27
Checking Foreline Pump Oil Level and Oil Condition .....	27
Changing Foreline Pump Oil .....	27
Flushing the Pump Oil.....	30
Changing the Oil Mist Cartridge .....	31
Checking Cooling Fans .....	32
<b>MS Maintenance Procedures.....</b>	<b>33</b>
General Recommendations .....	33
Recommended Tools and Materials.....	33

Common Procedures .....	34
Turning Off the Mass Spectrometer .....	34
Turning Off the Mass Spectrometer with Nitrogen Purge .....	36
Moving the Mass Spectrometer Away From the GC .....	37
Removing the Analyzer Assembly .....	37
Removing the Source/Ion Trap Assembly .....	40
Reinstalling the Source/Ion Trap Assembly .....	41
Reinstalling the Analyzer Assembly .....	44
Turning On the Mass Spectrometer .....	46
Checking the Vacuum Status.....	48
Baking Out the Mass Spectrometer .....	49
Checking Ion Trap Operation.....	49
Cleaning Procedures .....	50
Cleaning the External Source .....	50
Cleaning the Internal Ionization Assembly.....	60
Cleaning Ion Trap Components .....	64
Replacing a GC Column.....	71
Removing the Capillary Column from the System .....	71
Installing a New Capillary Column in the System .....	72
Replacing Consumable Components .....	75
Replacing External Source Filaments .....	75
Conditioning the Filaments.....	77
Replacing Internal Source Filaments .....	77
Replacing the Electron Multiplier .....	79
Replacing the Damping Gas Getter .....	81
Replacing the Turbomolecular Pump.....	81
Filling the Calibration Compound Vial .....	82
Changing Operational Configuration .....	83
Changing from Internal to External Configuration.....	84
Changing from External to Internal Configuration.....	84
Changing from Internal to Hybrid Configuration .....	84
Changing from External to Hybrid Configuration .....	84
Changing from Hybrid Mode to External Configuration .....	84
Changing from Hybrid Mode to Internal Configuration .....	84
Switching Between External and Internal Sources .....	84
Changing the Transfer line Position from External to Internal .....	85
Changing the Transfer line Position from Internal to External .....	87
Installing or Removing the Hybrid Plug .....	89
<b>Chemical Ionization Options .....</b>	<b>91</b>
Introduction .....	91
Internal Configuration CI .....	91
External Configuration CI .....	91
Hybrid Configuration CI.....	92
Installing CI Reagent Gas.....	92
CI Reagent Gas Requirements .....	93
Setting Up the CI Reagent Gas Supply .....	93
Checking the Reagent Gas Plumbing for Leaks .....	96
Setting Flows of CI Reagents in Internal Configuration.....	97
Internal Mode Default Parameters for CI Reagents .....	97
External Mode Default Parameters for CI Reagents .....	97
Ion Intensities for Standard CI Reagents .....	98
Setting Flows of CI Reagents in External Configuration .....	98
Setting Flows of CI Reagents in Hybrid Configuration .....	98
The Liquid CI Inlet Option .....	99

Filling/Refilling Reservoir Bulb .....	99
Switching from Liquid to Gaseous CI Reagent Operation .....	100
<b>Troubleshooting .....</b>	<b>101</b>
How to Isolate a GC/MS Problem.....	101
Checking the Data System .....	101
Checking the GC .....	101
Checking the Mass Spectrometer .....	101
Troubleshooting Problems with Spectra.....	102
No Spectrum Appears.....	102
Loss of High Mass Peaks.....	103
Part of the Spectrum is Missing .....	103
Poor Resolution with Acceptable Air and Water Levels.....	104
Troubleshooting High Baseline at High Masses .....	104
Checking for Leaks .....	105
Establishing Conditions Required for Leak Checks.....	105
Fixing a Large Air Leak .....	106
Fixing a Small-To-Moderate Air Leak .....	107
Checking GC Connections.....	107
Troubleshooting Air Leaks Using Leak Detection Gas .....	107
Fixing High Water Levels.....	108
GC Troubleshooting.....	109
Using the COLTEST Sample for Troubleshooting .....	109
Running the COLTEST Sample .....	109
Troubleshooting Common Chromatographic Problems.....	110
Correcting Solvent Tailing or Broadening Problems.....	110
Correcting Tailing Sample Peaks for Particularly Active Components .....	111
Correcting Low Response and Severe Tailing with High Boiling Point Compounds .....	111
Correcting Leading Sample Peaks (Reverse Tailing) .....	111
Correcting Poor Resolution <sup>1</sup> .....	111
Lack of Peak Size Reproducibility .....	112
Correcting Peak Splitting (Particularly for Low Boilers) .....	112
Correcting Extra, Unexpected Peaks in the Chromatogram.....	112
Correcting Retention Time Differences Between Runs .....	113
<b>Miscellaneous Procedures and Instructions.....</b>	<b>115</b>
Other Documents.....	115
Site Requirements .....	115
Site Preparation .....	115
Power .....	115
Quality of Power .....	118
Operating Environment.....	118
Temperature.....	118
Humidity .....	119
Exhaust System .....	119
Gas Requirements.....	119
Helium - GC Carrier Gas.....	119
Methane, Isobutane, Ammonia - CI Reagent Gases (with CI option only).....	120
Cryogenics .....	120
How to Install the 4000 MS.....	120
How to Move the 4000 MS .....	121
Parts and Supplies.....	122
Electronics.....	122
Pneumatics .....	122

Analyzer, Attached to Top Flange.....	122
Analyzer, Attached to Manifold .....	124
Chemical Ionization.....	124
Vacuum .....	124
O-Rings .....	125
Miscellaneous/Other .....	125
Test Samples .....	126
Varian Service .....	127

# Introduction

This hardware manual is composed of six sections. The first section is a functional description describing the operating principles of the spectrometer and details of the subsystems that make up the instrument. The next two sections describe the various maintenance procedures that need to be carried out to keep the instrument in proper working condition. The fourth section describes the installation and set up of the Chemical Ionization hardware. The fifth section provides troubleshooting procedures for resolving problems that may be encountered when using the instrument. The final section provides miscellaneous information including site requirements, installation instructions and parts lists.

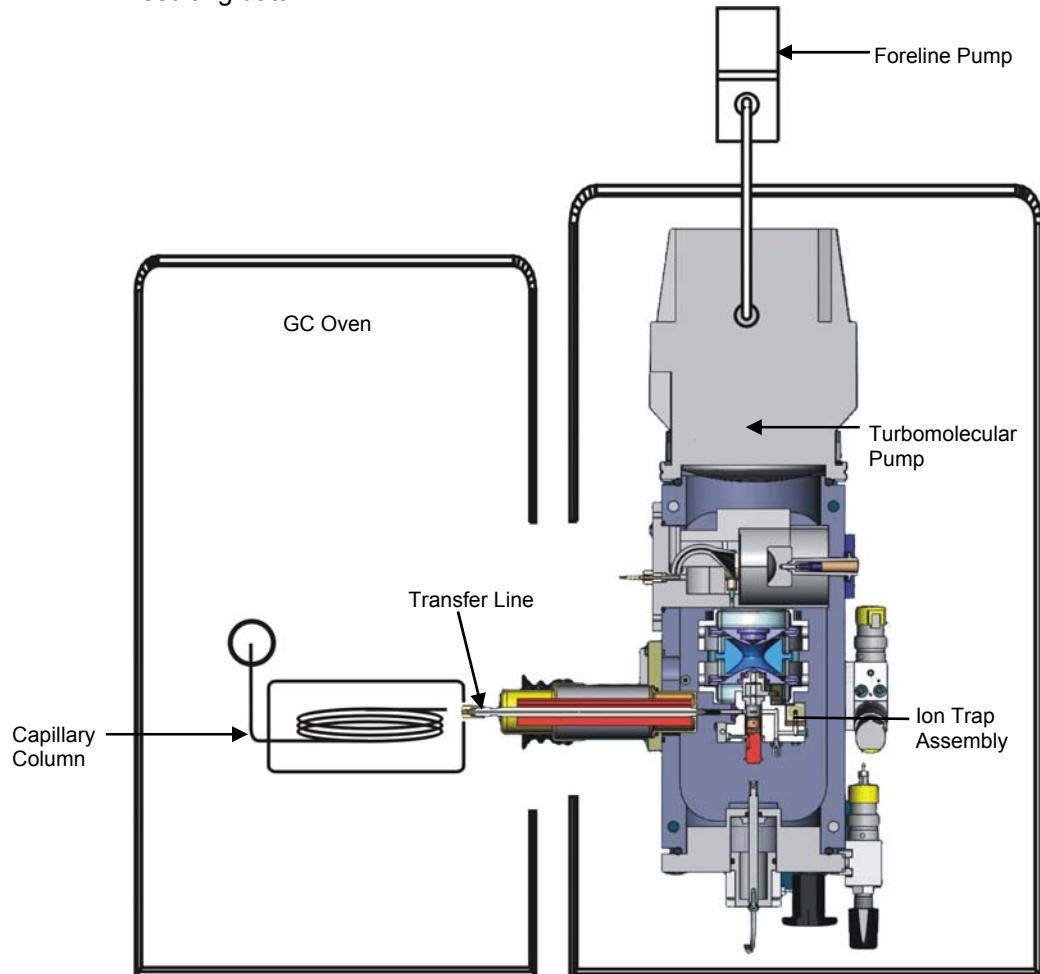
The manual is geared towards online use, particularly the Maintenance section that contains extensive links to common procedures.



# Functional Description

## Overview

Each subsystem of the 4000 Mass Spectrometer is described. The mass spectrometer is like an analyzer contained in a vacuum manifold surrounded by electronics components that drive the analyzer operation and acquire the resulting data.



*System Block Diagram (Shown in External Ionization Mode)*

Samples are first injected into the GC either manually or by way of an autosampler. The sample is vaporized and the gas goes through a column in the GC oven. After separation in the column, the sample enters the mass spectrometer through a heated transfer line. The MS analyzer consists of three parts: the source, ion trap, and detector. The samples flow from the transfer line into either an external source, where the sample is ionized, or directly into the ion trap for ionization. Once ionized, the ions are stored in the ion trap where they are systematically ejected for analysis. After ion ejection, the detector (consisting of a conversion dynode and electron multiplier) senses the ions.

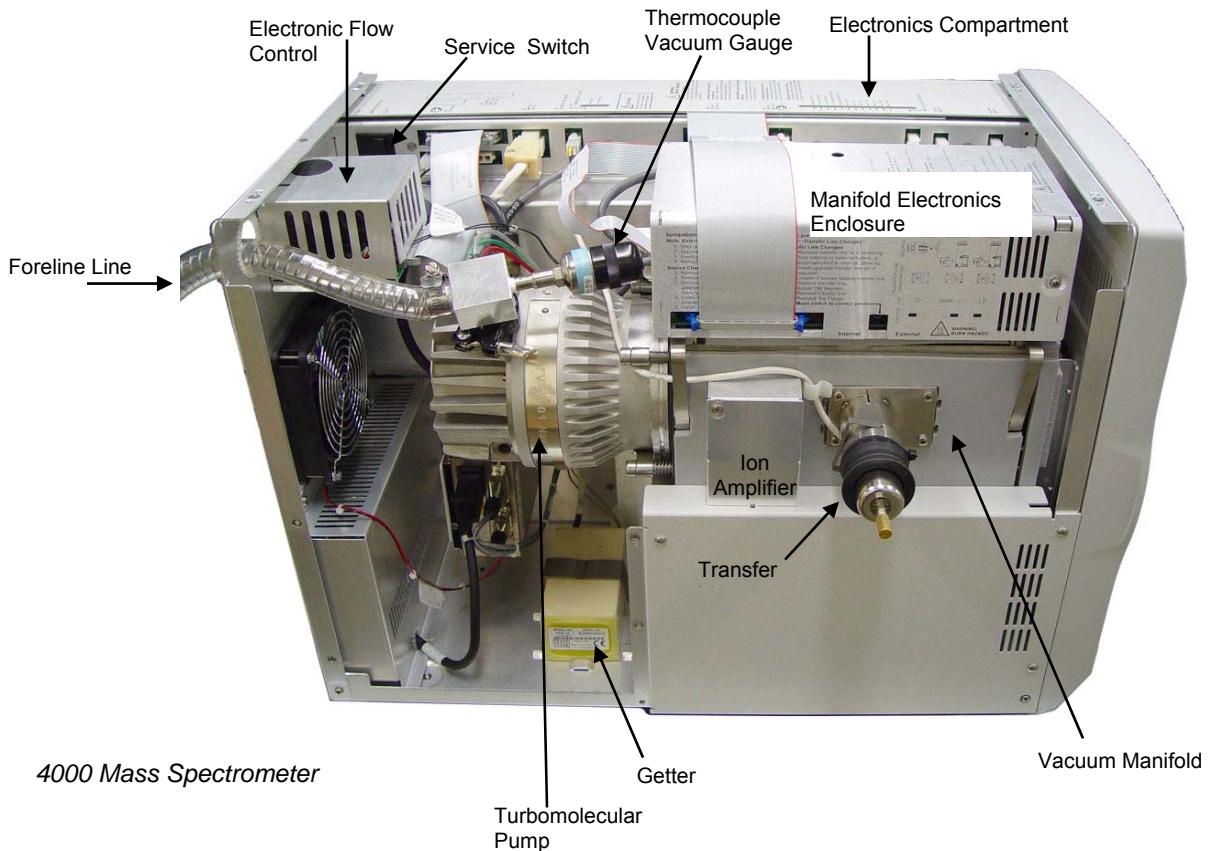
The vacuum manifold maintains the necessary vacuum conditions for proper analyzer operation. A turbomolecular pump and foreline pump create the necessary vacuum in the manifold. Various pneumatics components feed required gases into the vacuum manifold. An ion gauge and thermocouple gauge measure the vacuum levels in the manifold and foreline respectively.

Physically the vacuum manifold is mounted on top of an RF coil assembly. RF generation and ion detection electronics components are placed around the RF coil assembly. Some source and trap related electronics components reside in an enclosure mounted to the vacuum manifold top flange. A system controller and power board are contained in an enclosure formed by a central bulkhead and outer cover.



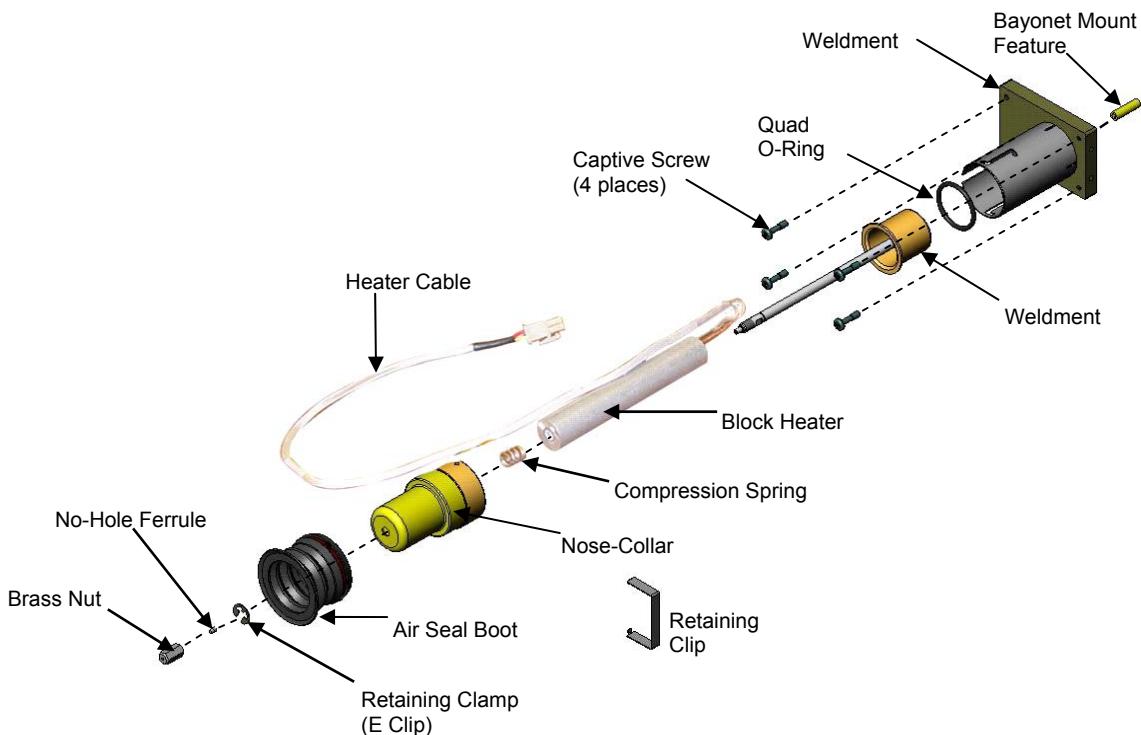
### WARNING: SHOCK HAZARD

**HIGH VOLTAGES INSIDE.** No user serviceable parts under screw-attached covers. Contact your local Varian, Inc. service representative for instrument repair and service.



## Transfer Line

A stainless steel tube transfer line directly couples the GC to the mass spectrometer. The purpose of the transfer line is to keep the GC column warm as the column enters the mass spectrometer to avoid condensation of the sample, which could result in tailing. One end of the transfer line enters a hole in the right side of the GC before passing into the GC oven. The other end enters the vacuum manifold in one of two positions, depending on where the sample is to be ionized. If the sample is to be ionized in the external source, the transfer line is inserted into the source volume. If the sample is to be ionized in the trap, the transfer line is inserted into a hole in the trap close to an electron generating filament. Two different tips must be used to extend the GC column into the point of ionization, depending on the mode used. A short Polyimide tip is used for internal ionization and a long metal tip is used for external ionization. The body of the transfer line consists of a stainless steel body fitted with a center tube, a heat exchanger, and a boot. The heat exchanger is an aluminum cylinder that contains a cartridge heater and a thermocouple as the temperature sensor. The temperature sensor measures the temperature of the tube. The cartridge heater heats the cylinder, which distributes heat evenly throughout the length of the transfer line tube. The boot of the transfer line, which mates to the GC, prevents hot air from leaking from the GC oven.



*Exploded View of Transfer Line*

A bayonet mount feature secures the transfer line. Before you remove the trap, push gently on the bayonet mount as you twist it counterclockwise and pull the mount out. Make sure the transfer line extends out from the trap.



## CAUTION

**Failing to remove the transfer line before removing the trap may result in damage to the transfer line tip.**

The power board supplies power to the cartridge heater via a transfer line heater cable. The heater cable projects out from one end of the transfer line. It then plugs into a soft-shell connector on the top of the power board panel.

The transfer line temperature is set in the Temperature Dialog in System Control. The maximum temperature that the transfer line can sustain is 350 °C; the minimum temperature depends on the GC oven and trap temperatures. In general, the transfer line temperature can be set as much as 30 °C below the maximum column operating temperature and not cause adverse chromatographic effects (e.g., retention time shifts or peak broadening).

After the sample stream passes through the transfer line it is ionized either in the ion trap or in the external source.

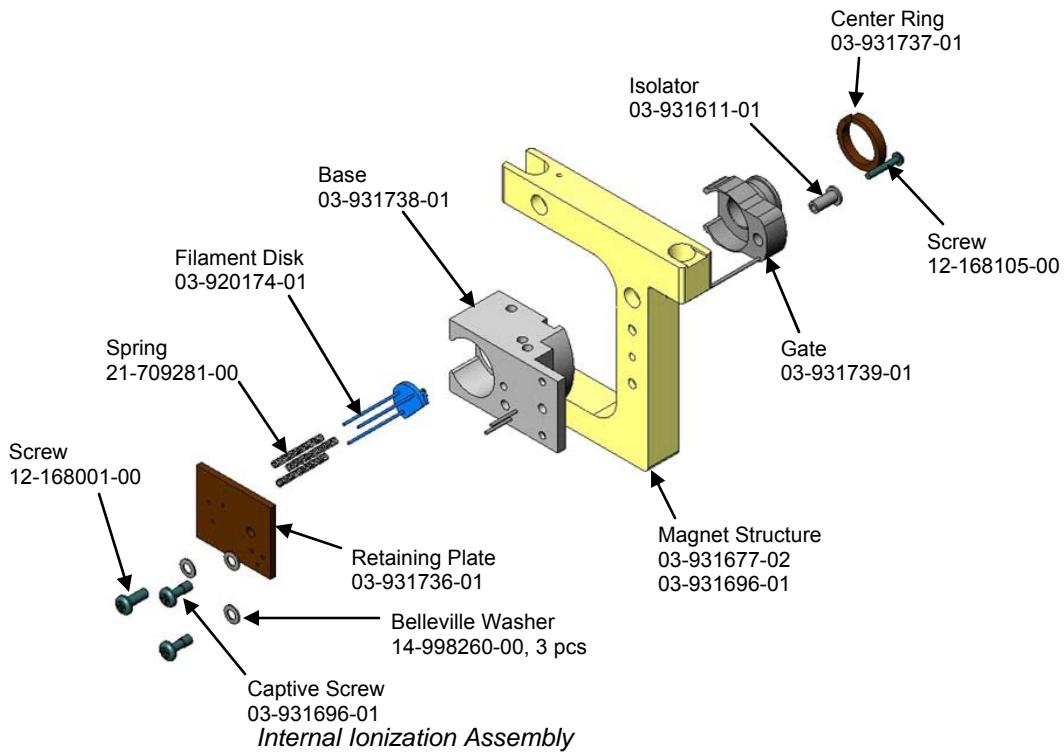
---

## Analyzer

The Analyzer consists of an Internal Ionization Assembly or External Source, the Ion Trap and a Conversion Dynode/Electron Multiplier Detector. All components except the detector are mounted on a flange, which also contains an enclosure holding electronics related to the analyzer. For the purposes of this manual, this assembly is referred to as the “**Analyzer Assembly**”. The Internal Ionization Assembly or External Source is attached to the Ion Trap Assembly. This combination is referred to as the “**Source/Ion Trap Assembly**” in this manual.

### Internal Ionization Configuration

When the system is in internal ionization configuration, ions are generated directly in the ion trap. Electrons for ionization are produced and gated by an internal ionization source that resides just outside the ion trap's entrance electrode. The source consists of a filament assembly and electron gate electrode with associated mounting hardware. It is held on a U shaped structure, which is also used to hold collimating magnets for external ionization.



The filament assembly consists of two filaments and a repeller plate. The two filaments are mounted side-by-side, with each filament approximately equidistant from the entrance hole of the oven's electron focusing lens. Note that the 4000 MS only uses one filament at any given time; the extra filament is provided as a backup in case the first one burns out. The repeller plate is a stainless steel plate that is held at a lower potential than the filament to repel the electrons into the trap.

Each filament is a rhenium ribbon. When sufficiently heated by electric current, the filament produces electrons by thermionic emission. The filament emission current refers to the flow of emitted electrons from the filament. The filament emission current is set in the Internal EI or (CI) Properties tab dialog in the 4000 MS Method. Emission current settings range from 5 to 100  $\mu$ A.

---

**NOTE:** It is unlikely that two filaments will have the same net flow of electrons into the ion trap. Thus, the signal amplitudes from two different filaments will probably not be the same. A typical difference is 2:1, but it may be as high as 5:1.

---

The electron gate is a cylindrical electrode that controls the entry of electrons into the ion trap cavity. When electrons emitted from the heated filament are not needed for ionization, the electron gate is held at a -150 Vdc potential. An anodization layer insulates the electron gate from the filament end cap.

When the ion trap requires electrons, the electron gate potential changes from -120 to +120 Vdc. The gate potential remains positive for a variable length of time, e.g., from 10  $\mu$ sec to 65 ms. During this interval, the electrons are focused into the ion trap cavity with sufficient energy to achieve electron ionization of the

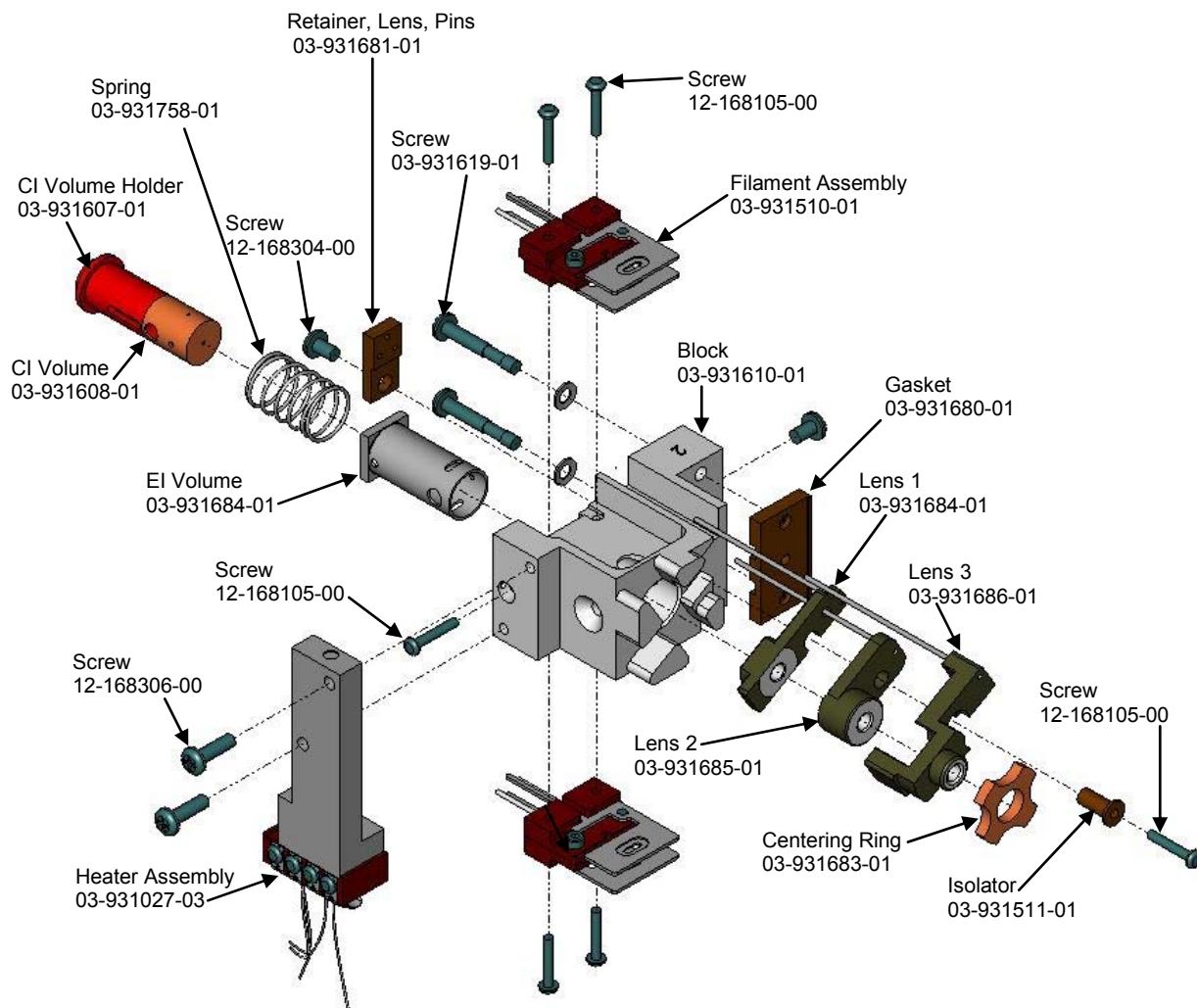
sample molecules (or of the reagent gas molecules in the case of chemical ionization).

## External Ionization Configuration

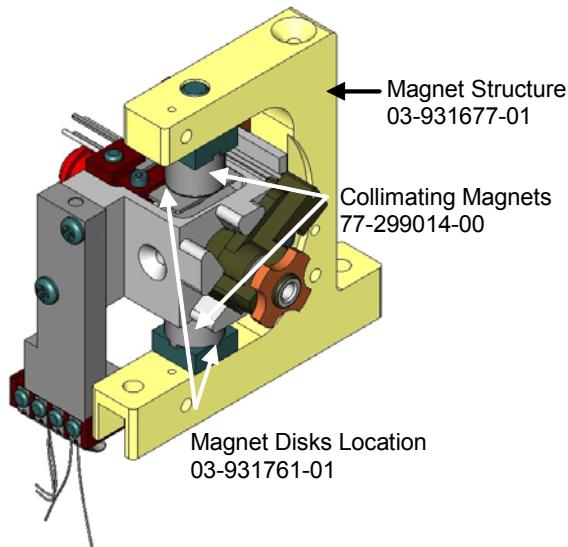
In external ionization configuration, either positive or negative ions are generated outside the trap in an external source and then injected into the trap. The external source is also used to produce reagent ions for hybrid ionization, which then ionize the sample inside the trap. The external source is an assembly that consists of an ion volume surrounded by two filament assemblies, a set of lenses, a collimating magnet, and a heater, all supported by a source block.

The sample enters the source volume where it is ionized. Both low and high pressure ion volumes are used by the system. Electron ionization (EI) uses the low pressure volume. Chemical ionization (CI) uses the high pressure volume. Both volumes are thin stainless steel cylinders that are chrome plated to minimize any reactions with the sample. The low pressure volume is open at the end facing the trap. The high pressure CI volume is sealed at that end with a small hole to allow ions to be entrained in the gas stream that flows from the volume. Both volumes have additional holes to allow ionizing electrons and the sample to enter. The CI volume also has an opening for the CI reagent. When in use, the CI volume is inserted into the EI volume by a pneumatically-activated plunger which is controlled by the software.

The sample in the case of EI or reagent in the case of CI is ionized by electrons generated by one of two filament assemblies. Each assembly has a rhenium filament sandwiched between a repeller plate and electron lens, all supported by a ceramic base. The filament generates electrons through thermionic emission resulting from the heat generated by current flowing through the filament. During ionization, the repeller is set to a negative voltage and the electron lens to a positive voltage to gate electrons into the source. When ionization is not taking place, these voltages are reversed to prevent electrons from entering the source. This patented pulsed ionization technique reduces ion noise during mass scanning and also reduces contamination of the ion volume. The repeller and electron lens voltages need to be properly balanced, using an Auto Tune routine, to keep the electron current stable during switching. In addition to gating electrons into the ion volume, the electron lens also focuses the electron beam. To collimate the electron beam further, two magnets adjacent to each filament assembly collimate electrons into the source.



*External Source Components*



*External Source Assembled*

After the sample is ionized, three lenses are used to direct the resulting ions towards the ion trap using electrostatic focusing. In the case of EI, the first lens also extracts ions from the source.

The center (L2) lens also acts to gate the ions into the trap by changing its polarity. The lenses are nickel-plated stainless steel cylinders with an anodized insulating layer to prevent the lens from shorting together. Each lens has a voltage connecting post.

A heater maintains the source at an elevated temperature. Electrical connections are made to the source through a flexible printed circuit cable that connects to the electronics through a printed circuit board mounted to the top flange. A heat shield between the source and the flexible cable protects the solder joints.

## Hybrid Configuration

The 4000 MS supports a unique mode of operation called hybrid chemical ionization. In the hybrid configuration, reagent ions are generated in the external source then drawn into the ion trap to react with analytes from the GC column. This approach has a number of potential advantages including avoiding ion molecule reactions with the neutral reagent and avoiding losses of negative ions that occur when they move from the external source to the trap. The hybrid mode requires the external ionization option and a security chip to be present but does not involve any unique hardware. In the hybrid mode, the external source must be in place and the transfer line must be positioned with the sample directly entering the ion trap.

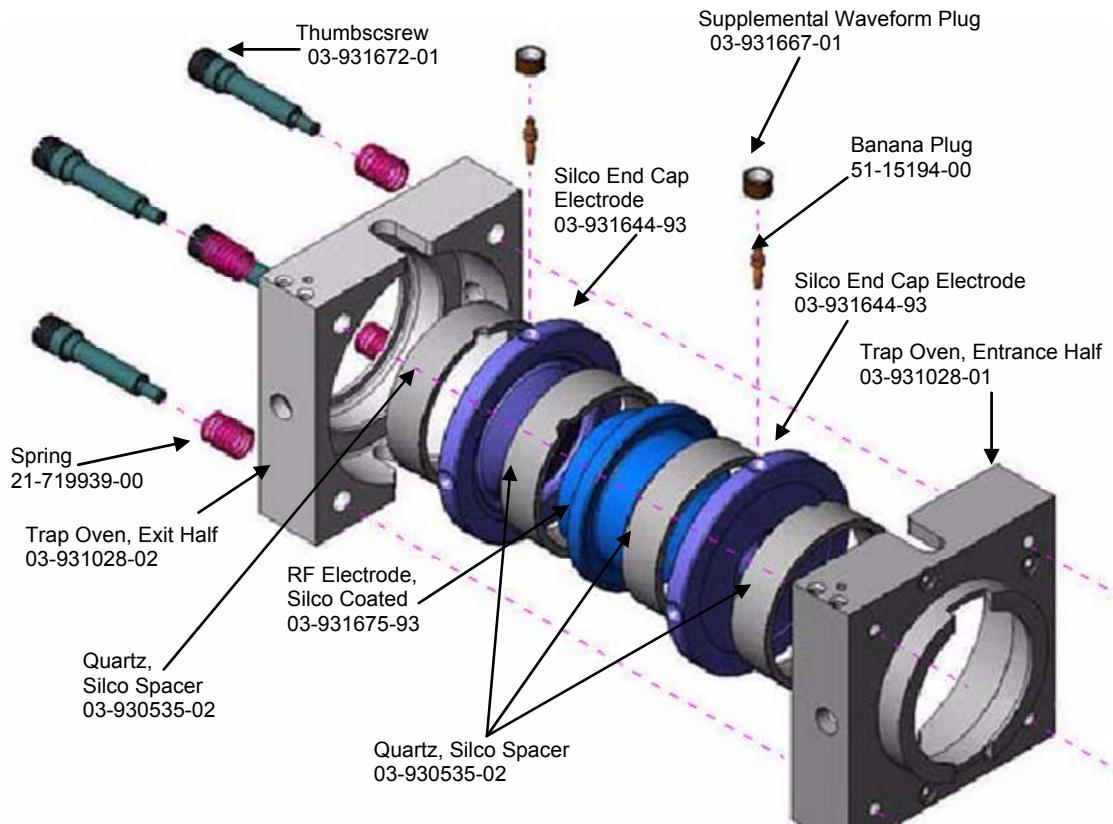
## Ion Trap

The ion trap assembly consists of three electrodes separated by quartz spacers, and contained in a heated oven. The three electrodes are the entrance, ring and exit electrodes. These electrodes have hyperbolic inner surfaces that together form a cavity in which ionization, fragmentation, storage, and mass analysis take place.

There is a single hole in the center of both the entrance and exit end cap electrodes. The hole in the entrance electrode allows the entry of ionizing electrons when the system is configured for internal ionization. The hole in the exit end cap allows the exit of ions to the detector. There are also holes in the edge of the end caps in which banana plugs are placed that make contact with springs that carry supplemental waveform signals. One of these holes in the entrance end cap also acts as the sample inlet to the ion trap in internal and hybrid ionization modes.

Four identical quartz or silica-coated spacers separate the central ring electrode from the entrance and exit end cap and from the trap oven plates. The trap oven and its clamping plate hold the electrodes and spacers in place.

The RF generator assembly provides high voltage 1 MHz RF voltage that is applied to the RF ring electrode through a feedthrough on the underside of the vacuum manifold. Under the proper RF voltage, the ion trap electrodes create a three-dimensional, hyperbolic electric field. This field is capable of trapping the ions in stable, aperiodic orbits. In the presence of helium damping gas, the ions are cooled towards the center of the trap. As the RF voltage increases, the ion trajectories become unstable in increasing order of mass to charge ratio. The ion trap ejects the ions and sends them to the conversion dynode and then to the electron multiplier for detection.



*Ion Trap*

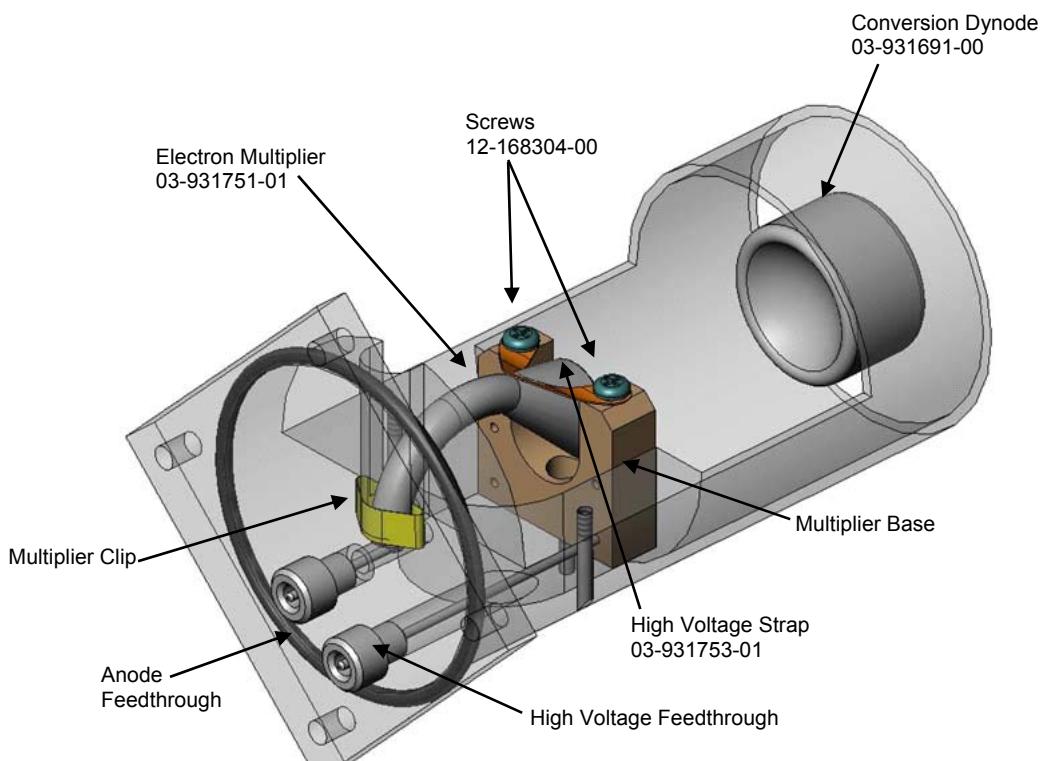
During mass analysis, a dipole voltage at the trapping RF frequency is applied across the end caps to offset the ions from the center of the trap. Two additional supplemental waveforms are applied to the end caps. The dipole signal is applied out of phase across the end caps while the quadrupole signal is applied

in phase. These supplemental waveforms interact with the ions and cause ejection when they correspond to one of the secondary secular frequencies of ion motion. The end caps receive these signals by way of small banana plugs that are inserted into the electrodes. The plugs receive the signal in turn from springs attached to feedthroughs in the upper flange.

A DC offset voltage can also be applied to all three electrodes in the trap. The DC offset is used in external ion mode to assist in the introduction of ions into the trap.

## Detector

After ions are ejected from the trap, they are detected by a combination conversion dynode/electron multiplier detector. The detector is enclosed in a cylindrical stainless steel shield that prevents metastable ions from entering the source.



4000 MS Detector

After exiting the trap, ions are first accelerated onto an off axis conversion dynode that generates a combination of positive ions and electrons through secondary electron emission. The conversion dynode is made up of a rounded stainless steel cup suspended on a post. The cup is manufactured with a smooth surface finish to prevent spurious field emissions. If positive ions are to be detected, the conversion dynode is set to a large negative voltage (typically -10 kV). In this case, the secondary electrons will be attracted to the relatively positive multiplier. For negative ions, the conversion dynode is set to a large positive voltage, in which case positive ions from the dynode are attracted to the

relatively negative multiplier. In addition to allowing the detection of both positive and negative ions, the off axis conversion dynode eliminates detection of photons that would be seen by an on axis detector.

The continuous-dynode electron multiplier consists of a lead-oxide/glass, funnel-like resistor. A negative voltage of between -800 and -3000V is applied to the front end of the electron multiplier, referred to as the cathode. The back end of the cathode is held near ground potential, and is referred to as the anode.

Electrons or ions emitted from the conversion dynode strike the cathode with sufficient velocity to dislodge additional electrons from the inner curving surface of the cathode. The increasingly positive potential gradient draws the ejected electrons into the electron multiplier, further accelerating them in the process. Because the electron multiplier is curved, the ejected electrons do not travel far before they again strike the inner surface of the multiplier, resulting in the emission of more electrons. This configuration produces a cascade of electrons that is accelerated toward ground potential at the exit end of the cathode.

The anode collects the electrons and passes the resulting ion signal to the ion amplifier that is mounted on the side of the vacuum manifold directly next to the multiplier. The ion current is proportional to the total number of ions that the ion trap ejects. Typically, the voltage applied to the electron multiplier should be adjusted until the gain is about  $10^5$ , i.e., until each electron or positive ion that enters the electron multiplier generates approximately  $10^5$  electrons.

---

## Vacuum System

The analyzer is contained in a vacuum manifold maintained at a pressure of 10  $\mu$ Torr. A turbomolecular pump provides the vacuum required. The turbo pump is backed by a mechanical rotary foreline pump, which also performs the initial evacuation of the vacuum manifold during pump down. A thermocouple gauge is used to measure the foreline pressure and an ion gauge to measure the vacuum manifold pressure.

### Vacuum Manifold

The analyzer is contained in a nickel-plated aluminum vacuum manifold that provides feedthroughs for the various electrical and pneumatic lines that are required. A top flange feeds the end cap voltages and supplies all the source electrical connections by way of a printed circuit board feedthrough. A front flange feeds the CI and Calibration gases and supports the CI ion source switching mechanism. A side flange provides multiplier connections. All three flanges are sealed by Viton® O-rings. The manifold has line voltage heaters in its base to provide heat for bake-out. Insulating material surrounds the manifold to retain the heat. The turbomolecular pump is mounted horizontally to the rear of the manifold.

### Foreline Pump

The foreline pump has two purposes. The first is reducing the vacuum system pressure to a level that will allow the operation of the high vacuum turbomolecular pump. The second is maintaining the vacuum system pressure by removing the turbomolecular pump's exhaust gases.

The foreline pump is connected to the turbomolecular pump by a 2.1m (84 in.) length of 1.9 cm (0.75 in.) ID vacuum tubing. The pump plugs into the rear panel

outlet labeled "LINE VOLTAGE - PUMP ONLY" on the rear of the MS. Power is supplied through this outlet and is controlled by the power switch on the rear panel.

The foreline pump used on the 4000 MS is a Varian DS-102 two-stage rotary vane pump with a pumping speed of 90 L/min and a vacuum potential of  $1.5 \times 10^{-3}$  Torr ( $2 \times 10^{-1}$  Pa).



#### **WARNING: CHEMICAL HAZARD**

If you use the 4000 MS to analyze hazardous materials, be sure to direct the foreline pump exhaust to an exhaust system that complies with applicable safety regulations.

### **Turbomolecular Vacuum Pump**

The Varian TV-301T Turbomolecular Vacuum Pump provides the high vacuum for the 4000 MS. Under normal operating conditions, this pump supplies a vacuum of approximately  $10^{-5}$  Torr ( $1.33 \times 10^{-3}$  Pa) in the manifold region outside the ion trap assembly. The pump is rated at 230 liters/second; it is air cooled and thermostatically protected. If the temperature of the pump housing near the bearing exceeds 60 °C, the pump will automatically shut down.

A turbomolecular-pump controller regulates and supplies power to the pump. The controller sits below the pump in the analyzer compartment of the spectrometer. Turning off the main power switch on the rear panel of the mass spectrometer shuts off power to the turbomolecular-pump controller and thus to the pump.



#### **CAUTION**

The electronics service switch does not turn off the vacuum pumps.

The turbomolecular-pump controller monitors the pump's rotational speed. The controller sends a signal proportional to the pump speed to the controller board via the power board. You can monitor the turbomolecular pump speed from the Diagnostics or the Startup/Shutdown tab dialogs in System Control.

If the pump speed falls below 94% of its maximum operating speed, the VACUUM OK signal read by the Controller board turns off. The filament, electron multiplier, RF generator, CI reagent gas valve, and calibration gas valve turn off automatically. This condition probably indicates a major air leak in the system or that the pump is too warm.

### **Ion Gauge**

An ion gauge is present on the bottom of the vacuum manifold. Its design is based on the Bayard-Alpert gauge tube. The specifications for the gauge are similar to those of commercially available gauges. Fixed pressure readings with nominally identical gauges may exhibit variations of  $\pm 15\%$ . An accuracy of  $\pm 25\%$  in mid-range for any one gauge is considered typical.

The ion gauge generally exhibits good repeatability. However, the ion gauge response depends on gas composition. A given pressure of air and water will give a different reading than that of helium. The ion gauge is meant to be a rough indicator of vacuum conditions. It is not a precise quantitative tool.

The gauge uses thoria-coated iridium (ThO-Ir) filaments. These filaments are burnout resistant, and therefore exhibit high tolerance to air and water in the vacuum manifold. There is a time delay associated with heating the filament to allow it to stabilize. Stable readings will be obtained in 15 – 20 seconds.

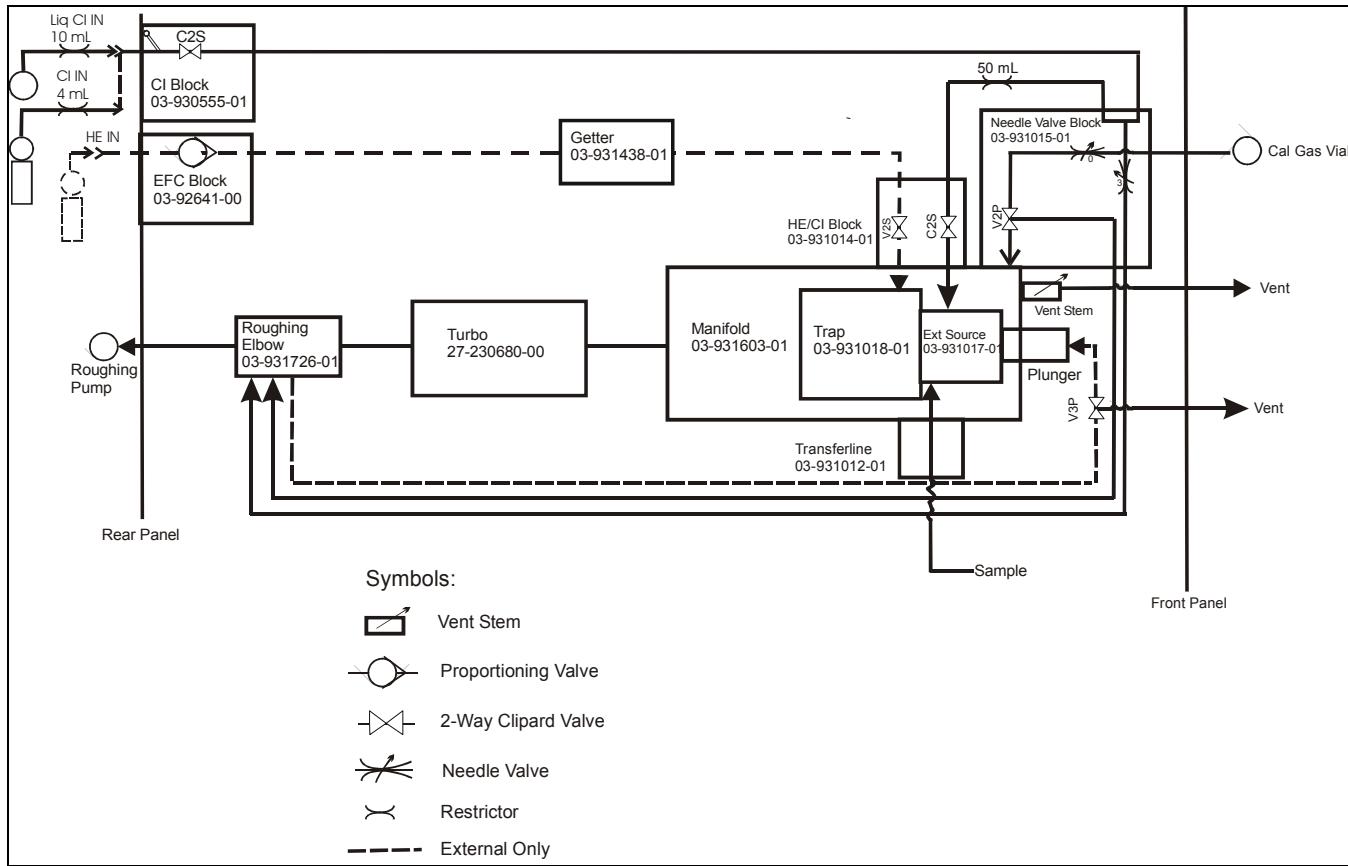
The ion gauge measures pressures between 0.1 and 10,000 Torr. A logarithmic amplifier on the ion detection board amplifies the collector current, and the data system interprets this current as measured pressure. Ion gauge pressures can be monitored from the Manual Control, Diagnostics, and Startup/Shutdown tab dialogs in System Control.

## Thermocouple Gauge

A thermocouple gauge is attached to the foreline pump hose to measure pressure to check for gross leaks and foreline pump failure. The thermocouple gauge is a simple, rugged, vacuum gauge that is used to measure vacuum pressures in the 2 Torr (267 Pa) to  $1 \times 10^{-3}$  Torr ( $1.3 \times 10^{-1}$  Pa) range. The gauge's main purpose is to enable the detection of gross leaks and foreline pump failure.

# Pneumatics

Pneumatics components deliver the required gases to the analyzer including helium damping gas, calibration gas (FC-43), and various Cl reagents.



Pneumatics Interconnections

## Helium Flow

In internal ionization and hybrid mode, helium damping gas is provided to the trap through the GC column flow. In external ionization mode, the helium damping gas must be provided separately. Helium enters through a Swagelok® fitting in the back of the instrument. It is then immediately routed through an electronic flow controller (EFC) that maintains a constant flow, set through the workstation in the Module Attributes tab dialog in Manual Control. The EFC measures the pressure drop across the flow path and then adjusts the position of an electronically controlled valve to keep the proper flow (see “Electronic Flow Control” on page 25). After passing through the EFC, the helium flows through a heated getter to remove water and other contaminants from the system. The getter normally operates at about 400 °C.



### WARNING: FIRE HAZARD

**It is critical to run only helium through the getter. Running air or any oxidizing gases may destroy the getter and result in hazardously high temperatures and fire.**

Helium enters the vacuum manifold through a solenoid valve on the side of the vacuum manifold. The controller board monitors the temperature of the getter on a continuous basis. If high temperature, loss of inlet pressure or vacuum failure is observed, the controller shuts off flow of helium on both sides of the getter.

## Calibration Gas Flow

The calibration compound is perfluorotributylamine (PFTBA) or  $C_{12}F_{27}N$ , also known as fluorocarbon-43 (FC-43). A small glass vial inside the front door of the 4000 MS holds the compound. The flow of calibration gas into the manifold is set manually via a needle valve. The needle valve is in a block below the CI reagent needle valve inside the front door of the 4000 MS. The MS Workstation controls the opening and closing of a three-way solenoid-operated valve downstream of the needle valve. When the Cal Gas flow is off, a vacuum is placed on the vial, by way of a line connected to the foreline elbow, to prevent a pressure build up that would result in a pulse of calibrant when the gas is turned on.

## The CI Reagent Gas Flow

The CI reagent enters the system through a solenoid valve on the back of the instrument. It then passes through a restrictor and second solenoid valve that is in the same block, on the side of the manifold, as the helium solenoid valve. A line to the roughing elbow is attached to the CI line to pump away some of the reagent to prevent pressure pulses when the CI is turned on. The CI control needle valve controls the flow in this vacuum line that in turn controls the flow of reagent into the source by changing the split ratio. After passing through the solenoid valve, the flow passes through the magnet structure inside the vacuum manifold. In the case of Internal Chemical Ionization, the reagent then flows into the entire manifold. In the case of External Chemical ionization, the flow is routed directly into the CI source volume.

# Electronics



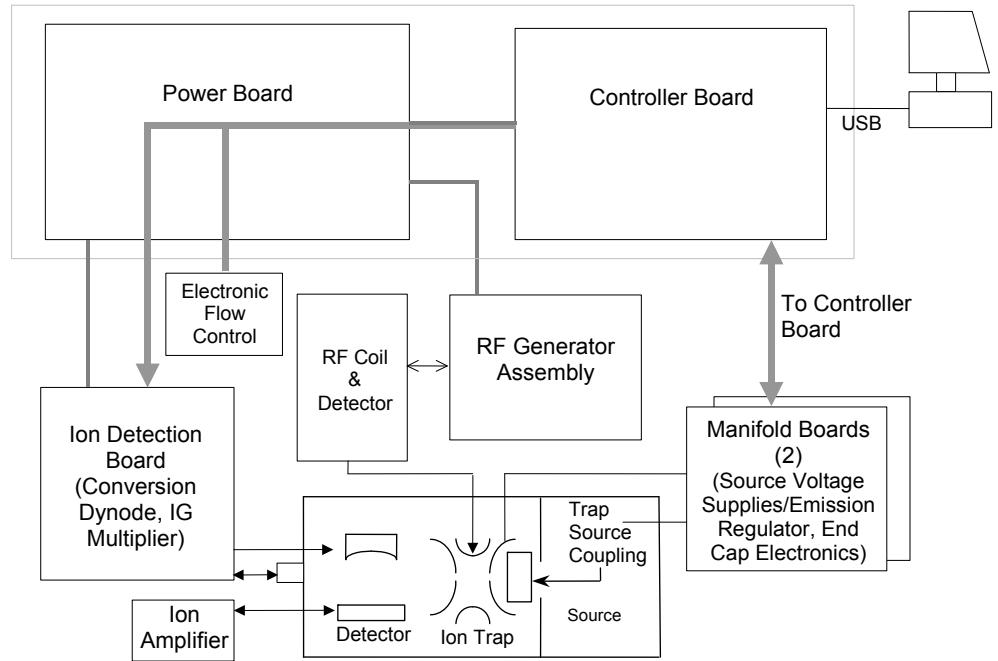
## WARNING: SHOCK HAZARD

**HIGH VOLTAGES INSIDE.** No user serviceable parts under screw-attached covers. Contact your local Varian, Inc., service representative for instrument repair and service.

The electrical functions of the 4000 MS are distributed among eight boards (see block diagram) each carrying out some specific functions. In some cases, the boards are located as close as possible to the associated part of the spectrometer. The RF coil plays a part in generating the trapping field RF and is a power entry subsystem in the back of the instrument. The functions of the boards are as follows:

- Controller Board – controls spectrometer operations and acquires data generated by the system.
- Power Board – generates all power sources for the instrument, controls the temperature of heated zones, routes signals between other boards and drives all solenoid valves.
- Lower Manifold Board – drives various source voltages and controls filament operation.
- Upper Manifold Board – contains circuitry that handles supplemental excitation waveforms and trapping field dipole switching.
- RF Generator Assembly – generates and controls the RF trapping field.
- Ion Detector Board – has the circuitry for the Multiplier and Ion Gauge, both of which detect ions in the system.
- Ion Amplifier – an electrometer that amplifies the ion signal.
- Electronic Flow Controller – the module that controls the flow of the helium damping gas.

A power input sub-system distributes line voltage to various components as needed.



*4000 MS Electronics Block Diagram*

## Controller

The controller (see block diagram) is the “brains” of the spectrometer, controlling all operations as well as acquiring all data. The controller executes scan functions, sets various static voltages and switches components such as valves. Commands and data are communicated between the controller and the MS Workstation computer through a universal serial bus (USB) interface.

The processing subsystem of the controller utilizes two TI DSP (Digital Signal Processing) microchips. The use of two processors allows time critical operations, handled by the scan processor, to be separated from non-time critical operations, handled by the communication processor. The processors each have their own local memory where programs reside and a shared dual processor memory that is used to hold data and exchange command or status information. The scan processor handles instrument control, including scan function execution and data acquisition, in a synchronous manner. Receiving of commands from the workstation and transmission of accumulated data is performed asynchronously by the communications processor.

Acquisition method segments are pre-downloaded in their entirety to the communications processor prior to their execution and stored in shared memory. The segments are then activated at the appropriate time by the controller. Multiple method segments can be preloaded. 32 megabytes of dynamic random access memory (DRAM) is used to store a library of waveforms used for scan function supplemental waveforms. The combination of preloaded waveform libraries and preloaded segments eliminates any delays between segments.

Various switched components in the system (such as solenoid valves) are controlled through latches. Analog control voltages are set by the scan processor through a set of digital to analog converters ranging in resolution from 10 bits for lens voltages to 16 bits for the trapping field RF level.

A number of specialized functions are implemented on the controller using field programmable gate arrays. (FPGAs). These functions include an acquisition controller, waveform/memory controller, and RF scanning module.

## Power Board

The power board supplies power to all electronic components except the turbomolecular pump controller. It also controls a number of heaters and solenoid valves as well as providing signal routing between the controller board and other boards in the system.

---

**NOTE:** The switching power supply is protected by a 5A, Non-Time-Delay, fuse.

---

Switching power supplies are utilized for all voltages. The following switching power supplies reside on the board:

- A +5 Vdc power supply, which supplies +5 Vdc voltage to all digital circuits.
- -15V and +15 Vdc power supplies, which supply the voltages to the analog circuits on the power board and the manifold electronics assembly.
- +20V and -20 Vdc power supplies, which supply the voltages to the Controller and RF generator board's analog circuitry.
- A +24 Vdc power supply which provides power for the solenoid valves, heaters, the EFC, electronics compartment fan, and the electron multiplier power supply.
- A +60 Vdc power supply, which supplies unregulated +55 Vdc voltage to the RF generator board.
- The 200-volt power supply that supplies voltages to various lens circuits and gate circuits as well as the ion gauge.

The following circuits also reside on the board:

- Four heater control circuits that provide feedback control for the manifold, trap, external source and transfer line heaters. The trap heaters use proportional integral (PI) control circuits. Because there is an integrator component in this controller, removing power from the circuit may produce a lengthy stabilization time, e.g., up to two hours (dependent on the temperature set point).
- Four solenoid control circuits, which turn the calibration gas, Cl reagent gas, Cl shutoff valve and EI/Cl volume solenoids on and off.
- The diagnostic multiplexer circuit, which routes the voltage output of various components, and circuits on the power control board to the Controller board. You can access these voltage outputs through the diagnostic pages.

Mounted on the top edge of the power board are 15 monitor LEDs. When illuminated, these lights indicate that the voltages of the various circuits on the

power board are at their proper levels, and that there are no faults. During normal operation, all green LEDs should be on.

The power board supplies most of the regulated voltages for other electronic subsystems in the spectrometer. The voltages include +5 volts for digital components,  $\pm 15$  volts for analog components (such as amplifiers), +24 volts for all the heaters except the manifold, 60 volts for the trapping field RF generator.

## Manifold Electronics

The manifold electronics consists of two boards stacked in an enclosure directly on top of the vacuum manifold. The boards perform a variety of functions related to the ionization and mass scanning processes. Functions related to the external source include providing lens voltages and heater control. These boards provide filament control for both external and internal ionization.

The function of the upper manifold board is to handle the signals that are applied to the ion trap end cap electrodes. As explained in the user guides, dipole waveforms are applied to the end caps during the ionization, isolation and mass scanning processes. Quadrupole waveforms are applied during the mass scanning process. The dipole signal is applied, out of phase, to the two end caps to provide a signal across the end caps. The quadrupole is applied in phase to provide a voltage between the end caps and the ring electrode. Waveform signals are received from the controller board through the power board. They are then buffered by high-power operational amplifiers and applied to the end caps through transformers that step up the waveform voltage. Two transformers apply the dipole waveforms, one for high frequency dipole waveforms and the other for low frequency square waves applied during non resonant CID. A trapping field dipole (TFD) voltage is applied during the mass scanning process to offset the trapped ions from the center of the trap. The TFD signal is derived from trapping field RF currents flowing in the end caps coupled from the 1 MHz signal applied to the ring electrode by the RF generator and coil. The TFD is switched on and off by changing the impedance between end caps and ground; when the TFD is off, a low impedance is switched in. When the TFD is turned on, a high capacitive impedance on one end cap and inductance impedance on the other end cap are switched on, resulting in the out of phase dipole signal.

The lower manifold board handles a number of source related electronics functions. It has amplifiers that apply the appropriate lens voltages to the source, based on set points received from the controller board. The source filament emission regulator circuit is also present on the board. In addition, there is also conditioning electronics that produce high-level temperature measurement signals from resistive temperature devices (RTDs) on the source and traps that are used for temperature control and diagnostic purposes.

## RF Generator Assembly

The RF generator assembly consists of an RF generator circuit board, an RF detector circuit board, and the RF coil. A shielded housing beneath the vacuum manifold encloses the coil and RF detector circuit board. The RF generator circuit board is attached to the back of the shielded housing.

The RF generator circuit board receives an analog signal from the controller board that is proportional to the current mass position in the scan, which is in turn proportional to the desired RF voltage applied to the ion trap. The RF detector circuit board sends a signal proportional to the actual amount of RF voltage applied to the ion trap to the RF generator board. The RF generator board

compares the desired and actual amount of the RF voltage and adjusts the gain of an RF amplifier to cause the actual RF voltage to equal the desired RF voltage. Since the high voltage required at the ion trap exceeds the capabilities of conventional electronic amplifiers, a resonant LC circuit consisting of the RF coil and the ion trap capacitance is used. At resonance, the RF voltage at the ion trap end of the coil is about 150 times that at the RF generator circuit end of the coil.

## **Ion Detection Board**

The ion detection board contains key elements of the electronics associated with detecting ions by either the electron multiplier or ion gauge. The board contains a power supply that applies voltage to the cathode of the electron multiplier. That supply consists of a chain of voltage multiplier circuits that are switched between –800 and –3000 volts by a multiplier on signal from the controller. The ion detection board also has the emission current regulation circuitry for the ion gauge, as well as the electronics to obtain and condition its vacuum signal.

## **Ion Amplifier**

The ion amplifier converts the current received from the electron multiplier to voltage that can then be read by the controller board analog to digital converter. This amplifier boosts the signal by a factor of  $10^7$ . To maximize the bandwidth, the amplifier is mounted on the side of the vacuum manifold as close to the multiple output feed-through as possible.

## **Electronic Flow Control**

An electronic flow controller (EFC) controls the flow rate of helium damping gas in external ionization mode. The EFC maintains the proper flow using a closed loop feedback control system. The flow set point is set through a digital to analog converter (DAC) that receives its setting from the controller board. The control electronics then reads the flow by measuring the pressure across a known orifice using two pressure transducers. It sets the required flow using a proportional solenoid valve. The relationship between flow and differential pressure is factory calibrated. Ambient temperature is measured to compensate for flow differences with temperature. The EFC also is used to control the state of the helium cutoff valve at the manifold. This valve is closed if excess getter temperature is detected or if the helium inlet pressure drops below 20 psi.

## **Power Input Subsystem and Turbomolecular Pump Controller**

The power input subsystem contains the following circuits and switches:

- Main power switch
- SERVICE switch
- Line voltage switches

## Main Power Circuit

Line power of 90 - 130 Vac, 60 Hz  $\pm 3$  Hz (or 180 - 230 Vac, 50 Hz  $\pm 3$  Hz) first enters the rear panel of the mass spectrometer through J1, and then passes through a line filter and the circuit breaker. After the circuit breaker, power is split in two directions. One path supplies the turbomolecular pump controller and foreline pump via J2. The second path goes to the electronics service switch, which controls power going to the power board and the rest of the electronics. The electronics service switch allows the vacuum to be maintained in the event that the electronics need to be serviced.

The turbo pump controller provides startup power to the power board, in addition to regulating the speed of the turbo pump.



### **WARNING: SHOCK HAZARD**

**In the event of an emergency, shut off all power.**



### **WARNING**

**If the equipment is used in a manner not specified in this manual, the protection provided by the equipment may be impaired.**

# Periodic Maintenance

---

## Procedure Interval

To ensure peak GC/MS performance, you need to perform periodic maintenance on the vacuum and cooling systems. The following list identifies relevant maintenance intervals.

- Check the foreline pump oil level and oil condition weekly
- Purge foreline pump oil weekly
- Check cooling fans weekly
- Change foreline pump oil and filter at least every nine months

---

## Checking Foreline Pump Oil Level and Oil Condition

Ideally, the level and condition of the pump oil should be checked with the pump switched off and warm, though a reasonable assessment can be made with the pump running. The oil level should be between the maximum and minimum levels on the sight glass. If the oil level falls below the minimum level, use a funnel to gradually add more oil (88-299517-00) through the filler port until the oil level is centered between the maximum and minimum levels.

---

NOTE: Pump models are subject to change. If not using a model DS-102 pump, refer to the pump manual for details.

---

The pump oil should be clear and light amber in color. If the oil becomes thick, dark in color, becomes opaque, or has a burnt smell, change it and the Oil Mist Filter Cartridge as described in Changing Foreline Pump Oil.

---

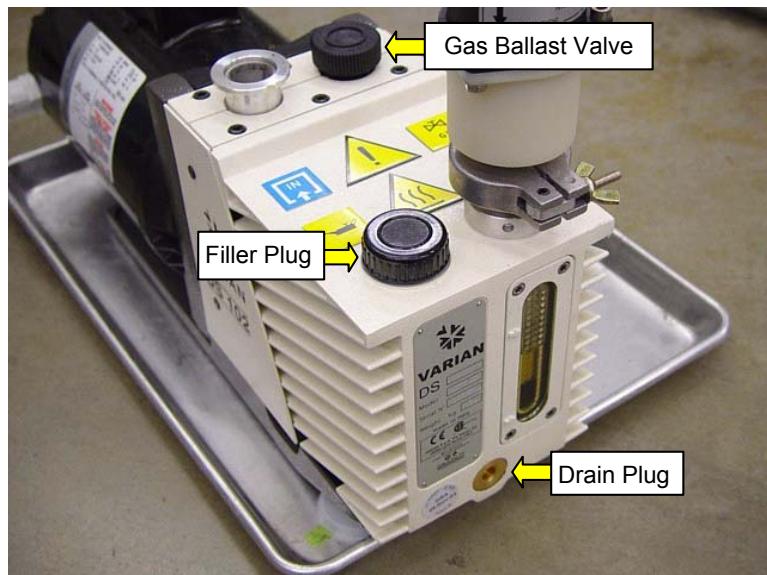
## Changing Foreline Pump Oil

To ensure peak performance and maximum pump lifetime, change the pump oil whenever the oil becomes thick, dark in color, and has a burnt smell, or at least every six months. The oil change should be performed while the oil is warm but not immediately after stopping the pump.

Materials Needed

- 5/16" Allen Wrench
- Varian GP Oil (88-299517-00)

- 1.0-liter (1 US qt) or larger container



To change the pump oil:

1. To turn off and vent the MS, go to "Turning Off the Mass Spectrometer" (page 34).

**Disconnect the pump's power cord from the rear of the MS.**



**WARNING:  
SHOCK HAZARD**

Dangerous high voltages are present. Unplug power cord.



**WARNING:  
BURN HAZARD**

**Hot Surface. Take appropriate precautions. Wait for the pump to be cool enough to touch before continuing the oil changing operation.**

2. Disconnect the vacuum hose from the foreline pump by removing the clamping ring.
3. Pull the hose free, and place the seal on a clean lint-free surface for later use.
4. Carefully place the foreline pump on a raised surface. The surface should be high enough to allow a 1.0-liter (1 US qt) or larger container to be placed under the drain port when the pump is tilted forward. A container with an opening diameter of at least six inches will make this task easier.
5. Place an oil pan beneath the drain port to catch any spillage.



## CAUTION

The pump weighs at least 22 kg (48 lb.). Use proper lifting techniques to avoid physical injuries.



## WARNING: CHEMICAL HAZARD

Hazardous chemicals may be present in the used pump oil. Avoid contact with skin.



## WARNING: EYE HAZARD

Use proper eye and skin protection.

6. Remove the filler plug on top of the pump.
7. With the container in place to catch the oil, slowly remove the drain plug in the front of the pump using a 5/16 Allen wrench.



## WARNING: CHEMICAL HAZARD

Toxic residues from mass spectrometer samples will build up in used pump oil. Dispose of all used pump oil in accordance with applicable regulations. Place a hazardous chemical warning label on the container.

8. Tilt the pump forward and hold until oil flow ceases.
9. Return the pump to the horizontal and refit the plug.
10. Run the pump for approximately ten seconds with the intake port open. This will remove any residual oil from the pumping block.



## CAUTION

Avoid breathing oil mist coming from the exhaust port during this operation.

11. Remove the plug, tilt the pump, and drain any remaining oil.
12. Return the pump to the horizontal.
13. Wipe the oil residue from the drainage port, and refit the drain plug.
14. If the pump oil was particularly dirty, flush the pump.
15. Fill the pump with fresh oil (88-299517-00) through the filler port until the oil level reaches the maximum level in the sight glass. A funnel may be helpful.
16. Replace the filler plug.
17. Run the pump for at least one hour with the gas ballast valve open to achieve a good vacuum.

## **Flushing the Pump Oil**

The pump should be flushed if the pump oil is particularly dirty. After draining the pump, (as described previously in steps 1 – 14) do the following:



### **CAUTION**

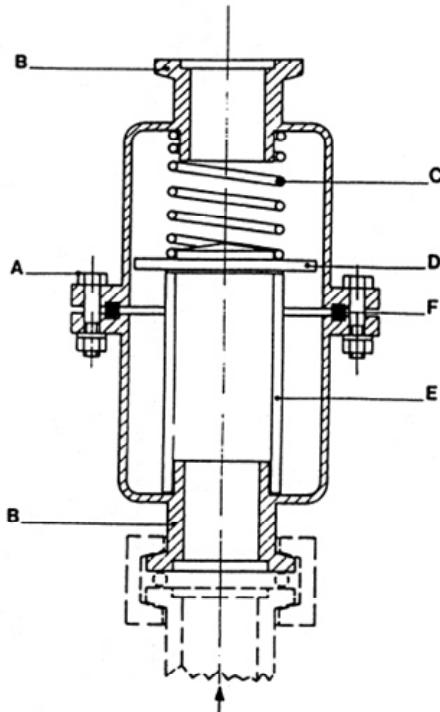
**Avoid breathing oil mist coming from the exhaust port during this operation.**

1. Pour 330 mL (0.35 US qt) of fresh pump oil in through the inlet port then run the pump.
2. Stop the pump, drain the flushing oil, and then continue filling with fresh oil.
3. Change the oil mist cartridge.

# Changing the Oil Mist Cartridge

Replace the cartridge of the oil mist eliminator on the exhaust port of the pump when you change the oil. The part number for a package of cartridges is 27-101002-00. There are two in a package.

Note: When the cartridge is saturated, excessive mist or oil sprays out, and the cartridge must be replaced.



## Disassembling the oil mist eliminator

1. Remove assembly screws **A**
2. Remove Upper housing **B**
3. Remove Spring **C**
4. Remove Valve **D**
5. Remove Cartridge **E**
6. Remove O-ring **F**
7. Clean the parts with a dry cloth.
8. Degrease with a water soap solution.
9. Rinse with clean water and dry.

## Reassembling the oil mist eliminator

1. Install a new cartridge in lower housing **B**
2. Press gently to check that it is firmly seated.
3. Install valve **D** with polished side toward cartridge.
4. Center the spring **C** over the valve, fit gasket, **F** in the groove.
5. Cover entire assembly with the second casing **B**
6. Tighten the two casings **B**, using screws **A**

---

## Checking Cooling Fans



To prevent overheating, do not block air intakes.

The cooling fans maintain an optimal temperature for the turbomolecular pump and the other electronics modules. Without the cooling fans, the lifetime of the turbomolecular pump and temperature-sensitive PC-board components can be shortened. To ensure proper operation of the cooling system, operate the MS with its covers in place. In addition, be sure to check the fans at least once each week. The MS is equipped with two fans on its rear panel. The function of these fans is to pull air into the instrument. To check fan operation proceed as follows:

1. Make sure that the main power switch and service switch are turned ON.
2. Place a large sheet of paper over one of the fan guards.
  - a. If the paper is sucked toward the fan guard, the fan is working.
  - b. If the paper is not sucked toward the fan guard, the fan is broken. Contact your Varian Customer Support Representative to arrange for a replacement.
3. Check the second fan in the same manner. If the fans are excessively noisy, e.g., if they whine or whir, one of the fans may be about to fail and it should be replaced.
- 4.

# MS Maintenance Procedures

---

## General Recommendations

There are a number of considerations to take into account when maintaining a high-vacuum trace analysis instrument such as the 4000 MS. In particular, considerable care must be taken not to introduce contaminants into the system.

Wash your hands before working on the system. Hand creams and highly perfumed soaps should be avoided.

Gloves should be used when handling any parts that are internal to the analyzer. Care must be taken when using gloves since many types of gloves can leave chemical residues. Powder free Nitrile gloves are best to be used followed by lint-free cotton or lint-free nylon gloves. Nylon and Nitrile gloves should not be used to handle parts at elevated temperatures.

Keep all tools clean and free of grease or other contaminants.

Store sources and transfer line tips in the containers provided. The containers are designed to be contaminant-free.

Take particular care to eliminate particles inside the vacuum manifold and on sealing surfaces. Clean and filtered compressed air and chemical wipes (such as Kimwipes®) can be used to remove such particles.

Cover open manifolds or exposed parts when they are not being worked on. Chemical wipes or aluminum foil work well.

---

## Recommended Tools and Materials

Use the following tools and materials for performing MS maintenance procedures.

- Tweezers or long nose pliers
- Longneck Phillips head screwdriver
- Longneck flat head screwdriver
- 3/16" wrench (or transfer line tool provided)
- 5/16" wrench
- 1.5 mm Allen wrench
- Toothbrush
- Beakers
- Ultrasonicator

- Thin blade knife (such as an X-acto® knife)
- Pasteur pipettes
- Gloves - powder-free Nitrile, or lint-free cotton or lint-free nylon
- Chemical wipes such as Kimwipes®
- De-ionized water
- Isopropyl alcohol, methanol or methylene chloride
- Acetone
- Mild detergent (ph 6 to 7.5)
- Aluminum oxide
- Cotton swabs
- Sandpaper

## Common Procedures

The procedures in this section are common to many of the maintenance procedures described later in this section. The manual contains links to these common procedures where appropriate. Click the link to jump to the common procedure indicated, then press the back arrow located on the Navigation Toolbar to return to the original procedure. The page numbers of these procedures are also indicated in the manual. It may be necessary to use the Acrobat View menu to display the Navigation toolbar, if it isn't already visible.

### Turning Off the Mass Spectrometer



#### **WARNING: BURN HAZARD**

**Allow heated zones to cool before disassembly.**

1. Shut down the mass spectrometer through the Startup/Shutdown tab in System Control. Click the Shut Down button in the upper left corner of the screen. The heaters will be turned off and the speed of the turbo pump will be gradually reduced to 35% of full speed. It may take several hours for full shutdown and cooling to take place.

Manual Control	Auto Tune	Temperatures	Diagnostics	Startup/Shutdown	Acquisition
Status and Control		Current Set Points		Operating Conditions	
Conditions: Analysis	Shut Down	Heated Zones	Trap Temperature: 220 C Manifold Temperature: 50 C Transferline Temperature: 280 C	Heated Zones	Trap Temperature: 220 C Manifold Temperature: 49 C Transferline Temperature: 280 C
State: Ready		Vacuum System		Vacuum System	
		Status: Ready	Pump Spin Speed: 100 %	Pump Spin Speed: 100 %	Current: 199 mAmps

To speed the shutdown process, the system can be powered down and purged with Nitrogen. See "Turning Off the Mass Spectrometer with Nitrogen Purge" (page 36).

Hide Keypad Event Messages ▾

May 07 09:04:37: Turning Getter OFF.  
May 07 09:04:37: Turning Damping Gas OFF.  
May 07 09:04:37: Shutdown: Pump/Heated Zones are shutting down.  
May 07 09:04:38: DO NOT PERFORM MAINTENANCE UNTIL SHUTDOWN IS COMPLETE.  
May 07 09:08:32: Shutdown: Pump is shut down.  
May 07 09:08:32: Shutdown: Still waiting for Heated Zones to cool to 50 degrees C.  
May 07 09:08:32: IF YOU DO NOT WANT TO WAIT FOR THE HEATED ZONES TO COOL,  
May 07 09:08:32: SIMPLY EXIT THE SOFTWARE NOW. HOWEVER, DO NOT PERFORM  
May 07 09:08:32: MAINTENANCE UNTIL HEATED ZONES ARE COOL AND POWER IS REMOVED.  
May 07 09:08:32: When you vent the system, DO NOT OPEN THE VENT VALVE MORE THAN 1 FULL TURN.  
May 07 09:08:32: Then, wait about ten minutes, slightly rotate the transfer line and retract it before removing  
May 07 09:08:32: the analyzer assembly.  
May 07 10:35:17: Shutdown: Heated Zones are Cool.  
Shutdown: Complete.  
PLEASE TURN OFF CIRCUIT BREAKER AT THE BACK OF THE INSTRUMENT AND  
REMOVE POWER PLUG FROM POWER BEFORE PERFORMING MAINTENANCE.

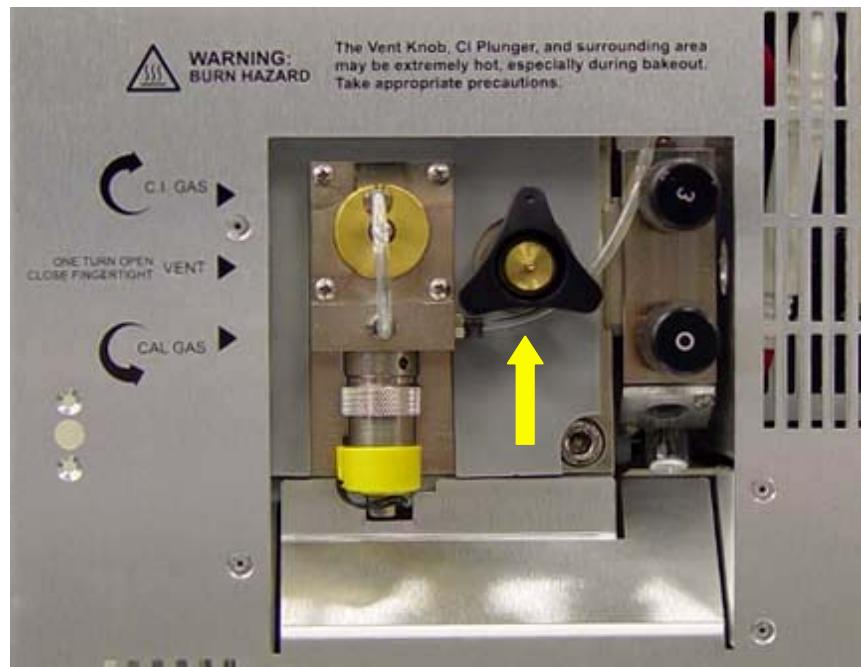
Instrument 1 : May 06 17:04:15 Single Sample Completed. 'Resume' will continue the Open SampleList.

2. Once the shutdown is complete as indicated in the shutdown log window, exit the System Control program and then shut off the turbomolecular pump, foreline pump, and all electronics by turning off the main power switch on the back panel.
3. Disconnect the 4000 MS power cord.



**WARNING:  
BURN HAZARD**

Dangerous high voltages are present. Unplug power cord.



4. Open the front-panel door and turn the vent valve one turn counterclockwise.
5. Listen for the sound of the turbo pumps spinning down and wait until the turbomolecular pump has completely stopped. Leave the vent open for about 10 minutes to allow the pressure to equilibrate.
6. Close the vent valve by turning it clockwise fully.

## Turning Off the Mass Spectrometer with Nitrogen Purge

Purging the vacuum manifold with nitrogen after the trap has partially cooled can reduce the shutdown time.



### WARNING: BURN HAZARD

**Allow heated zones to cool before disassembly.**

1. Shut down the mass spectrometer through the Startup/Shutdown tab in System Control. Click the Shut Down button in the upper left corner of the screen. The heaters will be turned off and the speed of the turbo pump will be gradually reduced to 35% of full speed. It will take a few minutes for this to take place.

Manual Control	Auto Tune	Temperatures	Diagnostics	Startup/Shutdown	Acquisition
Status and Control					
Conditions: Analysis		Shut Down			
State: Ready					
Vacuum System					
Status: Ready					
		Current Set Points		Operating Conditions	
		Heated Zones		Heated Zones	
		Trap Temperature: 220 C		Trap Temperature: 220 C	
		Manifold Temperature: 50 C		Manifold Temperature: 49 C	
		Transferline Temperature: 280 C		Transferline Temperature: 280 C	
		Vacuum System		Vacuum System	
		Pump Spin Speed: 100 %		Pump Spin Speed: 100 %	
				Current: 199 mAmps	



2. Open the front door and attach a source of nitrogen at 5 PSI pressure or less through a polyurethane tube to the barbed fitting at the center of the vent valve.
3. Once the pump spin speed has reached 35% and the trap temperature has fallen to 150 °C, exit the system control program, and then shut off the turbomolecular pump, foreline pump, and all electronics by turning off the main power switch on the back panel.
4. Disconnect the 4000 MS power cord.



## WARNING: SHOCK HAZARD

Dangerous high voltages are present. Unplug power cord.

5. Open the vent valve one turn counterclockwise with the Nitrogen flow on.  
**NOTE: Opening this valve more than one turn risks damage to the equipment.**
6. Listen for the turbomolecular pump to fully spin down, then wait one hour for the trap to cool down.
7. Close the vent valve by turning it fully clockwise. Remove the nitrogen line.

## Moving the Mass Spectrometer Away From the GC

1. Turn off the GC column oven and heater through the 3800 GC keyboard by pressing the Column Oven button and the blue soft key labeled Turn Oven Off.



## WARNING: SHOCK HAZARD

Dangerous high voltages are present. Unplug power cord.

2. Open the GC oven door. Make sure that about 30 cm (12 in.) of the mass-spectrometer end of the capillary column is hanging freely and that the column is not caught on the column rack or cage.
3. Check for sufficient lengths of pneumatics tubing at the back of the instrument to move the spectrometer.
4. While keeping an eye on the capillary column in the GC oven, gently slide the mass spectrometer away from the GC with the transfer line lined up with the GC hole. As you slide the mass spectrometer, take care not to allow the column to bind or kink. When you have fully withdrawn the mass spectrometer from the GC, the distance separating them should be ~23 cm (9 in.). The transfer line should be fully removed from the GC oven.

## Removing the Analyzer Assembly

1. Move the mass spectrometer away from the GC.
2. Remove the top cover from the MS instrument by lifting it up.



**WARNING:  
SHOCK HAZARD**

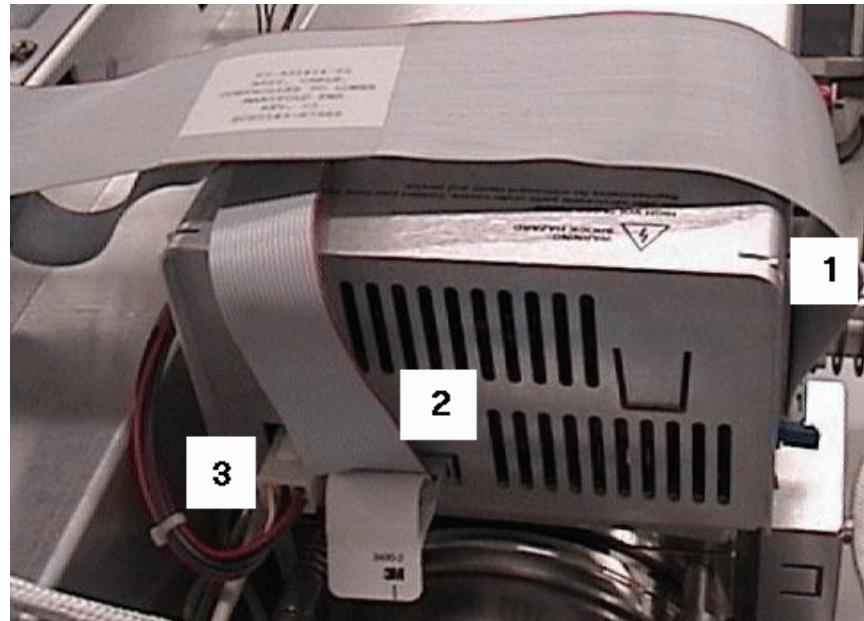
Dangerous voltages exposed when cover is removed. Unplug power cord.



3. **Be sure the transfer line is cool.** Retract the transfer line by grabbing the front nose and turning counterclockwise while pulling out. A mild amount of force may be needed to release residual vacuum. If the transfer line does not pull out, reopen the vent to be sure the analyzer is at atmospheric pressure.



If the transfer line is still difficult to retract, try using the transfer line tool or a 3/16" wrench to twist the end of the transfer line counterclockwise as you retract it. After the transfer line has been fully retracted, lock it into position by turning it clockwise.



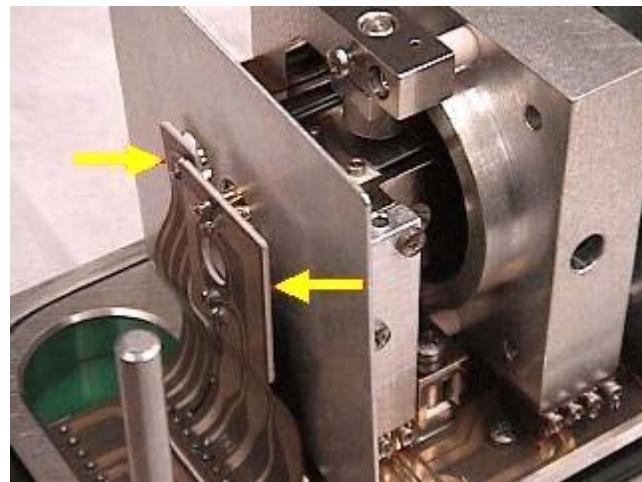
4. Press out on the release tabs to remove the controller to manifold cable (1).
5. Pull on the white pull-tab to remove the manifold lens cable (2).
6. Press down on the locking connector and pull out to remove the manifold power cable (3).

 **CAUTION**

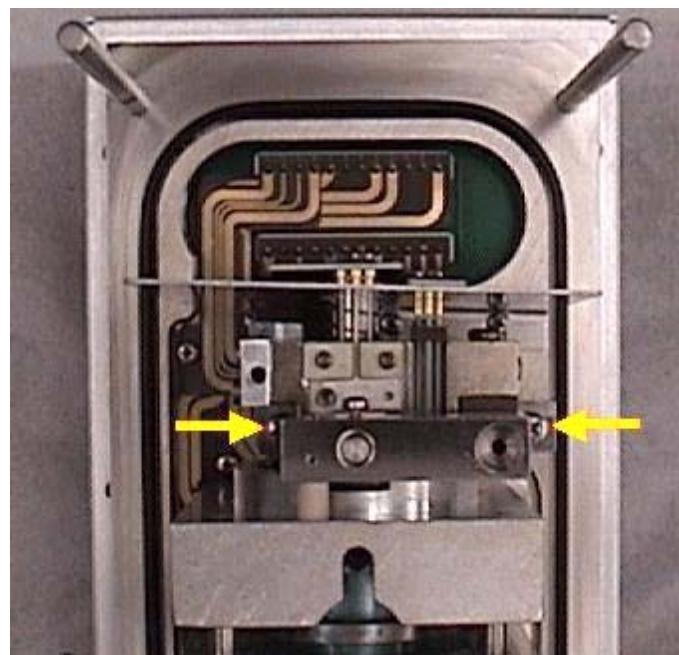
**Verify that the transfer line is fully retracted and locked into position to prevent damaging its tip.**

7. Lift the analyzer assembly up and out of the manifold and place it upside down on the work area.
8. Cover the manifold opening with a Kimwipe® or other low lint material to avoid dust.
9. From this point, use clean tools and wear powder-free gloves.

## Removing the Source/Ion Trap Assembly



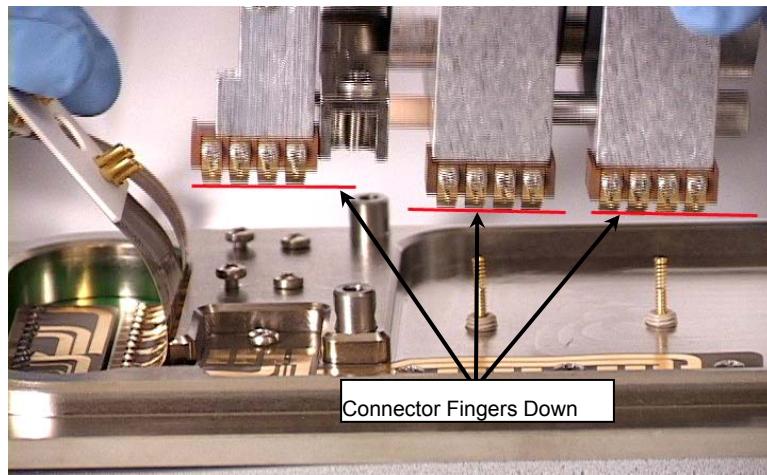
1. Using your thumb and forefinger, gently wiggle each connector attached to the external source or internal ionization assembly, while pulling the connector off the pins. It is best to keep the internal source filament adaptor attached to the flex cable if in internal ionization mode. Use tweezers or pliers to get the connector off if necessary.
2. Loosen the two screws holding on the heat shield and slide it away from the source or remove it.



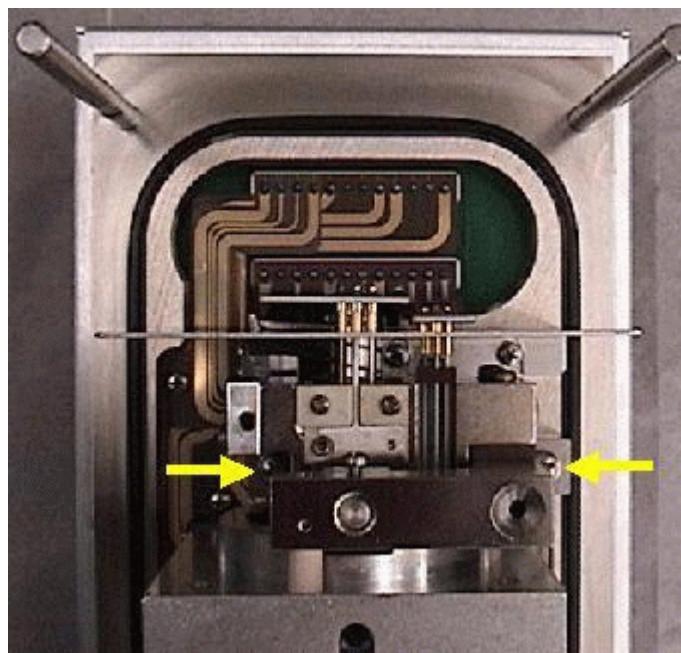
3. Fully loosen the two screws that attach the trap assembly to the top flange.
4. Lift the assembly out and place the assembly on the holder provided (if servicing the ion trap) or on a lint-free cloth with the source facing upwards. Never rest the assembly on its heater connectors or source pins.

## Reinstalling the Source/Ion Trap Assembly

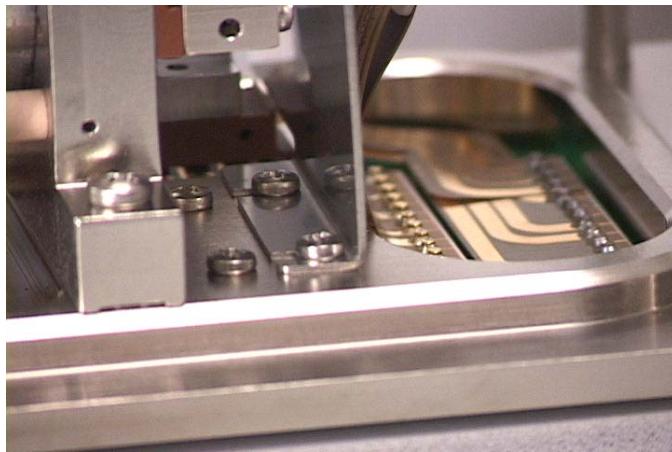
Check to see that all the connection fingers are even as shown. If any fingers are substantially bent, bend them back in line with the other fingers.



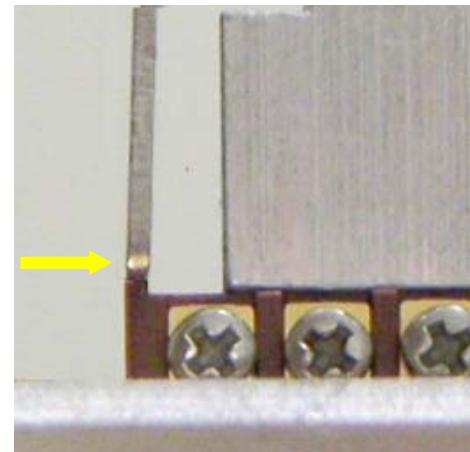
1. Place the Source/Ion Trap assembly on the analyzer flange with the connector fingers down. Align the two screw holes on the flange with the screws on the Source/Ion Trap assembly then tighten the two screws evenly.



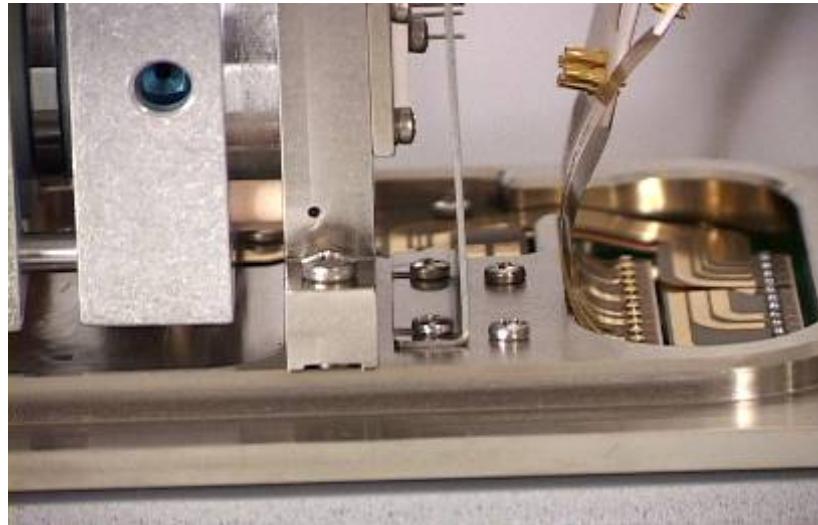
2. Replace the heat shield and tighten the two screws securing it to the flange. If the external source is in place, the shield should be positioned on the rear set of screws. The shield should be positioned on top of the ridge on the source heater block, see figure that follows. If using the internal configuration, the screws should be closer to the center part of the source. Be sure the two screws in the alternate shield position are also tightened down.



*Heat Shield Position with External Ionization*

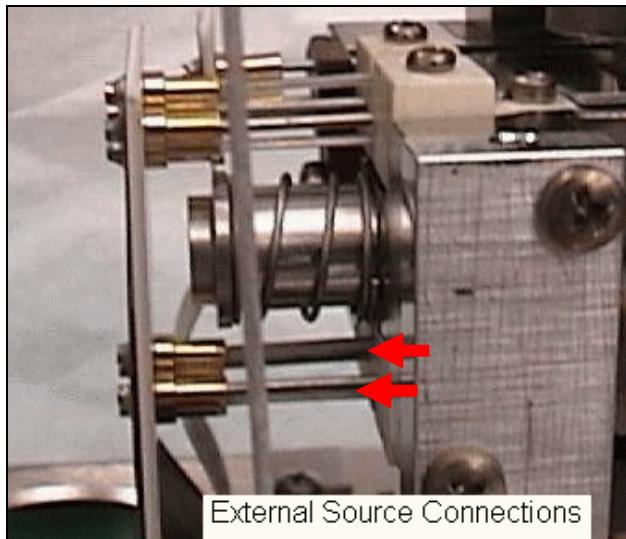


*Shield - Heater Block Alignment*

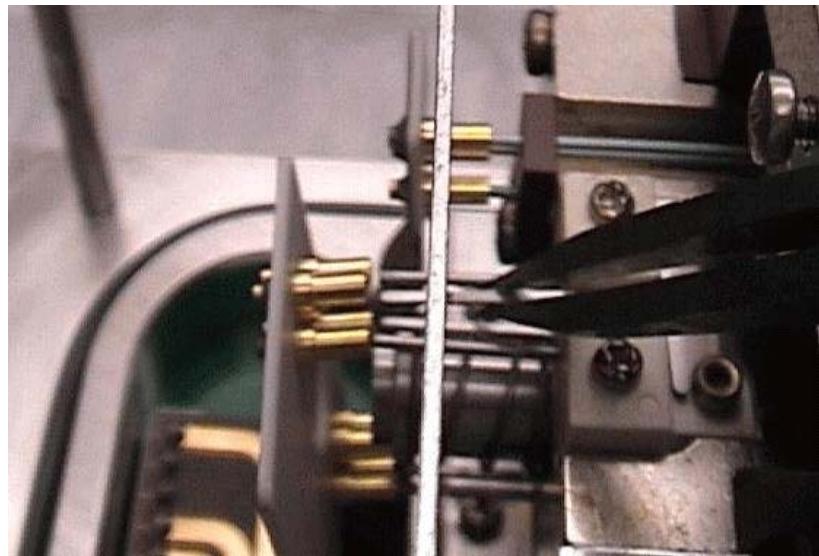


*Heat Shield Position with Internal Ionization Source*

3. Check source connection pins for proper alignment and straighten as necessary.



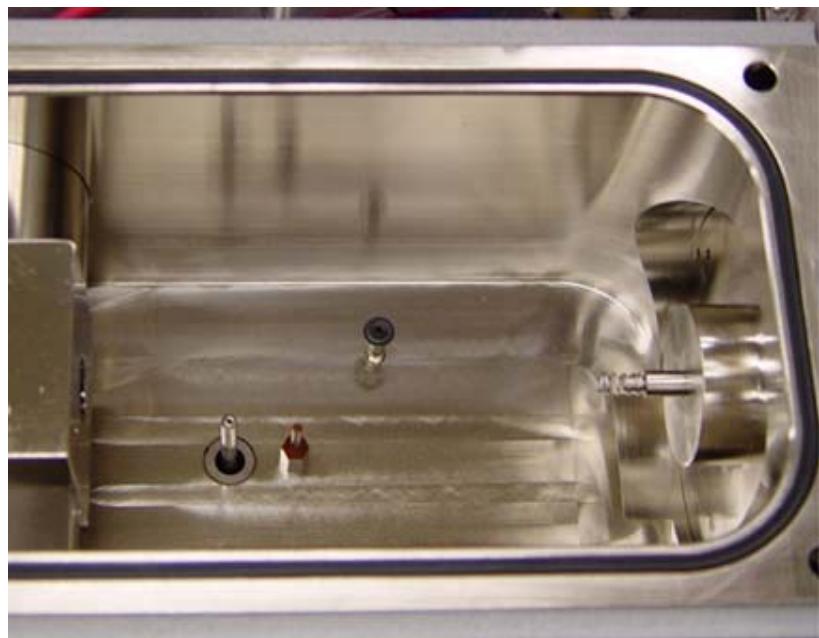
4. Push the connectors onto the source pins. Each pin must be correctly aligned to prevent the pins from being bent.



5. If a pin is not aligned, use a pair of tweezers to move the pin into alignment.

### **Reinstalling the Analyzer Assembly**

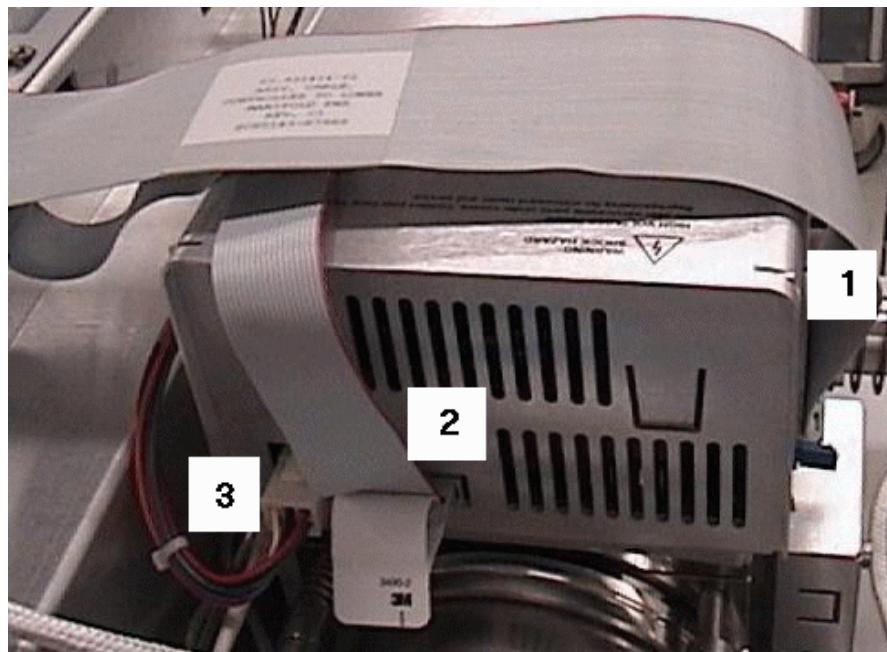
Prior to installing the analyzer, check for any particles inside the manifold or on the analyzer assembly. If necessary, blow out any particles with clean and filtered compressed inert gas. Inspect the upper flange O-ring for particles and clean if needed.



There are three tubes protruding from the bottom of the manifold. Verify that these tubes are pointing straight up. If they are bent more than 20 degrees or damaged, they need to be replaced.



1. Be sure the transfer line is still retracted. The analyzer assembly has four metal pins that need to align with four holes in the manifold. Align these pins and slide the analyzer into the manifold. Ensure the wire harnesses and pneumatic lines between the manifold and bulkhead are not crimped when the analyzer is Reinstalled.



2. Reconnect the three cables associated with the analyzer:

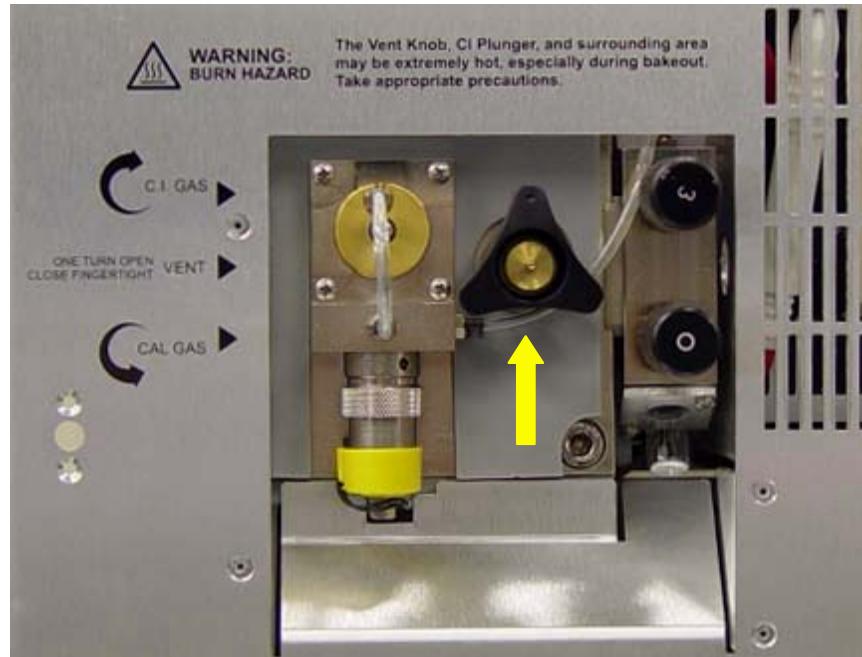
- The controller to manifold cable (1)
- The manifold lens cable (2)
- The manifold power cable (3)



3. Gently push the transfer line assembly towards the manifold to check that it slides all the way into the manifold and does not stop prematurely. If the transfer line stops, remove the analyzer and check the tip on the transfer line. It may be bent and need to be straightened or replaced to ensure proper operation of the transfer line. Also, for external mode, be sure the hybrid mode plug is not in place. Once the transfer line slides all the way in, turn it clockwise to lock it into place.

## Turning On the Mass Spectrometer

1. Make sure the vent in front of the mass spectrometer is closed (turned clockwise completely).



2. Check that all cables are plugged in.
3. Check that the column from the GC is installed properly, the transfer line is locked in its operating position and the GC is operating.
4. Plug in the MS power cable into the rear of the instrument.
5. Turn the power switch on the rear panel to its ON position. The foreline pump should turn on and then stop gurgling after about 10 to 20 seconds. If the pump continues to gurgle then check that the analyzer assembly is seated on the manifold properly, (there should be no gaps).
6. Start up the System Control program.
7. Go to the Startup/Shutdown tab dialog in System Control if the program doesn't automatically start there.

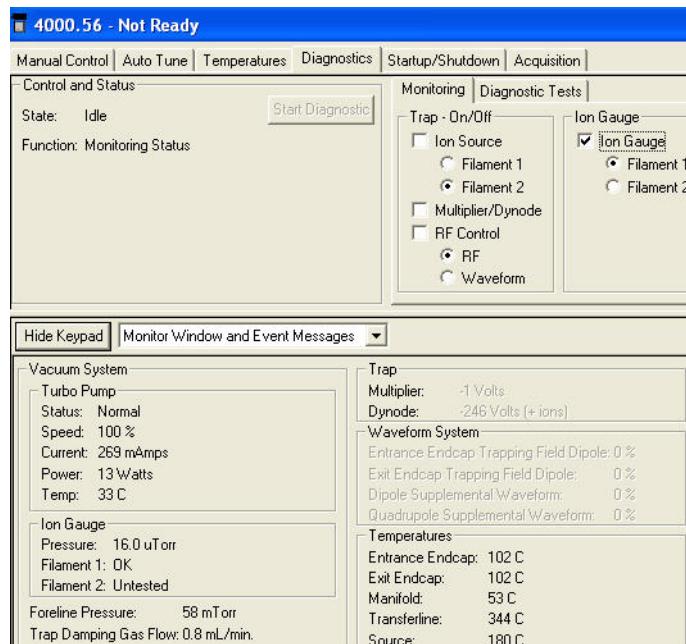
Manual Control	Auto Tune	Temperatures	Diagnostics	Startup/Shutdown	Acquisition
Status and Control		Current Set Points		Operating Conditions	
Conditions: Analysis	<input type="button" value="Shut Down"/>	Heated Zones		Heated Zones	
State: Ready		Trap Temperature: 220 C		Trap Temperature: 221 C	
Vacuum System		Manifold Temperature: 50 C		Manifold Temperature: 49 C	
Status: Ready		Transferline Temperature: 250 C		Transferline Temperature: 252 C	
Pneumatics		Vacuum System		Vacuum System	
Damping Gas: Off	<input type="button" value="Turn On"/>	Pump Spin Speed: 100 %		Pump Spin Speed: 100 %	
Getter Control		Pneumatics		Current: 214 mAmps	
Heater: Off	<input type="button" value="Turn On"/>	Flow Rate: OFF		Flow Rate: 0.0 mL/min.	
		Getter Control		Inlet Pressure: 85 PSI	
		Temperature: OFF		Temperature: 25 C	

*Startup/Shutdown page*

8. Go to “Checking the Vacuum Status” on page 48.
9. If in external or hybrid mode, turn on the damping gas and getter heater using the buttons in the lower left of the startup/shutdown dialog.
10. Go to “Baking Out the Mass Spectrometer” on page 49.
11. Go to “Checking Ion Trap Operation” on page 49.

## Checking the Vacuum Status

Select the Diagnostics tab in System Control.



### Vacuum System Field

The vacuum readings (at the lower left of the screen) tell a lot about the state of the MS after pump down (and during operation). Typical operating ranges for the 4000 MS in internal mode are:

Speed	100%
Current	200 – 300 mA
Power	9 – 13 Watts
Ion Gauge Pressure	< 20 $\mu$ Torr
Foreline Line	< 50 mTorr

If the Pump Spin Speed does not steadily increase, there may be a leak in the system. Large leaks will be indicated by a turbo speed less than 100%. Small leaks will show up by an increase in the pump current once at 100% or in the ion gauge pressure (See **Diagnostics Mode** section in the 4000 GC/MS Operation Manual, 03-914999-00.) Small leaks are diagnosed by changes in the ion gauge reading and can be pinpointed using the leak check section in the internal or external service method. For more detail on troubleshooting leaks, go to the “Troubleshooting” section (page 107) on checking for air leaks.

## Baking Out the Mass Spectrometer

Any time the system is vented you should bake out the system to eliminate water and contaminants in the vacuum manifold.



### WARNING: BURN HAZARD

The vent knob, CI plunger and surrounding area may be extremely hot, especially during bakeout. Take appropriate precautions.

To bake out the Mass Spectrometer, proceed as follows:

1. Open the System Control and click on the Temperatures tab dialog.
2. Select **Bakeout** and enter a bakeout time of 2 to 6 hours.
3. Use the following temperatures:
  - Trap at 220 °C or 10 °C higher than the analysis temperature.
  - Transfer line at 280 °C.
  - Manifold at 120 °C.
  - Source at 220 °C.
4. Click on Start Bakeout.

## Checking Ion Trap Operation

To check the ion trap operation, proceed as follows:

1. Once bakeout is finished, re-establish the analysis temperature in the trap for at least 2 hours to achieve thermal equilibrium.
2. The manifold temperature should be at or below 50 °C.
3. Run Auto Tune.
4. Open the Manual Control page and activate the “C:\VarianWS\4000MSservice\4000 MS Int (or Ext) Service.mth” file.
5. Use the second segment of the method to check the background. Turn on the ion trap. The ionization time should be above 20,000 µsec. If the ionization time is below 20,000 µsec, continue to bake out the trap or check the GC for contamination.

# Cleaning Procedures

The cleanliness of the sources, ion trap and conversion dynode can have a significant impact on the performance of the mass spectrometer. The frequency of cleaning depends on the quantity and nature of the samples run, so no standard cleaning interval can be recommended. The troubleshooting guide in this manual describes some of the symptoms that result from dirty components and makes recommendations about when to perform cleaning procedures.

## Cleaning the External Source

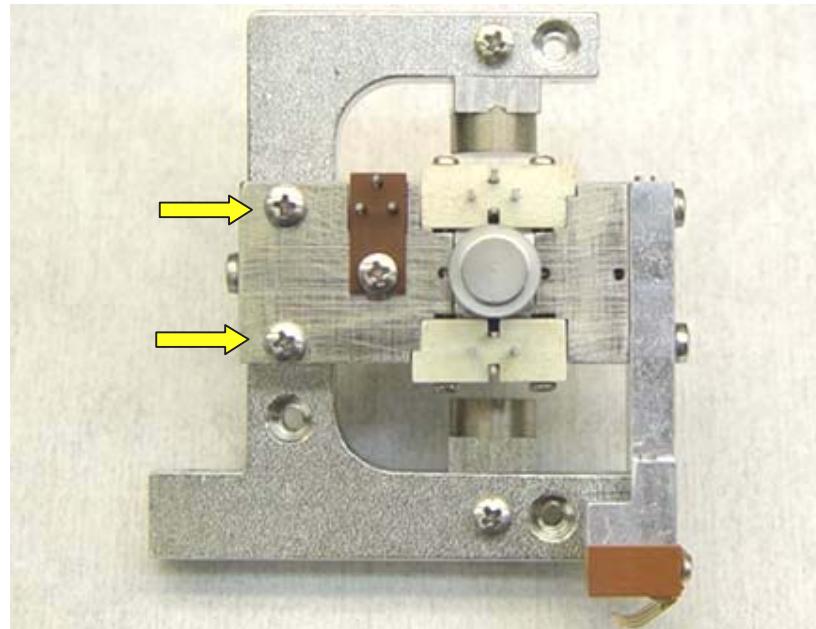


### WARNING: SHOCK HAZARD

Dangerous high voltages are present. Unplug instrument power cord.

- Go to “Turning Off the Mass Spectrometer” on page 34.
- Go to “Removing the Analyzer Assembly” on page 37.
- Go to “Removing the Source/Ion Trap Assembly” on page 41.

### *Removing the Source Holder*



1. Remove the two screws holding the source to the magnet structure.
2. Lift off the source assembly and place on its side on a lint-free cloth.

### ***Removing the Lenses***



---

**NOTE:** The lens parts are anodized to insulate each from the other. The anodizing process creates a black coating on the surface of these aluminum parts. Scratches in the coating can create a conductive path after re-assembly.

---

1. Place each part on a lint-free cloth after removing.
2. Remove the lens insulator.
3. Remove the lens holding screw.
4. Remove each lens.

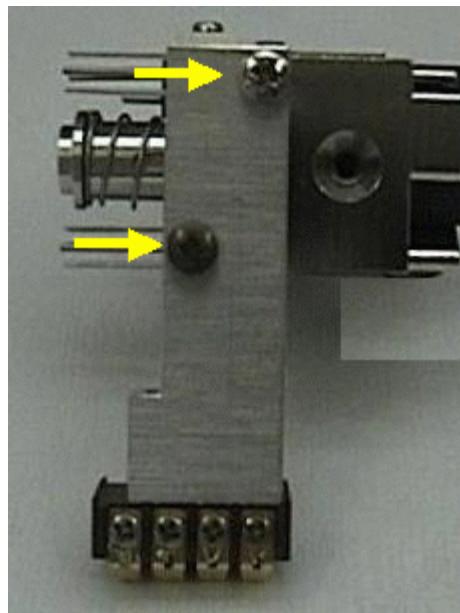


### ***Cleaning the Lenses***

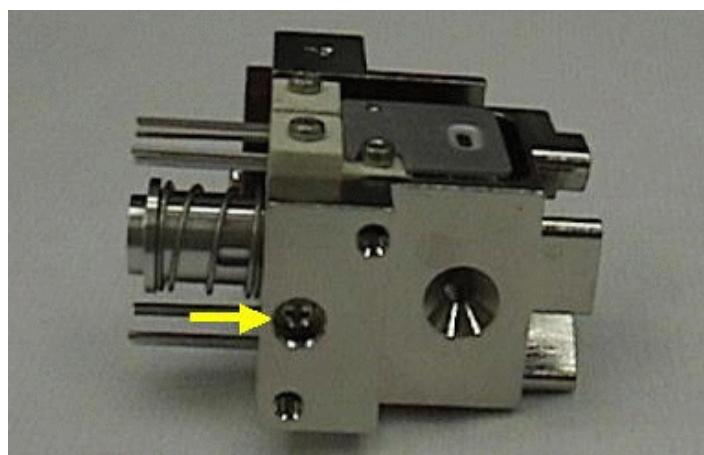
To clean the lenses you will need the following items:

- Cotton swabs
  - Isopropyl Alcohol or Methanol
  - Beakers
  - Ultrasonicator
1. Clean the center shiny part of each lens with a cotton swab and isopropyl alcohol or methanol.
  2. Sonicate the lenses in IPA or methanol for 1 minute.
  3. Dry the parts in air or in an oven set to approximately 120 °C for 30 minutes.

### ***Removing the Ion Volumes***



1. Remove the two source heater block screws.
2. Place the source heater block on a lint-free cloth.



3. Loosen the ion volume retaining screw until the spring pushes the CI ion volume out.

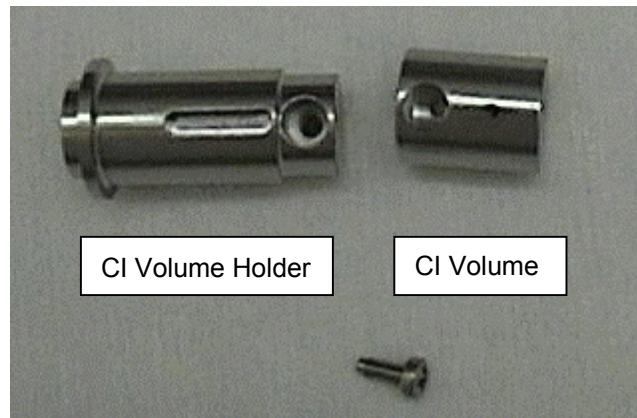


4. Turn the source assembly so the EI ion volume can fall out. If the ion volume does not fall out, loosen the source screw until it does fall out.

### ***Cleaning the EI/CI Ion Volumes***

To clean the ion volumes you will need the following items:

- Aluminum oxide
- Cotton swabs
- De-ionized water
- Isopropyl Alcohol or Methanol
- Beakers
- Ultrasonicator



1. Remove the spring from the CI ion volume.
2. Remove the screw to separate the ion volume from the ion volume holder.
3. Rinse the ion volume and holder with de-ionized water and sonicate them in de-ionized water for 2 minutes.
4. Sonicate another 2 minutes in isopropyl alcohol or methanol.

5. If the volume is discolored, perform the following steps.
  - Dip a cotton swab in de-ionized water and then aluminum oxide.
  - Gently scrub any discolored areas with slurry of aluminum oxide and water

---

NOTE: Do not allow the aluminum oxide to dry on the surface.

---

- Repeat steps 3 and 4 on the parts cleaned with aluminum oxide
6. Repeat these steps for the EI ion volume.
  7. Dry all the parts in air, or in an oven set to approximately 120 °C for 30 minutes.

### ***Re-Assembling the Cl Ion Volume***



1. Line up the large holes in the holder and Cl ion volume.
2. Slide the Cl ion volume onto the holder.
3. Place the screw through the Cl ion volume into the holder and gently tighten. While tightening, ensure the tip of the screw remains centered in the hole in the Cl ion volume.

---

NOTE: Do not over tighten. Over tightening may cause the Cl ion volume to bulge and stick when moving in and out of position. Check to be sure the volume maintains its cylindrical shape.

---

4. Slip the spring over the assembly.

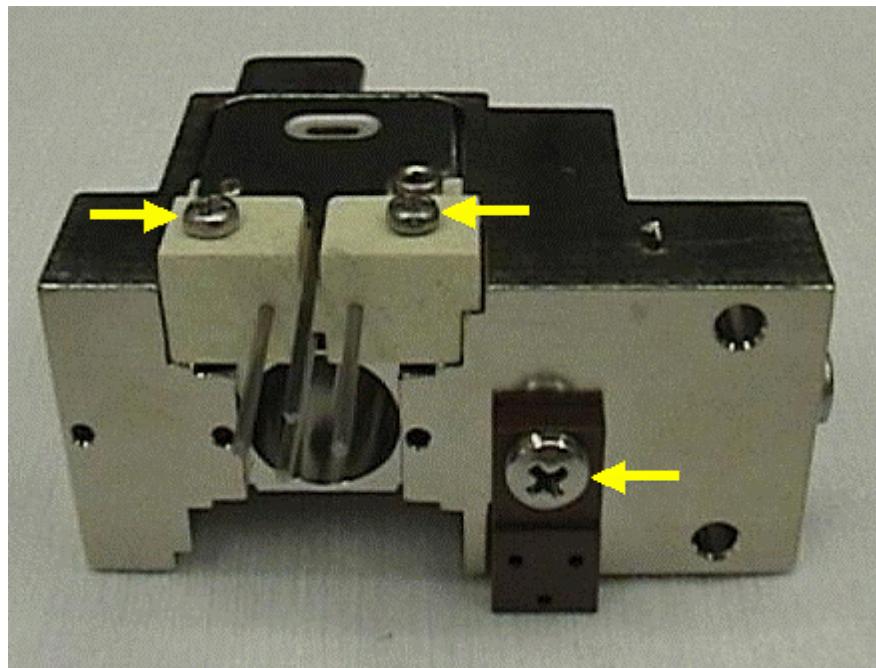
### ***Cleaning the Filaments and External Ion Source Block***

The filaments and source block normally do not require cleaning except when high pressure CI is performed. High pressure CI can coat the filaments with a carbon layer that needs to be cleaned to prevent leakage currents.

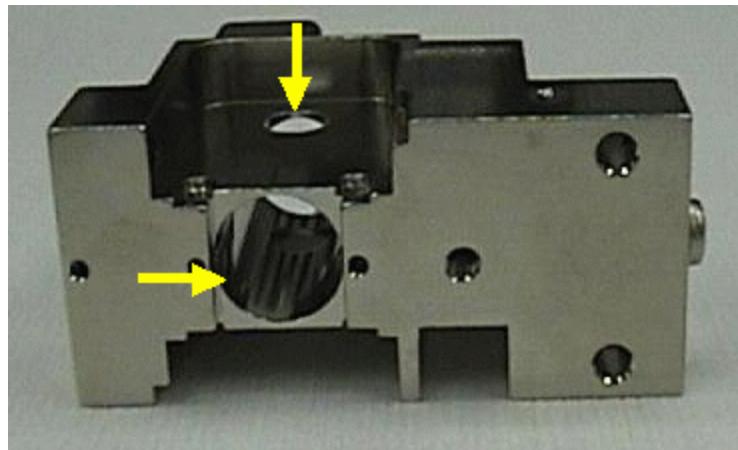
To clean the source block you will need the following items:

- Aluminum oxide
- Cotton swabs
- De-ionized water
- Isopropyl Alcohol or Methanol
- Beakers
- Ultrasonicator

### *Removing the Filaments*



1. Remove the two filament screws.
2. Lift out the filament and place on a lint-free cloth.
3. Remove the lens insulator screw and lens insulator.
4. Turn the source over and remove the second filament.



### *Cleaning the Source Block*

1. Use a cotton swab and slurry of aluminum oxide and de-ionized water to clean the ion volume hole and the filament entry holes.

---

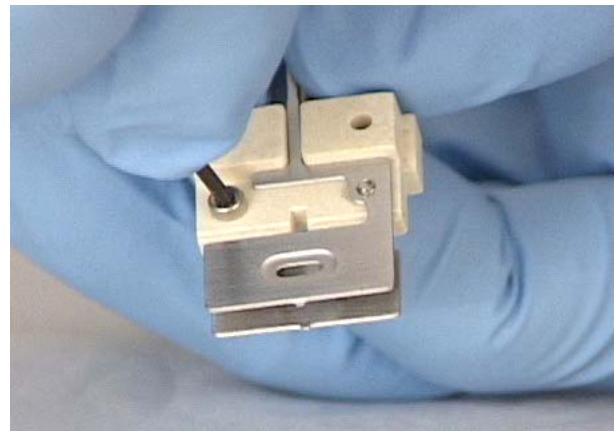
NOTE: Do not allow the aluminum oxide to dry on the source block

---

2. Rinse thoroughly with de-ionized water.
3. Sonicate in de-ionized water for 2 minutes.
4. Sonicate in isopropyl alcohol or methanol for 2 minutes.
5. Dry the parts in air, or in an oven set to approximately 120 °C for 30 minutes.

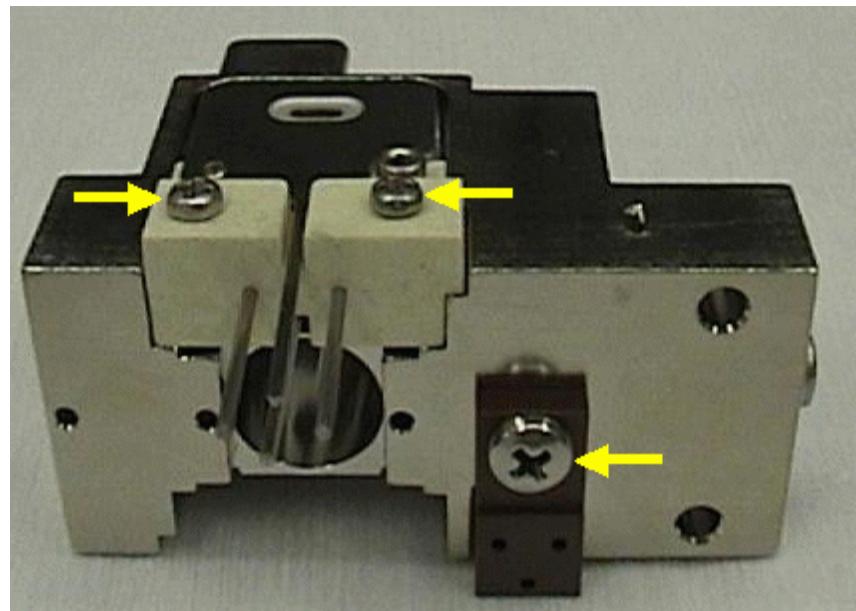
### ***Cleaning the Filaments***

1. Disassemble the lenses from filament base by removing the two socket cap screws using a 1.5 mm. Allen wrench.



2. Clean the base near and between the filament posts with a piece of super fine grade (400 grit) silicon carbide sand paper until most discoloration disappears. Be very careful not to touch or deform the filament during the process.
3. Using a sharp razor blade (or tip of utility knife), scrape off the same areas as thinly as possible, until a surface similar in color to the original base is exposed.
4. Wash off any powder and contamination with isopropyl alcohol or methanol. Dry the filament before installing.
5. Clean the lenses with a cotton swab and isopropyl alcohol or methanol. It is recommended to use a second swab after initial cleaning.
6. Reassemble the lenses onto the filament base.

### ***Reinstalling the Filaments***

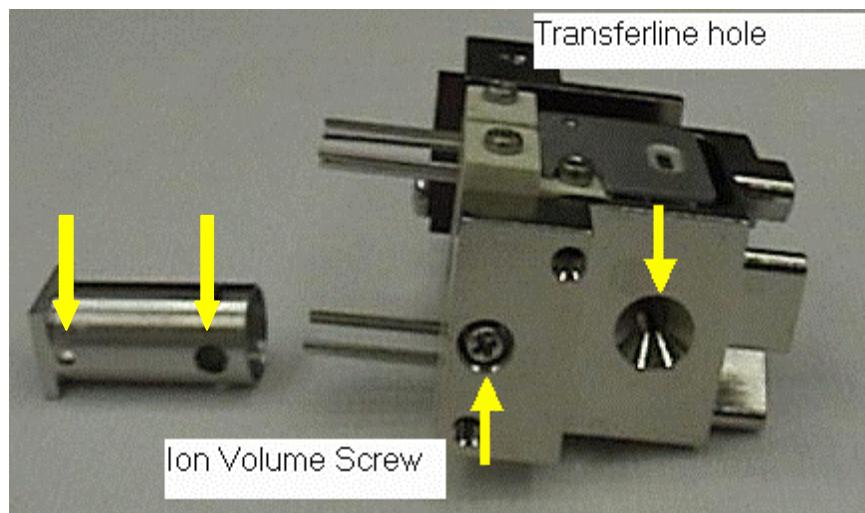


1. Install the lens insulator and lens insulator screw. Be sure the step in the lens insulator fits into the cut-out in the source.
2. Place a filament assembly into the source with the notched side down. Be sure the assembly is fully seated in place.
3. Place the two screws into the screw holes and tighten each screw evenly. Do not over tighten.
4. Turn the source assembly over and repeat the steps for the other filament.

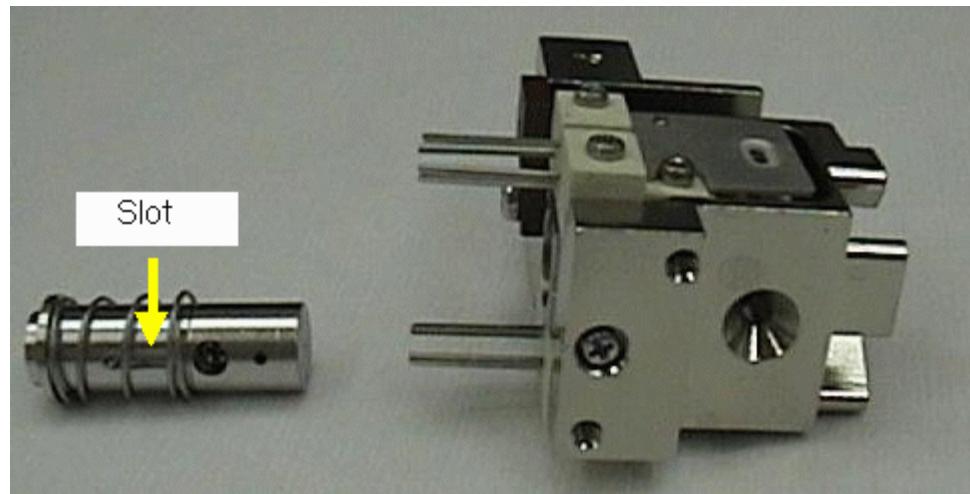
### ***Reassembling the External Source***

Reassembling the external source essentially reverses the process used to disassemble the source.

### ***Reinstalling the Ion Volumes***



1. Align the EI volume as shown with large and small holes on opposite ends. These holes need to align with the ion volume screw and transfer line hole.
2. Slide the EI ion volume into the source block.

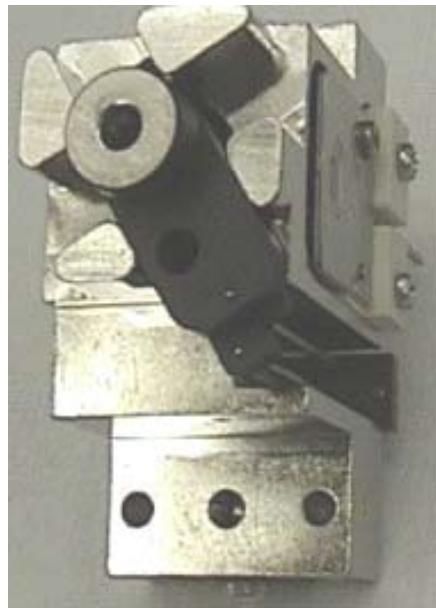


3. Slide the CI ion volume into the source block so that the slot is aligned with the ion volume screw.
4. Fully compress the spring. Ensure the proper hole in the CI ion volume aligns with the transfer line hole.
5. Hold the CI ion volume in and slowly tighten the ion volume screw until it stops. The CI ion volume should be captured by the screw entering the slot, but still be able to freely slide in and out of the EI ion volume. Adjust the slot position so that the CI ion volume slides freely after the screw is tightened.

### ***Reinstalling the Lenses***



1. Reinstall lens 1. The pin should slide through the left hole in the insulator.



2. Reinstall lens 2. The pin should slide through the middle hole in the insulator.



3. Reinstall lens 3. The pin should slide through the right hole in the insulator.



4. Reinstall the screw insulator and the lens screw through the lens and into the source.

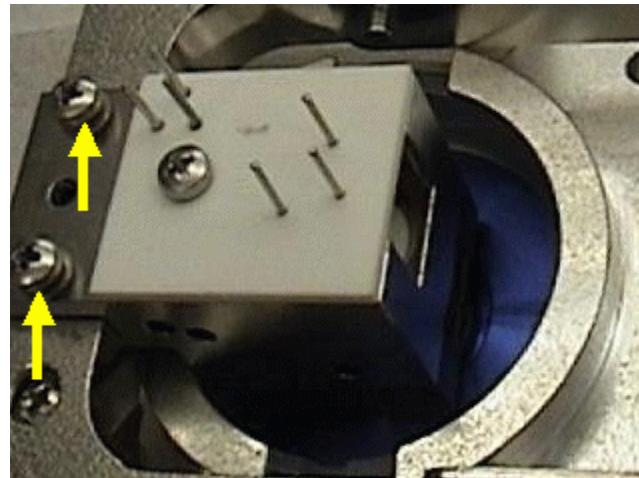


5. Push the centering ring onto lens 3.
6. Reinstall the external source assembly and tighten the two source mounting screws. Maintain source symmetry in the assembly while tightening the screws.
7. Reinstall the source heater assembly.
  - Go to “Reinstalling the Source/Ion Trap Assembly” on page 41.
  - Go to “Reinstalling the Analyzer Assembly” on page 44.
  - Go to “Turning On the Mass Spectrometer” on page 46.

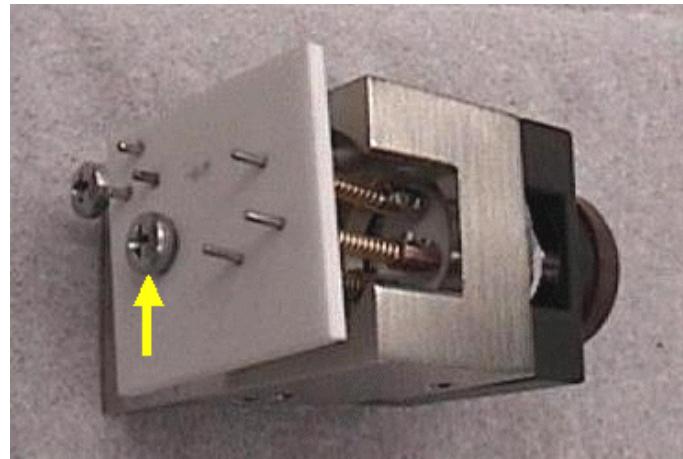
## Cleaning the Internal Ionization Assembly

- Go to “Turning Off the Mass Spectrometer” on page 34.
- Go to “Removing the Analyzer Assembly” on page 37.
- Go to “Removing the Source/Ion Trap Assembly” page 40).

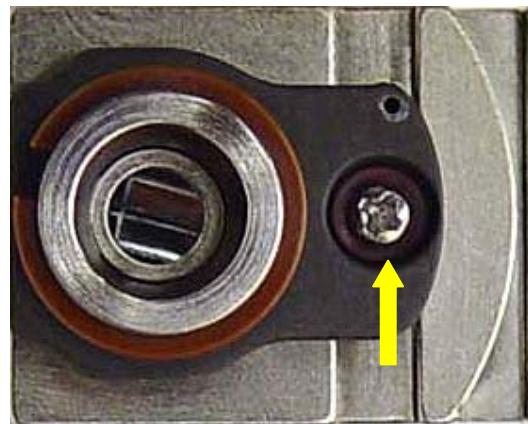
## *Removing the Internal Ionization Assembly*



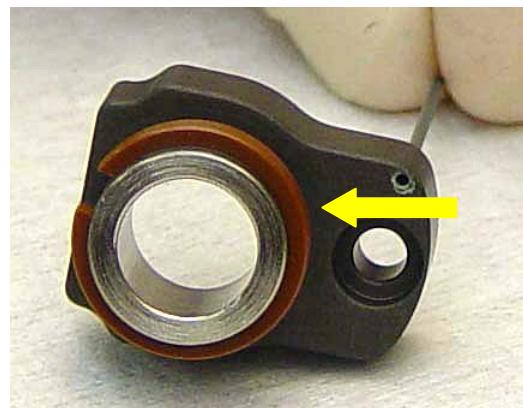
1. Place all parts on a lint-free cloth.
2. Loosen the two screws holding the internal ionization assembly until it can be lifted out. The screws are captured in the source plate.
3. Lift out the assembly and place it on a lint-free cloth.



4. Remove the filament retention screw.
5. Remove the ceramic plate.
6. Remove the filament assembly.



7. Remove the gate retaining screw.



8. The Center Ring is clipped around the end of the lens. Push a capillary pick into the gap in the ring, lift and slide the ring over the edge of the gate.
9. Pull the insulator off the lens.

### ***Cleaning the Gate***

To clean the gate you will need the following items:

- Cotton swabs
- Isopropyl Alcohol or Methanol
- Beaker
- Ultrasonicator

---

NOTE: The outside of the gate is anodized. Do not scratch the coating or the gate may short to the filament block.

---

1. Clean the shiny center of the gate with a cotton swab and IPA or methanol.
2. Sonicate in isopropyl alcohol or methanol for 2 minutes.
3. Dry in air or in an oven set to approximately 120 °C for 30 minutes.

### ***Cleaning the Internal Source Base***

To clean the Internal Source Base you will need the following items:

- Aluminum oxide
- Cotton swabs
- De-ionized water
- Isopropyl Alcohol or methanol
- Beaker
- Ultrasonicator



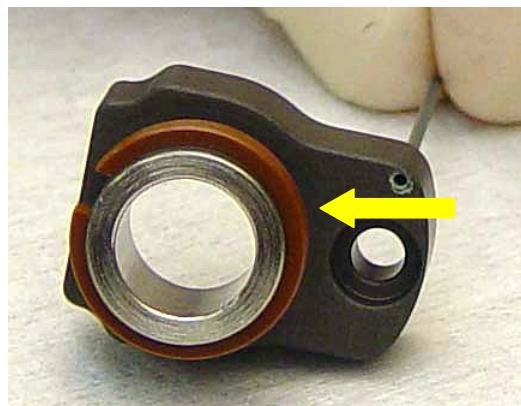
---

NOTE: Do not allow the aluminum oxide to dry on the base.

---

1. Clean the center tube in the Internal Ionization Base with a cotton swab using slurry of aluminum oxide and DI water.
2. Rinse thoroughly with DI water.
3. Sonicate in DI water for 2 minutes.
4. Sonicate in isopropyl alcohol or methanol for 2 minutes.
5. Dry in air or in an oven set to approximately 120 °C for 30 minutes.

### ***Re-assembling the Internal Ionization Assembly***



1. Push the Center Ring over the gate so it snaps into the groove around the edge.



2. Place the Gate on the Internal Source Base.
3. Insert the Insulator into the screw hole.
4. Place the screw into the Insulator and tighten.



5. Place the assembly into the Internal Source Base.

6. Reinstall the ceramic plate over the protruding pins.



7. Be sure the filament assembly is seated fully flat in the Internal Source Base.
8. Push down on the ceramic plate while installing the holding screw and tighten.

## Cleaning Ion Trap Components

### *Disassembling the Ion Trap*



1. If the source is left in place, place the trap assembly on the holder provided, with the source pins facing down.
2. Remove the four retaining screws.
3. Lift off the trap oven and place on a lint-free cloth.
4. If necessary, remove the quartz spacer from the trap oven if it is going to be cleaned.
5. Lift out each trap electrode and quartz spacer and place them on a lint-free cloth.

---

NOTE: Endcaps must be placed cone side up to avoid damage to the electrode.

---

## ***Cleaning the Silica Coated Electrodes***



### **CAUTION**

**DO NOT use aluminum oxide, other abrasives or harsh laboratory cleaners because this will remove the silica layer on the trap! Use only mild detergent (pH between 6 and 7.5).**

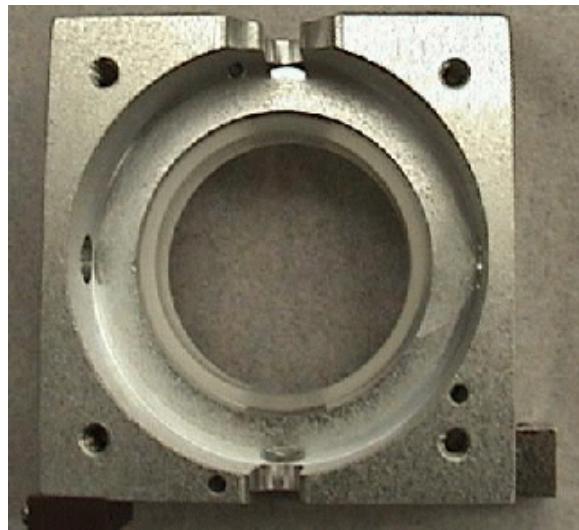
The protective surface layer of the silica-coated ion trap electrodes is very thin (only about 1  $\mu\text{m}$ ) but durable and it is strongly bonded to the bulk stainless steel body. However, **abrasives** such as aluminum oxide powder **must not be used** to clean the trap electrodes, because this will definitely destroy the silica layer! **Strongly acidic or strongly basic laboratory cleaners must not be used** to clean the trap parts because they will also remove the silica layer!

- Remove Polyimide banana plugs from the end caps.
  - Use a toothbrush and liquid hand soap or dish detergent (pH between 6 and 7.5) to gently scrub the trap parts.
  - Rinse in de-ionized water.
  - Rinse in methylene chloride or methanol.
1. Air dry or dry in an oven set to approximately 120 °C for 30 minutes.
  2. Replace the banana plugs, preferably in a different hole than prior to cleaning.

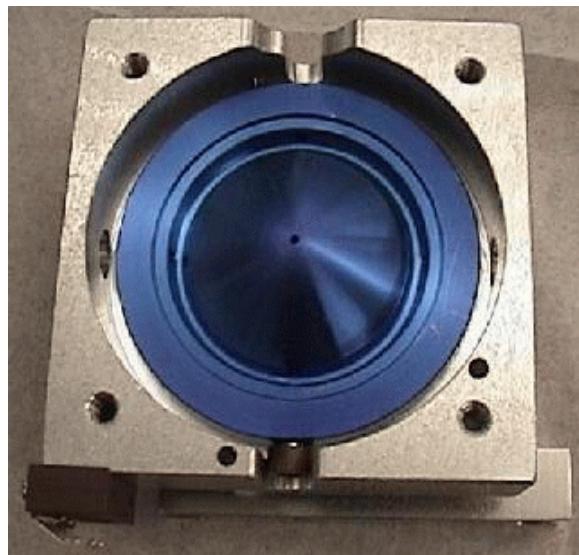
## ***Cleaning the Quartz Spacers***

1. Wipe all surfaces of the four quartz spacers with a clean, soft, lint-free cloth that has been dampened with reagent-grade acetone. Take care to avoid extraction of glove material by the acetone.
2. Rinse each of the quartz spacers with de-ionized water.
3. Rinse in isopropyl alcohol or methanol.
4. Dry the spacers in air or in an oven set to approximately 120 °C for 30 minutes.

### ***Re-assembling the Ion Trap***



1. Place the first quartz spacer in the bottom half of trap oven. Be sure the quartz is properly seated in the oven. The outside edge should have the same spacing around the perimeter and the quartz should not move when touched. The orientation of the notch in the spacer is not important.



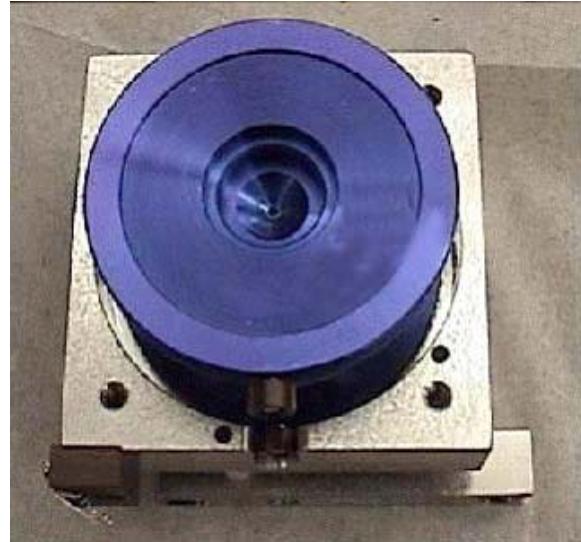
2. The two end cap electrodes are identical. Place the end cap electrode on the quartz spacer, cone side up, with the banana plug on the same side as the gold connectors. The handle of a wooden handled cotton swab may be helpful to gently guide the electrodes into the assembly.



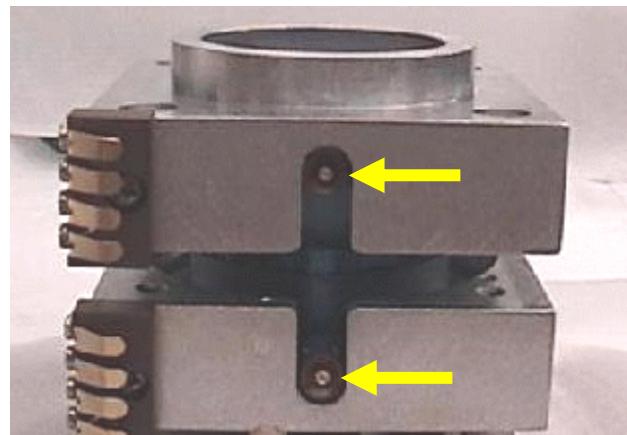
3. Place another quartz spacer on the end cap. Be sure the spacer is seated completely flat on the end cap.



4. Place the ring electrode on the quartz spacer.
5. Place another quartz spacer on the ring electrode. Be sure the spacer is seated completely flat on the electrode.



6. Place the last end cap electrode on the quartz spacer cone side in. The banana plug should be on the same side as the lower end cap.
7. Place the last quartz spacer on the end cap electrode. Be sure the spacer is seated completely flat on the end cap.
8. Place the oven top on the electrode stack with the gold connectors on the same side as the lower half. Check the oven and quartz spacers. There should not be any gaps. If the quartz is a very tight fit, try placing the quartz into the trap oven before putting the trap oven on the electrode stack.



---

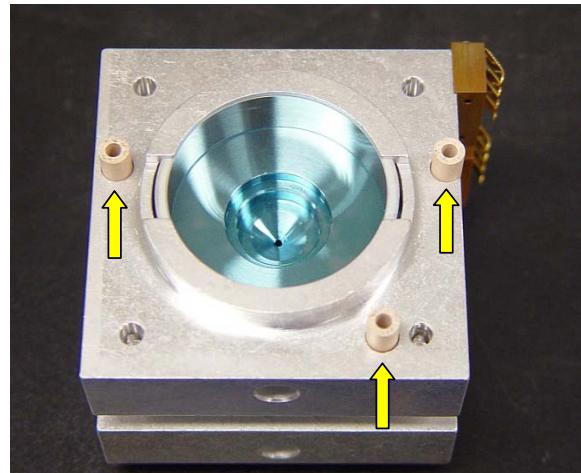
NOTE: The gold connectors on the trap ovens should line up on the same side. The banana plugs should be visible in the notches in the trap oven and should be seated all the way to the end of the grooves in the trap oven. If there is a gap, disassemble and recheck the alignment of the spacers and electrodes.

---

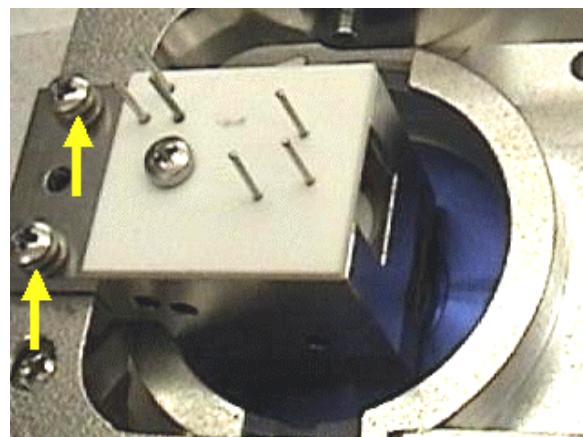


9. Reinstall the four screws and tighten them evenly until they stop.

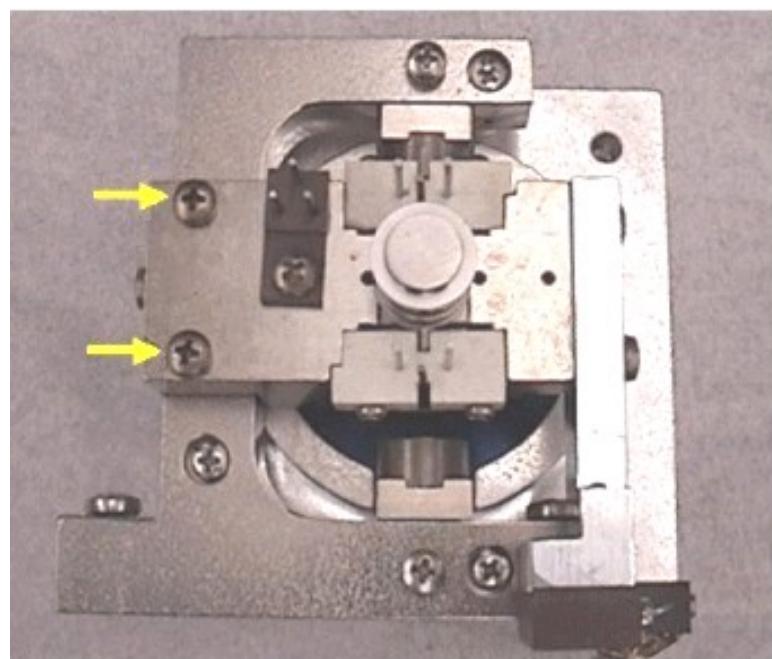
#### ***Reinstalling the Source***



1. With the trap oven screws on the bottom, place the ceramic spacers in their countersunk holes.
2. Align the magnet structure with the three ceramic spacers, insert the screws and tighten.
3. Place the source into the end cap and align the two screws. When the internal source is properly installed, the lens sits flat in the end cap and the source plate is flat against the magnet structure. Check for proper positioning then tighten the screws.



*Internal Source*



*External Source*

- Go to “Reinstalling the Source/Ion Trap Assembly” on page 41.
- Go to “Reinstalling the Analyzer Assembly” on page 44.
- Go to “Turning On the Mass Spectrometer” on page 46.

# Replacing a GC Column

## Tools and Materials Required:

- 3/16" wrench
- Ceramic scoring wafer
- 5/16" wrench
- Scribing tool
- Graphite/Vespel® ferrule
- Column measuring tool: 03-931805-01 (for internal mode)
- Methanol
- Lint free cloth

## Removing the Capillary Column from the System

1. Go to “Moving the Mass Spectrometer Away From the GC” on page 37.
2. Go to “Turning Off the Mass Spectrometer” on page 34.



### WARNING: SHOCK HAZARD

Dangerous voltages are present. Unplug power cord.



3. Use a 3/16" and a 5/16" wrench to loosen the brass nut on the end of the transfer line.
4. Remove the capillary column from the transfer line.
5. Remove the brass nut with ferrule from the column.
6. Remove the ferrule from the nut. Discard the ferrule. Alternatively, a new column nut can be used (03-949551-00).
7. From inside the GC oven, pull the transfer line end of the column back into the hole in the side of the GC. Leave the free end of the column on the floor of the oven.
8. Use a 5/16" wrench to loosen the capillary column nut that secures the column to the injector.

9. Carefully remove the nut, ferrule, and column from the injector.
10. Slide the column nut, along with the ferrule, off the end of the column if desired.
11. Carefully lift the column support cage, along with the column, from the column hanger and remove from the oven.
12. Seal the end of the column or insert the ends of the column into a septum.
13. Store the column and the support cage.

## Installing a New Capillary Column in the System

1. Remove the 4000 MS Top Cover



**WARNING:  
BURN HAZARD**

Dangerous voltages are present. Unplug power cord.

2. Unplug the transfer line heater cable from connector J37 on the bulkhead.



**WARNING:  
BURN HAZARD**

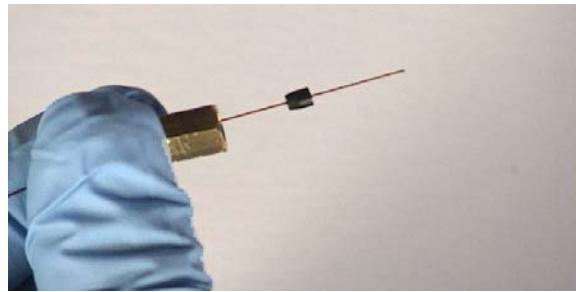
Confirm that the transfer line is cool.

3. Grasp the nose of the transfer line; then rotate counterclockwise as you press lightly toward the manifold. Gently slide the transfer line away from the manifold.
4. Remove the nose clip, and then pull the transfer line away from the analyzer.

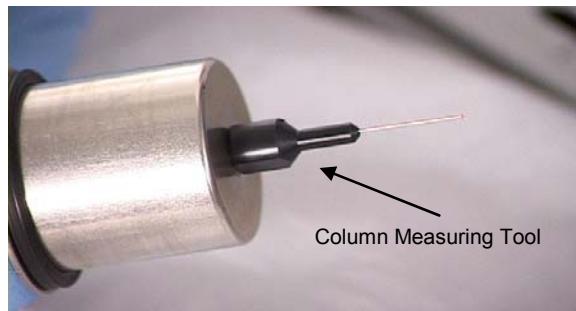


5. Wrap the transfer line in clean lint-free material and place it on a clean, dry surface.
6. Unwind about 60 cm (24") of the mass spectrometer end of the column from the support cage in the GC.
7. Place the column and its cage onto the column rack inside the GC oven.

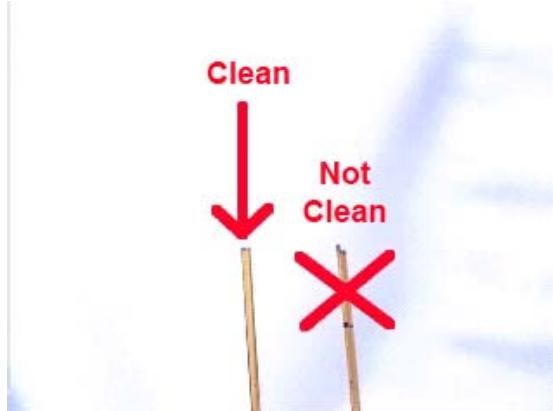
8. Install the GC end of the column into the GC injector (see GC manual for instructions).
9. Purge the column inside the GC oven with carrier gas for at least 15 minutes to remove residual air.
10. It is advised that you condition the column in the GC oven before connecting to the MS to prevent contamination. Do not exceed maximum allowable operating temperature for the column.
11. Insert the MS end of the column through the transfer line hole in the right side of the GC.
12. Slide a brass nut onto the column and slide the nut several inches down the column. The wide, threaded opening of the nut should face the end of the column.
13. Place a new graphite/Vespel ferrule on the column with the taper facing the nut. Slide the ferrule, along with the nut, about 30 cm (12") down the column.



If the system is in internal mode, replace the transfer line tip with the column-measuring tool. If the measuring tip is not available, a ruler will be needed to measure the extension length.



14. Carefully insert the tip of the column into the nose end of the transfer line. Slide the column all the way through the transfer line until the tip of the column projects a few inches beyond the transfer line tip.
15. Using a ceramic scoring wafer, score the column once lightly about 2 cm (1") from its end.

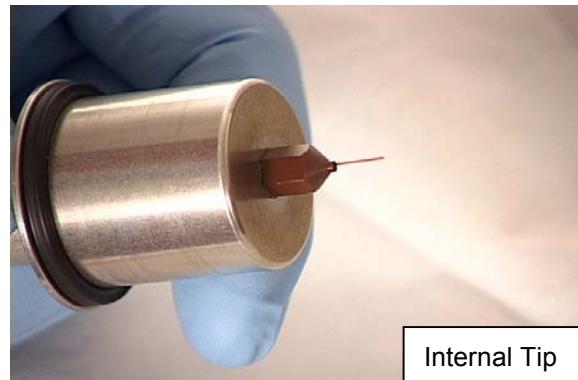


16. Bend the column slightly to break it at the mark. The column should break cleanly.
17. Using a Kimwipe® tissue dipped in methanol, carefully wipe the last 15 cm (6.0 in.) of the column. Be sure to wipe toward the end of the column so that the Kimwipe tissue fibers do not enter the opening at the column end.
18. Install the brass nut on the end of the transfer line, but do not tighten the nut completely.
19. Position the tip of the column so that about 1 mm (1/32") extends beyond the transfer line tip for External mode. If in Internal or Hybrid mode, the column should just barely extend beyond the end of the measuring tool. If there is no measuring tool available, the end of the column should be measured to extend 8 mm beyond the internal transfer line tip opening.
20. Grasping the transfer line securely with a 3/16" wrench, use a 5/16" wrench to tighten the brass nut. Tighten the nut until snug, but do not over tighten.

 **CAUTION**

As you tighten the nut, the position of the column in the transfer line may change. If this happens, loosen the nut and readjust the column until the column extends the proper distance from the transfer line tip.

21. If in Internal mode, replace the measuring tip with the actual brown transfer line tip.



22. Clean the tip end of the transfer line with methanol and pull any service loop back into the GC oven.

23. Position the transfer line so that the heater cable aligns with the slot on the right side of the transfer line.
24. Remove the analyzer assembly during this step to avoid damaging the transfer line tip. Insert the transfer line into the manifold, and install the clip on the transfer line into the holes provided.
25. Gently push the transfer line toward the manifold, and rotate the collar in the clockwise direction until the bayonet lock engages.
26. Route the transfer line heater cable below the transfer line, through the white retainer and under the thermocouple vacuum gauge. Then plug the transfer line heating cable to connector J37.
27. Replace the 4000 MS top cover.
  - Gently push the mass spectrometer toward the GC, until the transfer line nut is visible inside the GC oven. Take caution not to damage rear pneumatics lines. The boot should fit snugly into the hole on the side of the GC oven.
  - Turn the GC oven on through its keyboard by pressing the Column Oven button and the blue soft key entitled Turn Oven On.
  - Go to “Turning On the Mass Spectrometer” on page 46.
28. After the trap, source, and manifold temperatures have reached their setpoints, condition the new column to prevent MS contamination.

---

## Replacing Consumable Components

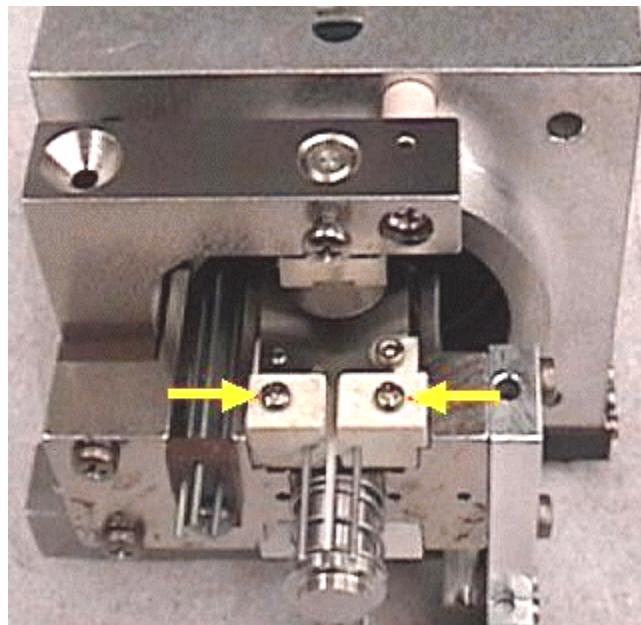
### Replacing External Source Filaments

- Go to “Turning Off the Mass Spectrometer” on page 34.
- Go to “Removing the Analyzer Assembly” on page 37.
- Go to “Removing the Source/Ion Trap Assembly” on page 40.



**WARNING:  
SHOCK HAZARD**

Dangerous voltages are present. Unplug power cord.



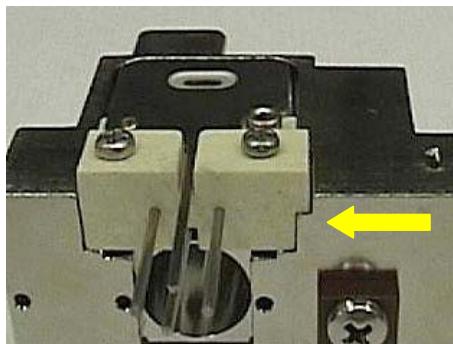
**CAUTION**

When removing the filament screws do not allow ceramic dust to fall into the ion trap. Use a flow of clean pressurized gas to blow off any dust observed.

***Removing Old Filament Assemblies***

1. Place the Source/Ion Trap Assembly on a lint-free surface with the filament screws in the horizontal position. Do not stand the ion trap on its end or ceramic dust may fall into the ion trap.
2. Remove the two Phillips screws.
3. Carefully lift the filament assembly out of the source holder.
4. Inspect the metal disc (03-931761-01) on the magnet for discoloration and carbon build up. If the disk looks dirty, slide the disc off and place a new one on the magnet.
5. Turn the assembly over and repeat the steps for the other filament and magnet disc.

## *Installing a New Filament Assembly*



1. Place the new filament into the source holder with the notched side down. Be sure the filament is seated firmly in place.
2. Place the two screws into the screw holes and tighten each screw evenly. Do not over tighten.
3. Turn the assembly over and repeat the steps for the other filament.



**New filaments undergo conditioning in the first few days of operation. It is recommended that filament tuning be checked daily during the first few days of full operation until the filaments remain solidly in tune.**

- Go to “Reinstalling the Source/Ion Trap Assembly” on page 41.
- Go to “Reinstalling the Analyzer Assembly” on page 44.
- Go to “Turning On the Mass Spectrometer” on page 46.

## **Conditioning the Filaments**

1. Run Auto Tune of electron lens voltage three times.
2. Run the method External\_Filament\_Conditioning.mth that applies multiple cycles of turning the filaments on/off.
3. Re-run the Auto Tune twice. Filaments are ready to go at this point.
4. Over the next four days, re-tune the lens voltages as required. Refer to the Ion Source indicators on the diagnostics page. If the Ion Source deviation number is larger than 2  $\mu$ Amps, the filament is probably off tune.

## **Replacing Internal Source Filaments**

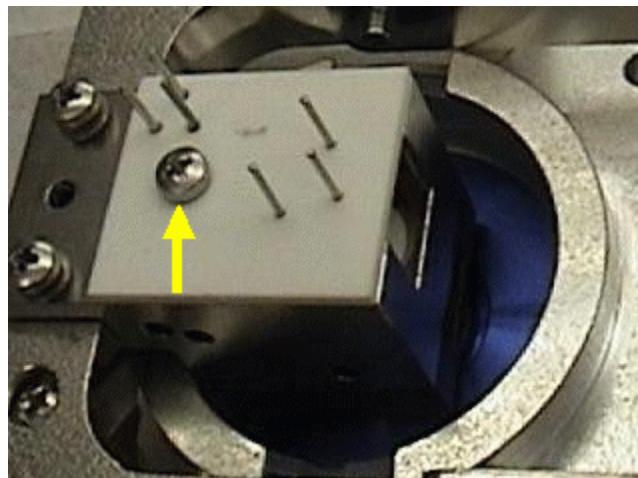
- Go to “Turning Off the Mass Spectrometer” on page 34.
- Go to “Removing the Analyzer Assembly” on page 37.
- Go to “Removing the Source/Ion Trap Assembly” on page 40.



## WARNING: SHOCK HAZARD

Dangerous voltages are present. Unplug power cord.

### *Removing Old Filament Assembly*



1. Remove the filament retention screw and place it on a lint-free cloth.



2. Lift off the ceramic plate and remove the filament assembly.

### *Installing the New Filament Assembly*

1. Transfer the springs to the new filament assembly.
2. Place the assembly in the filament block.
3. Reinstall the ceramic plate over the protruding pins.



4. Ensure that the filament assembly is seated flat in the filament block.
5. Reinstall the holding screw and tighten.
  - Go to “Reinstalling the Source/Ion Trap Assembly” on page 41.
  - Go to “Reinstalling the Analyzer Assembly” on page 44.
  - Go to “Turning On the Mass Spectrometer” on page 46.

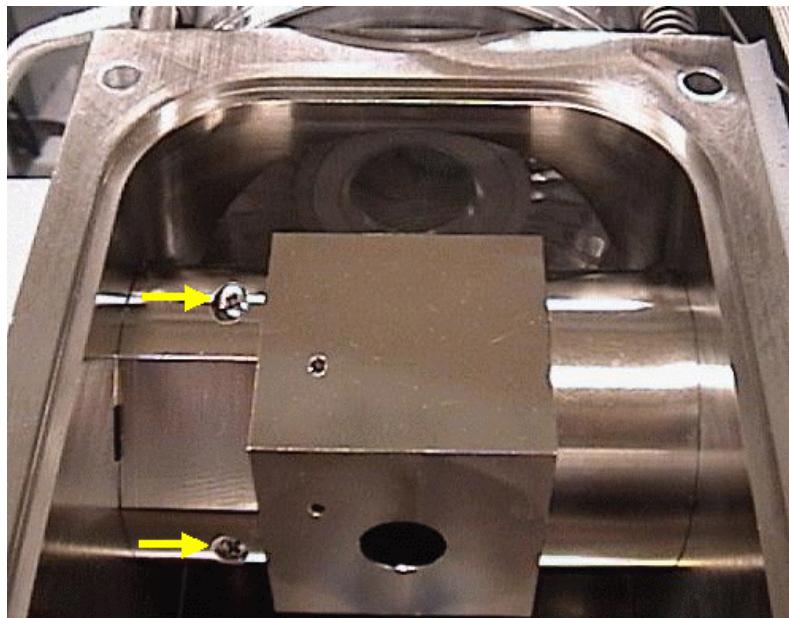
## Replacing the Electron Multiplier

- Go to “Turning Off the Mass Spectrometer” on page 34.
- Go to “Removing the Analyzer Assembly” on page 37.



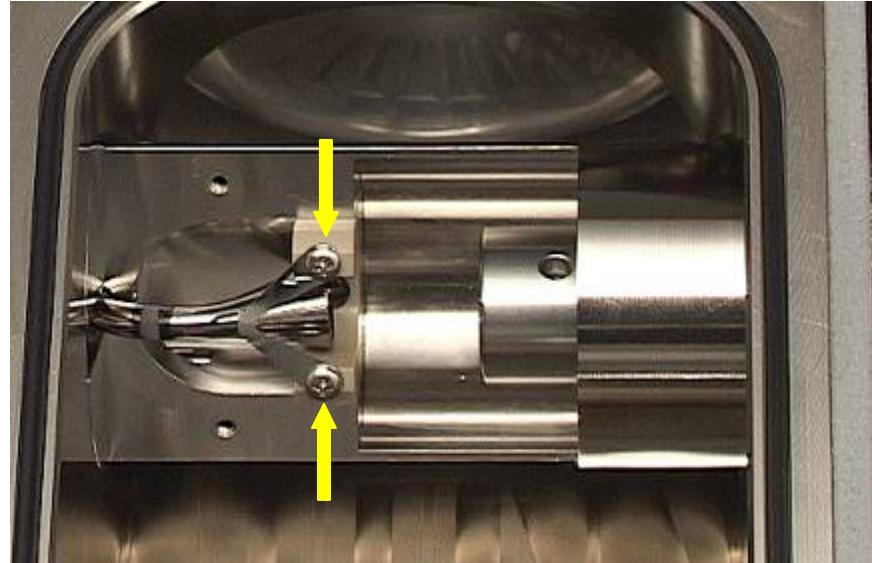
### WARNING: SHOCK HAZARD

Dangerous voltages are present. Unplug power cord.



### *Removing the Old Electron Multiplier*

1. Remove the two screws holding the multiplier cover in place.
2. Grasp the cubical part of the cover and lift straight out to remove the cover.
3. Place the cover on a lint-free cloth.



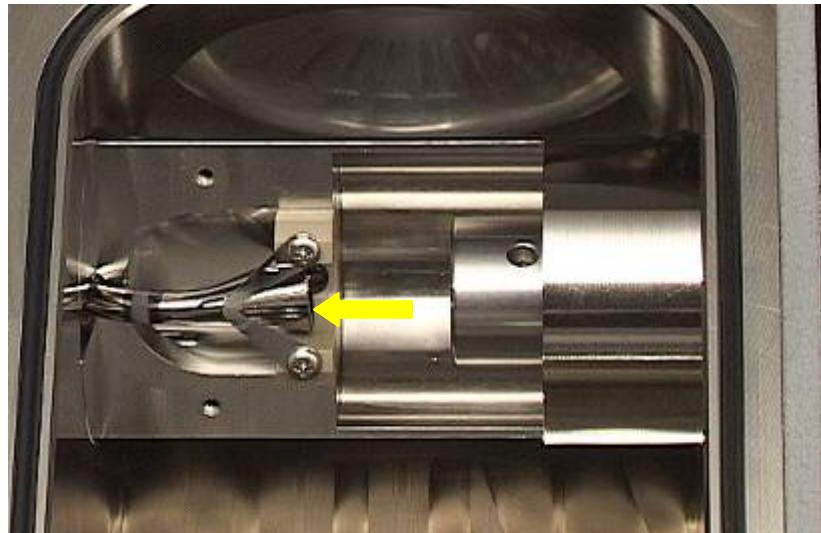
4. Loosen both multiplier retainer screws one turn.
5. The retainer bracket will swing down and out of the way, in the direction of the arrow, allowing the multiplier to be lifted out.



6. Lift out the multiplier

#### ***Installing the New Multiplier***

1. Place the multiplier into the holder as shown. The multiplier must be fully engaged by the clip at the bottom of the holder by pressing down and to the left as shown. Failure to properly engage the clip adversely affect performance. The horn should be near the centerline of the plastic holder.
2. Swing the holding bracket back into position and tighten the bottom screw.
3. Tighten the top screw.



4. Check the position of the multiplier so it is centered under the holding bracket. Be sure the notch in the multiplier cover is aligned with the throat of the multiplier. Visually check for any particles and remove if found. The cover is designed to have a tight fit and requires a small amount of force to push onto the mount.
5. Push the cover straight into place.
6. Reinstall the two screws and tighten.
  - Go to "Reinstalling the Analyzer Assembly" on page 44.
  - Go to "Turning On the Mass Spectrometer" on page 46.

## Replacing the Damping Gas Getter

The getter in the damping gas line removes water and contaminants from the damping gas helium supply. It has a limited life, the length of which is dependent on the amount of material to which it has been exposed.

To replace the getter, order a replacement kit (Part number 03-931124-91 Kit, Getter Replacement). Detailed instructions for replacing the getter are contained in this kit.

## Replacing the Turbomolecular Pump

To replace a Turbomolecular Pump, order a replacement kit (Part number 03-931119-91 Kit, Turbo Replacement, V301). Detailed instructions for replacing the pump are included in the kit.

### Tools Required

- Phillips Head Screwdriver
- L-Shaped 6 mm Allen wrench (provided in 4000 MS Ship Kit)
- Screen Pick (provided in 4000 MS Ship Kit)

1. Go to "Turning Off the Mass Spectrometer" on page 34.



## WARNING: SHOCK HAZARD

Dangerous high voltages are present. Unplug power cord.

2. Follow the replacement procedure included with the Turbo Replacement Kit.
3. Turn On the Mass Spectrometer (page 46) but DO NOT Start System control. The turbomolecular pump will go through a SoftStart conditioning process that will take about 30 minutes.
4. After 30 minutes start up System Control and go to the Startup/Shutdown page if this doesn't happen automatically.
5. Go to "Checking the Vacuum Status" on page 48.
6. Go to "Baking Out the Mass Spectrometer" on page 49.

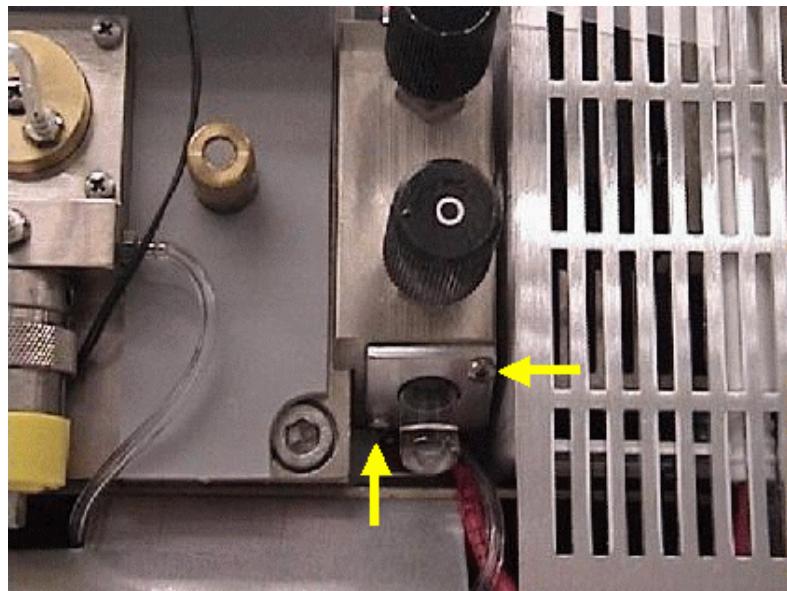
## Filling the Calibration Compound Vial

- The calibration compound used with the 4000 MS is perfluorotributylamine (PFTBA), which has the chemical formula C<sub>12</sub>F<sub>27</sub>N. This compound is also known as FC-43 (fluorocarbon-43).

---

NOTE: There is no need to vent the vacuum system before you fill the Cal Gas vial with calibration compound, provided the Cal Gas needle valve is closed. To close the Cal Gas needle valve, turn it fully clockwise.

---

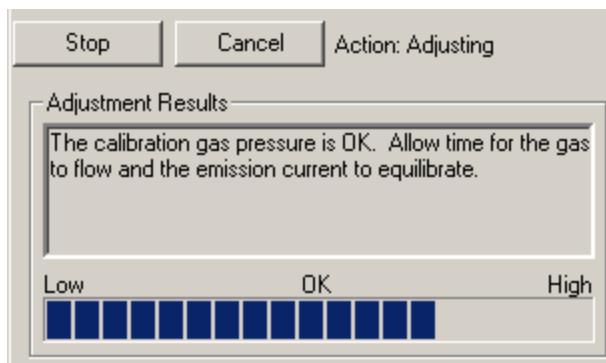


To fill the Cal Gas vial, proceed as follows:

1. Loosen the two retaining screws about 2-3 turns with a Phillips screwdriver.
2. Pull the Cal Gas vial down gently with a slight twisting motion until it clears the pneumatics manifold.
3. Refill the vial using a Pasteur pipette until the vial is filled just less than one half full with PFTBA compound (03-920353-00). Care must be taken not to overfill the vial to avoid inconsistent Cal Gas flow. Excess PFTBA can be

stored in the capped spare vial (03-931112-01) provided in the ship kit, or in a standard 2 mL autosampler vial.

4. While holding the vial vertically, carefully push the vial into the Cal Gas port on the manifold with a slight twisting motion.
5. After you have pushed the vial in as far as it will go, tighten the retaining screws.
6. Open the Cal Gas needle valve 10 turns counterclockwise. Leave the needle valve open for at least 30 minutes. Any excess Cal Gas and water vapor will be pumped away.
7. Under Manual Control's Checks and Adjustments tab, select Cal Gas Adjustment and press **Start**.
8. Adjust the Cal Gal pressure so that the indicator bar is near the center of the display, within the OK region.



9.

---

NOTE: Other adjustments that affect ion time, such as the multiplier gain, filament current and background levels will influence this adjustment.

---

## Changing Operational Configuration

Changing between ionization configurations requires swapping the source and/or placing the transfer line into its proper position for that mode. In this section, the major steps for each possible conversion are listed, followed by a series of general descriptions for handling the key steps in the conversion process.

The following steps are required for any system reconfiguration:

- Go to "Turning Off the Mass Spectrometer" on page 34.
- Go to "Removing the Analyzer Assembly" on page 37.

Perform appropriate change procedure described below.

- Go to "Reinstalling the Analyzer Assembly" on page 44.
- Go to "Turning On the Mass Spectrometer" on page 46.

## **Changing from Internal to External Configuration**

1. To switch sources from Internal to External, go to “Switching Between External and Internal Sources” on page 84.
2. To switch the transfer line position from entering the Ion Trap to entering the External source, go to “Changing the Transfer line Position from Internal to External” on page 87.

## **Changing from External to Internal Configuration**

1. To switch sources from External to Internal, go to “Switching Between External and Internal Sources” on page 84.
2. To switch the transfer line position from entering the External source to entering the Ion Trap, go to “Changing the Transfer line Position from External to Internal” on page 85.

## **Changing from Internal to Hybrid Configuration**

1. To switch sources from Internal to Hybrid, go to “Switching Between External and Internal Sources” on page 84.
2. Go to “Installing or Removing the Hybrid Plug” on page 89.

## **Changing from External to Hybrid Configuration**

1. To switch the transfer line position from entering the External Source to entering the Ion Trap, go to “Changing the Transfer line Position from External to Internal” on page 85.
2. Go to “Installing or Removing the Hybrid Plug” on page 89.

## **Changing from Hybrid Mode to External Configuration**

1. Go to “Installing or Removing the Hybrid Plug” on page 89.
2. To switch the transfer line position from entering the Ion Trap to entering the External Source, go to “Changing the Transfer line Position from Internal to External” on page 87.

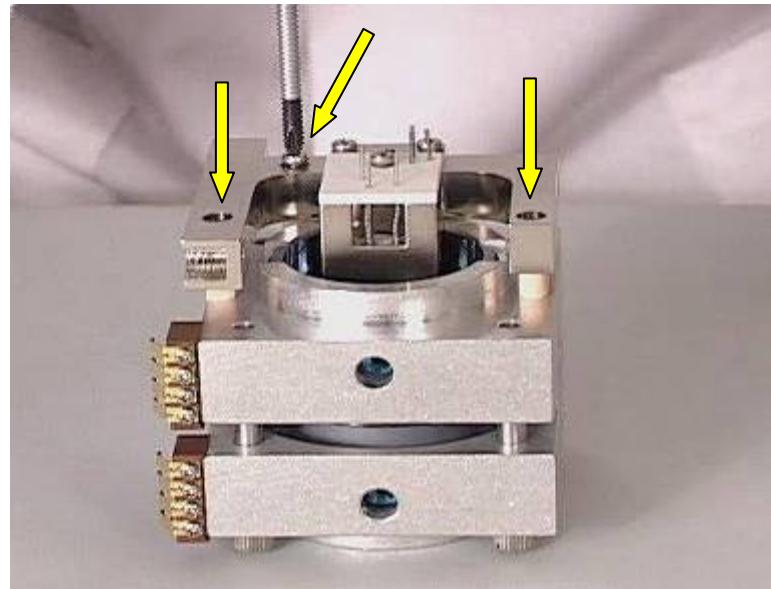
## **Changing from Hybrid Mode to Internal Configuration**

1. Go to “Installing or Removing the Hybrid Plug” on page 89.
2. To switch sources from External to Internal, go to “Switching Between External and Internal Sources” on page 84.

## **Switching Between External and Internal Sources**

1. To remove the Source/Ion Trap Assembly, go to page 40.

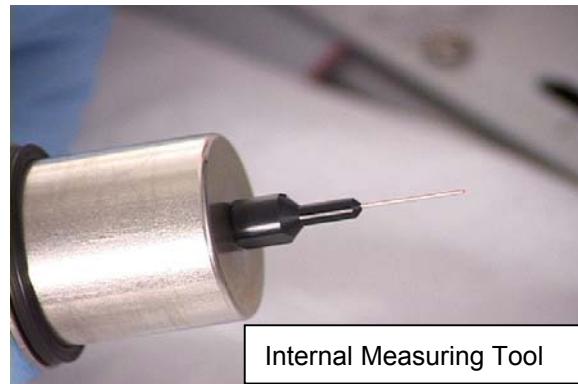
2. Swap sources by loosening the three screws on the magnet structure, pulling out existing source while leaving the ceramic spacers in place and placing the source on a lint-free surface.



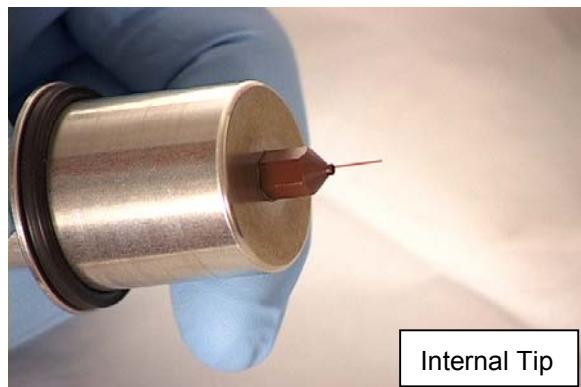
3. Take the screws from the source that was removed, and place them in the source being installed.
4. Position the replacement source with the three screws aligned into the ceramic spacers. If switching to external mode, be sure the centering ring is in place. Retighten the three screws. The source you removed should be stored in the box provided.
5. Go to “Reinstalling the Source/Ion Trap Assembly” on page 41.

## Changing the Transfer line Position from External to Internal

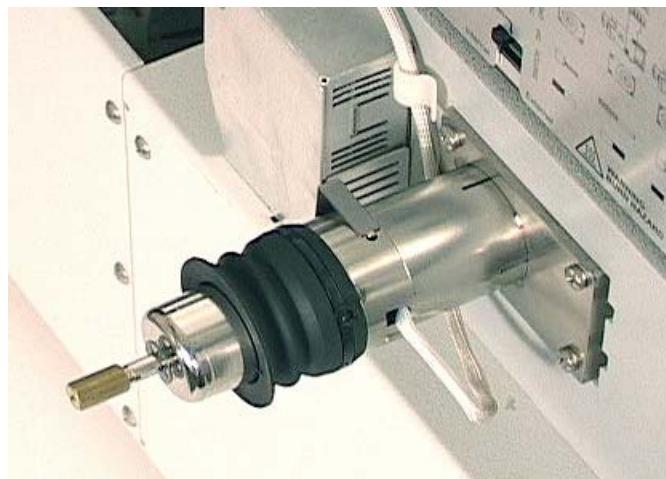
1. To move the 4000 MS away from the GC, go to page 37.
2. Unplug the transfer line heater cable from connector J37 on the bulkhead.
3. Be sure the transfer line is cool; then remove the transfer line assembly (Including the weldment) from the manifold by loosening the four captive screws holding it in place. Be sure not to lose the sealing O-ring.



4. Remove the external tip and replace it with the Internal measuring tool provided with the system. If you do not have an Internal measuring tool, you will need a ruler to measure the column length.
5. Using a sapphire-, or carbide-tipped scribing tool or ceramic scoring wafer, score the column once lightly at the end of the measuring tip and cleanly break the column. If there is no measuring tip available, cut the end of the column 8 mm beyond the internal transfer line tip opening after the internal tip is installed.
6. Remove the measuring tool.

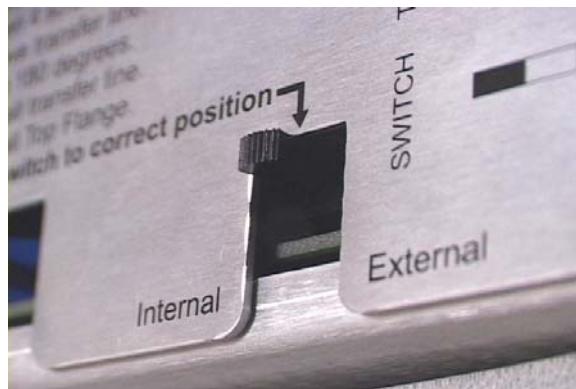


7. Screw the brown Polyimide internal tip onto the transfer line and clean the column and surrounding area with methanol and a lint-free wipe.
8. Return the transfer line assembly to the manifold, positioning it towards the rear of the instrument, and tighten the four screws. Be sure that the O-ring is clean and properly seated in the manifold groove (no kinks or twists).



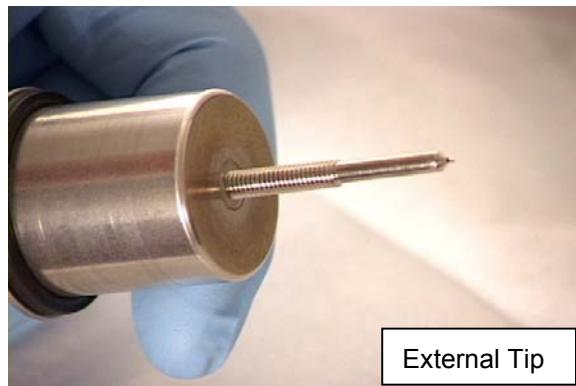
9. Route the transfer line heater cable through the white retainer clip on the side of the manifold and under the thermocouple gauge. Then plug it into J37 on the bulkhead.

10. Change the position of the ionization mode switch on the manifold electronics enclosure to the left (internal) position.



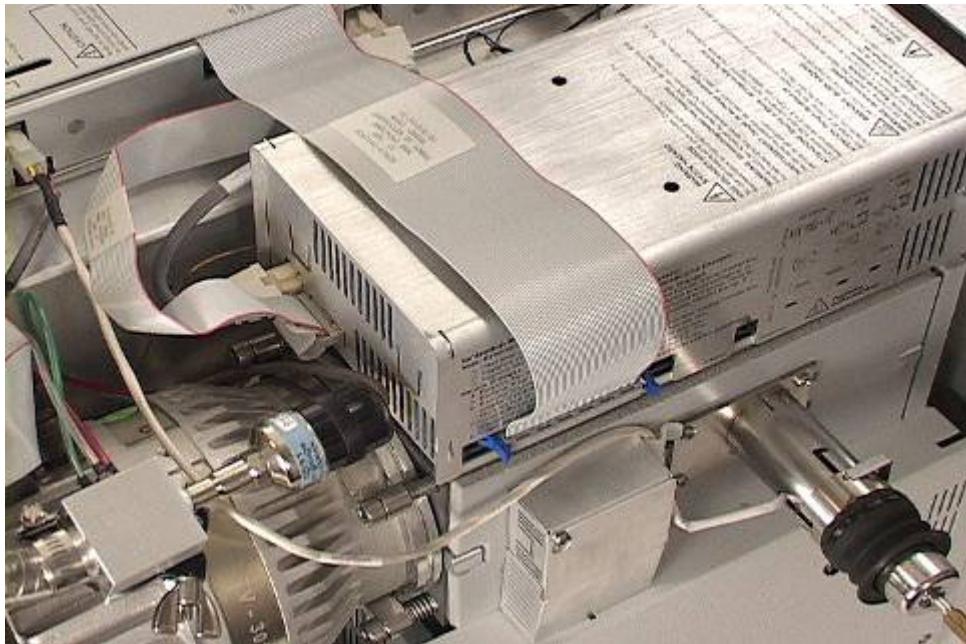
## Changing the Transfer line Position from Internal to External

1. To move the 4000 MS away from the GC, go to page 37.
2. Unplug the transfer line heater cable from connector J37 on the bulkhead.
3. Be sure the transfer line is cool; then remove the transfer line assembly (including the weldment) from the manifold by loosening the four captive screws holding it in place. Be sure not to lose the sealing O-ring.
4. Remove the internal tip and replace it with the long metal external transfer line tip provided with the external source. If necessary, a 3/16" wrench can be used to stabilize the transfer line and a 5/16" wrench used to remove the tip.

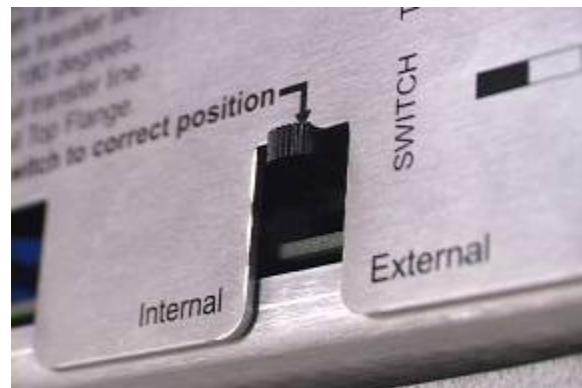


5. Loosen the brass nut at the GC side of the transfer line and then reposition the column until it extends 1 mm from the end of the tip. If the column won't move, it may be necessary to cut off the column before the transfer line, remove the ferrule from the brass nut, and reinsert the column using a new ferrule (as described in the column replacement procedure).
6. Replace the transfer line assembly, positioning it towards the front of the instrument, and tighten the four screws. Be sure that the O-ring is clean and properly seated in the manifold groove (that there are no kinks or twists).

7. Route the transfer line heater cable through the white retainer clip on the side of the manifold and under the foreline line. Plug the cable into J37 on the bulkhead.

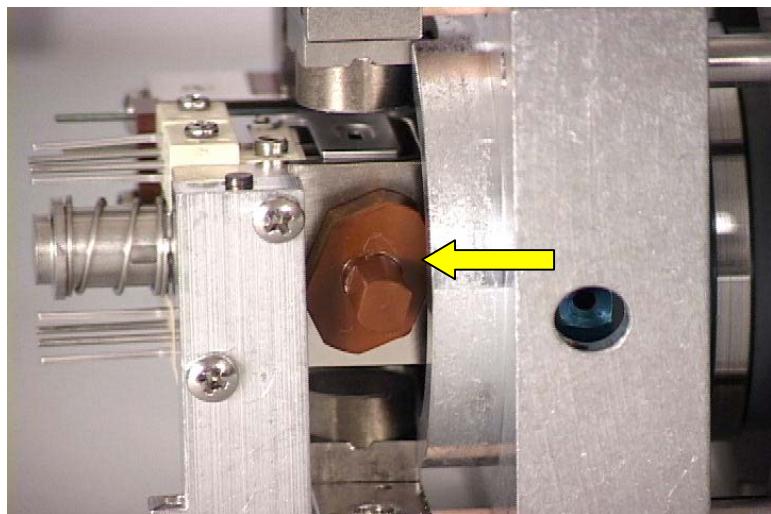


8. Change the position of the ionization mode switch on the manifold electronics enclosure to the right (External) position.



## Installing or Removing the Hybrid Plug

Operation in hybrid configuration requires a plug that prevents reagent gas from escaping the high pressure CI source through the unused transfer line hole in the CI Volume. The supplied plug is installed in the transfer line hole in the external source by inserting the plug and turning until the plug engages with the side of the source heater block. The plug is removed in the reverse fashion by turning the plug until it disengages.





# Chemical Ionization Options

---

## Introduction

Chemical ionization (CI) provides mass spectral data that complement electron ionization (EI) data for chemical analysis. In the 4000 MS, there are three optional modes of CI operation depending upon the instrument configuration – Internal Configuration positive CI (PCI), External Configuration positive or negative CI (PCI/NCI) or Hybrid Configuration positive or negative CI (PCI/NCI).

---

**NOTE:** The CI mode is an option on the 4000 MS. If your system does not have this option, you will not be able to perform CI analyses.

---

### Internal Configuration CI

When the 4000 MS is in Internal Configuration, the CI reagent gas (from an external gas cylinder) enters the analyzer through a length of a 4 mL/min restrictor tubing. The reagent gas is ionized by EI to form reagent ions. These reagent ions then ionize sample molecules entering the analyzer with He carrier gas from the capillary column. The operation and adjustment of reagent gases for the Internal Configuration CI option are described in the first part of this section. Internal CI is possible only in PCI mode.

An additional Liquid CI Inlet (or LCI Inlet) option allows the selection of certain liquids as sources for CI. A 50 mL/min restrictor is used for admitting reagent through the Liquid CI Inlet when one is in Internal Configuration. The operation of this option and switching between Liquid and Gaseous CI is described later in this section.

### External Configuration CI

When the 4000 MS is in External Configuration, the CI reagent gas (from an external gas cylinder) enters the external ion source through a length of 4 mL/min restrictor tubing. A special CI volume is automatically inserted into the EI volume (under software control) to create a high-pressure environment that enhances CI reactions. The reagent gas is ionized by EI to form reagent ions. These reagent ions react immediately with sample molecules entering the external ion source. Both positive and negative ions may be formed in these processes and the ions carried into the ion trap for analysis depend upon whether the user has specified to perform positive or negative CI in the 4000 MS Method section. The use of liquid CI reagents is not recommended for External Configuration CI because the pressure of relatively nonvolatile liquid reagents is too low for efficient CI processes to occur in external PCI or NCI modes.

## Hybrid Configuration CI

When the 4000 MS is in Hybrid Configuration, the CI reagent gas (from an external gas cylinder) enters the external ion source through a length of restrictor tubing. In standard Hybrid High Pressure Source (HPS) Configuration, a high pressure CI volume is automatically inserted into the EI volume to create a high-pressure environment to enhance CI reactions. The reagent gas is ionized by EI to form reagent ions. Both positive and negative ions may be formed in these processes and reagent ions of either positive or negative charge are transferred immediately to the ion trap. The polarity of ions carried into the ion trap for analysis depend upon whether the user has specified to perform positive or negative CI in the 4000 MS Method section.

Once reagent ions have been stored in the ion trap for the designated ion time, waveforms are applied to isolate only the reagent ions within a mass range designated in the 4000 MS Method. Finally, the chosen reagent ions react with neutral analytes entering the ion trap through the GC column.

An additional Liquid CI Inlet (or LCI Inlet) option allows the selection of certain liquids as sources for CI. A 200 mL/min restrictor is used for admitting reagent through the Liquid CI Inlet when one is in Hybrid Configuration. The operation of this option and switching between Liquid and Gaseous CI is described later in this section.

---

## Installing CI Reagent Gas

We recommend that the inlet gas line be as short as possible. Ideally you should secure the gas cylinder close to the rear of the 4000 MS so that the 4 mL/min restrictor tube can be attached by a 1/8" Swagelok fitting directly to the two-stage gas regulator and the other end of the restrictor attached through the CI Gas inlet into the MS. Make sure, however, that the gas line is long enough to run to the rear of the 4000 MS and to accommodate the movement of the mass spectrometer 9 inches (23 cm) to the right (for access to the transfer line and turbomolecular pump).

Gas cylinders or lecture bottles should not be stored where they can damage cables or gas lines, and they should be secured in accordance with standard safety practices. Lecture bottles have rounded ends and will require some means of support (e.g., Matheson Model 505 Non-Tip Stand).

Before installing the CI reagent gas supply, you should complete the following procedures:

- Tune the instrument in EI mode
- Check the 4000 MS system for leaks

## CI Reagent Gas Requirements

These paragraphs give the requirements for the reagent gases used for CI operation with the 4000 MS. The following reagent gases are recommended: methane and isobutane.

Use high-purity reagent gas for maximum sensitivity and good spectral quality. Impurities can react with sample ions, creating confusing mass spectral data.

The amount of reagent gas consumed during CI operation is very low (typically 1 to 2 mL/minute). Depending upon how much CI you plan to do, choose the size of the gas cylinder appropriately.

The requirements for the recommended gases are as follows:

Methane	Methane should have a purity of 99.99% or better. Use a gas cylinder with a two-stage pressure regulator that has a stainless steel diaphragm and maximum inlet pressure of 30 psi (200 kPa).
Isobutane	Isobutane should have a purity of 99.99% or better. Use a gas cylinder with a two-stage pressure regulator that has a stainless steel diaphragm and maximum inlet pressure of 30 psi (200 kPa).

The CI reagent gas should contain less than 1 ppm of water. Water in the CI reagent gas may interfere with CI operation.

If you need to use a longer line than the 4 mL/min restrictor alone, use pre-cleaned copper or stainless steel gas lines for methane or isobutane. All gas lines should be free of oil (and other contaminants) and preferably flame dried. If possible, use the pre-cleaned copper tubing from the GC Start-Up Kit.



### WARNING: CHEMICAL HAZARD

DO NOT flame! Dry the reagent gas lines with CI reagent gas present.

## Setting Up the CI Reagent Gas Supply

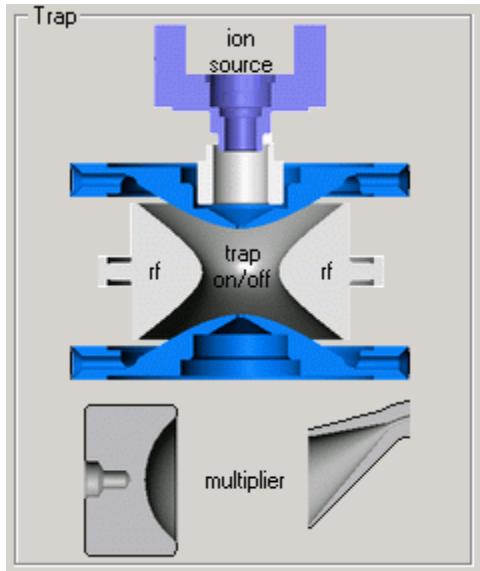
Use the following procedure to set up the CI reagent gas supply.



### WARNING: CHEMICAL HAZARD

CI reagent gases may be hazardous. Use proper protection when installing the reagent gas.

1. Enter System Control and select the Manual Control tab dialog.



2. Make sure that the electron multiplier, filament, and RF voltage are all off. The Multiplier, Filament (Ion Source), and RF text should be red or black - not green.

---

NOTE: Two solenoid-operated valves control the flow of CI reagent gas into the manifold. The valves are opened and closed by clicking on the CI button on the Instrument Control display. A needle valve controls the amount of reagent gas flowing into the manifold. The needle valve is mounted directly behind the door of the mass spectrometer. The needle valve is adjusted manually by using the knob labeled CI GAS. Turning the knob clockwise increases the flow of reagent gas into the manifold. See Functional Block Diagrams in the Pneumatics section.

---

3. Check that the CI Gas solenoid valves are closed. When these valves are closed, the CI Gas icon to the left of the ion trap symbol is not green. (If the CI icon is green, click on the icon so that it turns to black.)
4. Install a two-stage pressure regulator on the reagent gas cylinder or lecture bottle. Tighten the connection securely.

---

NOTE: A two-stage pressure regulator typically consists of the following components: Secondary valve, pressure adjustment valve, supply pressure gauge, and delivery pressure gauge

---

Reagent gas is turned on and off with the main valve on the cylinder or lecture bottle. The secondary valve on the pressure regulator is next in line. This valve is used for coarse control of the flow of gas from the gas cylinder up to the pressure adjustment valve. The supply pressure gauge is used to monitor the gas pressure in the bottle. The pressure adjustment valve is used to set the head pressure of the gas delivered to the mass spectrometer.

5. Connect one end of the 1/8" OD gas supply line to the pressure regulator.



6. On the back of the instrument, loosen the two screws that hold the plug in the CI Shutoff Manifold 2 to 3 turns. Remove the plug by pulling straight out and twisting.
7. Use the 4 mL/min restrictor tube for the supply line between the gas cylinder and the CI shutoff manifold. No ferrule is required on the mass spectrometer end of this tube. The seal is made with an elastomer O-ring.
8. Carefully insert the restrictor tube into the CI shutoff manifold hole (the one the plug came out of) until it is firmly seated. Tighten the two screws.
9. Ensure that the secondary valve on the regulator on the gas cylinder is closed.
10. Open the main control valve on the lecture bottle. Next, open the secondary valve and adjust the pressure valve to approximately 20 psi.
11. Open the mass spectrometer door. Verify that the CI GAS needle valve is turned fully counterclockwise.
12. Next, flush the gas line of air and water vapor by doing the following.
  - Monitor the foreline pressure on the diagnostics screen. Do not allow the foreline pressure to exceed 500 mTorr for more than 20 seconds.
  - Turn the adjustment valve clockwise to reduce the pressure.
  - Open the CI Gas solenoid valves by clicking on the CI icon in the Control and Status field of the Manual Control tab dialog in System Control. When the valves are opened, the CI button is green.
  - Evacuate the CI reagent supply line for about 30 minutes.

## Checking the Reagent Gas Plumbing for Leaks

To check for air leaks in the reagent gas line connections and the presence of water vapor in the gas line, follow the procedure using a leak detection gas to troubleshoot for air leaks in the Troubleshooting section. Depending upon the results you obtain, you may need to modify the procedure as follows:

If a large air leak exists, check the CI GAS fitting on the rear of the instrument and the fitting on the pressure regulator for tightness. Then recheck the air/water spectrum; or

If excess water vapor is indicated by a high 19/18 ratio, there may be water in the gas line and/or an atmospheric air leak in the reagent gas plumbing. Proceed as follows:

1. Shut off the flow of reagent gas into the manifold by closing the CI solenoid valves. If necessary, click on the CI icon in the Control and Status field of the Manual Control tab dialog in System Control. When the valves are closed, the CI button is black or red - not green.
2. Recheck the air/water spectrum. If the peak at mass 19 (for water) decreases, then water is present in the gas line. In this case, go to step 3. If the peak at mass 19 does not decrease significantly, little water is present in the gas line. In this case, the MS system probably has an air leak. You will need to fix the leak as described in the Troubleshooting Section. Be sure to check for leaks around:
  - The CI GAS port on the rear of the mass spectrometer
  - The fitting that connects the reagent gas line to the pressure regulator
3. To flush excess water from the gas line proceed as follows:
  - a. Ensure that the electron multiplier, filament, and RF voltage are off.
  - b. Open the main valve on the lecture bottle. (The secondary valve on the pressure regulator is already open.)
  - c. Turn the CI needle valve fully counterclockwise to divert gas to foreline pump.
  - d. Open the CI Gas solenoid valves and allow the system to pump down for about 1 hour.
  - e. Close the main valve on the CI Gas cylinder but keep the CI GAS solenoid valves open. Allow the system to pump down for about 15 minutes.
  - f. Recheck the air/water spectrum. If excess water is not present, go to paragraph: Setting Delivery Pressure of the CI Reagent Gas.

## Setting Flows of CI Reagents in Internal Configuration

After any leaks have been located and fixed, set the delivery pressure of the CI reagent by doing the following:

1. Ensure that the CI Gas solenoid valves are closed. If necessary, click on the CI icon in the Control and Status field of the Manual Control tab dialog in System Control. When the valves are closed, the CI button is black or red - not green.
2. Open the main valve on the lecture bottle. Using the pressure adjustment valve on the regulator, set the head pressure to about 20 psi.

You are now ready to operate the system in the CI mode. Read the respective users guides for more detailed operational information.

### Internal Mode Default Parameters for CI Reagents

Reagent Gas	Methane	Isobutane	Acetonitrile	d3-Acetonitrile	Methanol
Reagent Low Mass (m/z)	15	35	35	35	25
Reagent High Mass (m/z)	45	65	60	60	50
Reaction Storage Level (m/z)	35	35	33	33	25
Ejection Amplitude (v)	15	15	15	15	15
Target TIC	5000	5000	5000	5000	5000
Maximum Ionization Time (μsec)	2500	2500	2500	2500	2500
Maximum Reaction Time (μsec)	100	100	100	20*	100

\* Use short reaction times for deuterated reagents. Longer reaction times allow more H/D exchange with background water and the resulting spectrum will show more  $[M+H]^+$  and less  $[M+D]^+$ .

### External Mode Default Parameters for CI Reagents

Reagent Gas	Methane	Isobutane	Ammonia	Acetonitrile	d3-Acetonitrile	Methanol
CI Background (m/z)	45	65	35	60	60	50
Target TIC	5000	5000	5000	5000	5000	5000
Maximum Ionization Time (μsec)	2500	2500	2500	2500	2500	2500
Maximum Reaction Time (μsec)	100	100	100	100	20*	100

## Ion Intensities for Standard CI Reagents

The CI Adjust function gives recommendations of an acceptable level of CI reagent ions for each of the five standard CI reagents. The general principles used in implementing these tests are:

Methane	Adjust the reagent gas pressure so that the peak height at m/z 17 ( $\text{CH}_5^+$ ) is about 25% of that at m/z 29 ( $\text{C}_2\text{H}_5^+$ ). The ratio of the ions at m/z 17 to m/z 16 should be about 10:1. The ion at m/z 41 ( $\text{C}_3\text{H}_5^+$ ) should be visible.
Isobutane	Adjust the reagent gas pressure so that the peak heights at m/z 57 [ $(\text{CH}_3)_3\text{C}^+$ ] and m/z 43 [ $(\text{CH}_3)_2\text{CH}^+$ ] are about equal. There may also be an intense reagent ion at m/z 41 ( $\text{C}_3\text{H}_5^+$ ).
Acetonitrile	Adjust the reagent gas pressure so that the ion at m/z 42 [ $\text{CH}_3\text{CNH}^+$ ] is more than 5 times higher than at m/z 41. The valley between the 41/42 ions should reach a minimum at less than half the height of the m/z 41 ion. The m/z 54 ion [ $\text{CH}_3\text{CHCNH}^+$ ] will be present at 10 - 15% the height of m/z 42. Too much acetonitrile in the trap can cause early filament failures.
d3-Acetonitrile	Adjust the reagent gas pressure so that the ion at m/z 46 [ $\text{CD}_3\text{CND}^+$ ] is more than 5 times higher than at m/z 44. The m/z 58 ion [ $\text{CD}_3\text{CDCND}^+$ ] will be present at 10 - 15% the height of m/z 46.
Methanol	The ion at m/z 33 [ $(\text{CH}_3\text{OH})\text{H}^+$ ] will dominate the spectrum. No ion is observed at m/z 32, but a small peak is observed at m/z 31 and m/z 47.

In each case, by following these guidelines, the reagent gas pressure in the ion trap will be approximately  $1$  to  $2 \times 10^{-5}$  Torr (about  $1.3$  to  $2.6 \times 10^{-3}$  Pa). The CI reagent molecules comprise about 1% of the gas pressure in the ion trap. He atoms from column flow are present at 100 times this pressure.

## Setting Flows of CI Reagents in External Configuration

In External Configuration CI reagent flow is set using the ion gauge pressure measured in Manual Control.

1. Open the main valve of the methane (or isobutane) CI Gas cylinder and set the second—stage regulator pressure to 20 psi.
2. Open System Control. Turn on the CI and Ion Gauge using the check boxes beneath the 4000 MS icon. If the CI line has not been evacuated already, allow a few minutes for this process.
3. Adjust the CI valve so that the ion gauge reading is between 50 – 80  $\mu$ Torr.

## Setting Flows of CI Reagents in Hybrid Configuration

See the 4000 MS Operations Manual for advice on setting flows for CI reagents.

---

## The Liquid CI Inlet Option

Liquid CI is an effective tool for internal ionization CI. Because of the difficulty of getting sufficient CI reagent into the external source, Liquid CI is not recommended for external CI use. Once the Liquid CI inlet Assembly has been installed, it is possible to switch between using a pressurized CI Gas and using liquid CI reagents, without removing the assembly. Switching from Gaseous to Liquid CI Reagent Operation:

1. Loosen the 2 screws that attach the CI Gas restrictor to the CI shutoff block in the back of the instrument. If there is no Liquid CI restrictor attached to the Liquid CI Inlet Assembly, also loosen the two screws that attach the L bracket to the assembly.
2. Remove the 4 mL/min gas restrictor from the CI shutoff block.
3. Install the Liquid CI restrictor between the Liquid CI Inlet Assembly and the CI shutoff block. If you are in Internal Configuration, use the 50 mL/min restrictor (03-930024-01). For Hybrid Configuration, use the 200 mL/min restrictor (03-931440-01).
4. Tighten all screws.
5. Adjust CI reagent as described in the User Manual.

### Filling/Refilling Reservoir Bulb

1. Be sure the CI valves are closed. Loosen the four screws that retain the liquid CI reservoir cover. The screws may remain in the block.
2. Remove the reservoir cover.
3. Gently pull the bulb down to remove it from the block. The O-ring and O-ring retainer may stay attached to the bulb.
4. Use the reservoir cover as a stand for filling; place the bulb into the reservoir cover. Place O-ring retainer over the bulb stem. Place the O-ring over the bulb stem.
5. Use a 1 mL syringe to fill the bulb halfway with liquid CI reagent. This requires about 3 mL of reagent.
6. Pick up the reservoir cover with the bulb, retainer and O-ring, and insert the bulb stem into the block.
7. Orient the cover so that the four screws can engage the cover. Tighten the four screws, being careful not to strip the threads in the plastic cover.
8. After installing liquid CI, and each time the reservoir bulb is refilled with liquid, always use care when first opening the CI valves. Do not turn on the filament or multiplier for 2-3 minutes after opening the CI valves from the Manual Control Page.

## **Switching from Liquid to Gaseous CI Reagent Operation**

To switch from the Liquid CI Inlet back to a pressurized CI Gas (such as methane), the CI Gas line may be Reinstalled without removing the liquid CI inlet assembly.

1. Loosen the 2 screws that attach the liquid CI inlet restrictor to the back of the instrument. Also, loosen the 2 screws that attach the L-bracket to the liquid CI inlet block.
2. Remove the liquid CI restrictor end that inserts into the back of the instrument; rotate the restrictor out of the way.
3. Install the 4 mL/min CI Gas restrictor (03-930597-01) between the gas supply and the CI shutoff block, below the L-bracket.
4. Tighten all screws.
5. Adjust CI reagent as described in the User Manual.



### **WARNING**

If the equipment is used in a manner not specified in this manual, the protection provided by the equipment may be impaired.

# Troubleshooting

---

## How to Isolate a GC/MS Problem

In general, whenever you attempt to isolate a 4000 MS problem, you will check the system in the following order:

- Data System
- Gas Chromatograph
- Mass Spectrometer

---

## Checking the Data System

Please refer to the 4000 MS software release notes for relevant software troubleshooting procedures.

---

## Checking the GC

The simplest and most effective way of isolating a GC problem is to run a test sample. Running a sample will allow you to check several operational and performance factors, including the carrier gas supply, chromatographic characteristics, and sample-related problems.

The test sample that is most frequently run is the COLTEST mixture. This multiple component mixture is very well suited to troubleshooting injector and column problems. Please see "Running the COLTEST Sample" on page 109 for a description on the use of this test mixture.

To identify the source of a GC electronics problem, press the STATUS key and a CONTROL key, (i.e., injector, column oven, etc.), to determine if a fault is present. If a fault is present, the message FAULTED appears. Consult the 3800 GC manuals for information about fixing GC faults. Make sure that you are thoroughly familiar with all safety issues before you attempt to repair any electronics component.

---

## Checking the Mass Spectrometer

If your data system and GC are operating normally, the problem could be caused by the mass spectrometer or by the communication channel between it and the data system. Typical problems with the ion trap include lack of response (no spectra), low response, poor resolution, and mass mis-assignment.

The MS Workstation includes diagnostics tests for isolating problems associated with the mass spectrometer. These tests may be used to isolate simple ion trap problems, e.g., air leaks, burned-out filaments, electronic failures, etc.

A 4000 MS Service directory is included in the MS Workstation (C:\VarianWS\4000 MS Service). There are service methods in this directory for internal (4000 MS Int Service.mth) and external (4000 MS Ext Service.mth) modes. These service methods are designed to be used in Manual Control to identify common spectrometry issues such as elevated air/water and hydrocarbon background levels, mass assignment, and resolution.

In certain cases, you may need to separate physically the GC and MS to isolate an ion-trap problem. In these cases, remove the column from the injector, and plug its end with a septum. This will minimize the input of air. Maintain the column and transfer line at ambient temperature to prevent degradation of the stationary phase. You do not need to vent the MS vacuum system to complete this procedure.

If you wish to isolate the mass spectrometer further, you must remove the column from the ion trap by shutting down the system and capping the transfer line with a no-hole ferrule.

---

## Troubleshooting Problems with Spectra

The following describes the common problems a user may encounter with an ion trap mass spectrometer.

### No Spectrum Appears

If a spectrum fails to appear on the screen when you click on the ion trap icon in the Manual Control Page, regardless of mass range, you should investigate the following potential causes:

- If the method segment is a FIL/MUL Delay segment, ionization is turned off. When a segment is set up with the ionization off the trap icon is red.
- The “filament is open (broken).”
- The turbomolecular pump has stopped.
- An RF adjustment is required.
- The instrument parameters are inappropriate.
- The trap has been incorrectly assembled.
- There is a problem with the electronics.
- The system has not finished baking out.

Before you begin troubleshooting, however, be sure that you have baked out the 4000 MS for at least 2 hours. Run Diagnostics to determine if any hardware problems are present. If you have done this, and the missing-spectrum problem persists, continue as follows. These steps apply if either air/water or Cal Gas peaks are missing.

#### ***Check for an Open Filament***

Diagnostics will determine if one or both filaments are open.

- If necessary, replace the filaments.

### ***Check the Turbomolecular Pump***

Diagnostics will report the turbomolecular pump speed.

Make sure the pump speed reading is  $100 \pm 2\%$ .

- If it is not, inspect cooling fans for proper operation.

### ***Check the RF Adjustment***

Check whether an RF adjustment is needed (particularly after you have changed the ion trap temperature).

### ***Check the Parameter Settings***

Check whether you have set inappropriate method parameters.

- Make sure that the ionization storage level permits storage in the trap of the ions selected in the scan range.

If the spectrum returns, note which parameter(s) were causing the problem. If no spectrum is present, and the trap was recently disassembled, the assembly of the trap must be checked.

### ***Check Ion Trap Assembly***

1. Check whether you have incorrectly assembled the trap components.
2. Check whether there is a problem with the electron multiplier.

## **Loss of High Mass Peaks**

The loss of high mass peaks may be due to:

- RF ramp needs adjustment
  - Too many low mass ions (for example, air or water leak)
  - Improper method parameters
  - High trap temperatures may cause loss of high mass Cal Gas peaks
1. Check for an air leak.
  2. Check RF ramp adjustment.
  3. Reduce trap temperature to  $150^{\circ}\text{C}$ .
  4. Enter Method Builder, check method parameters.

## **Part of the Spectrum is Missing**

If you do not observe high- or low-mass ions in manual control but the ions in the mid-range of the spectrum appear normal, you should investigate the following possibilities:

- An RF adjustment may be required, particularly if you have just changed the ion trap temperature.
- The ionization RF storage level may be incompatible with the scan range.

- The trap temperature may be too high to allow you to observe all of the Cal Gas ions. Reduce trap oven temperature to 150 °C, and wait for thermal equilibrium.

### ***Check the RF Adjustment***

Check whether an RF ramp adjustment is needed.

### ***Check the RF Storage Level***

Check whether the RF storage level is incompatible with the scan range.

### ***Check the Trap Temperature***

Check whether the trap temperature is too high to permit you to observe all Cal Gas ions.

If the trap temperature is too high, the height of the mass 614 peak may be reduced, and the mass 502 peak may disappear entirely (above 200 °C). Reduce trap oven temperature to 150 °C and wait for thermal equilibrium.

## **Poor Resolution with Acceptable Air and Water Levels**

If the peaks are broader than you would have expected, you should investigate the following possible causes:

- There are too many ions in the trap (i.e., contamination or high column bleed).
- The supplemental waveform value is too high or too low.
- Supplemental waveforms are not functioning properly.

### ***Check the Ion Content of the Trap***

With the trap turned on, note the TIC (total ion current) value. If the TIC value exceeds 20,000 counts in full-scan mode, reduce the number of stored ions.

### ***Run Auto Tune***

If problems with the supplemental waveforms are suspected, run the Auto Tuners to reset these values.

## **Troubleshooting High Baseline at High Masses**

If the baseline on the Manual Control page increases sharply between masses 400 and 1000, there may be particles on the electrode surface.

### ***Check for Particles in the Trap***

In Manual Control, activate C:\VarianWS4000 MS Service\4000 MS (Int or Ext) Service.mth, go to segment 2, and turn the Trap on and the Ion Source off. If the trap is free of particles there will be no significant spiking above the baseline and the base amount will be less than 10. If spiking or a base amount greater than 10 is observed, the system should be shut down and the trap cavity and manifold area should be blown free of particle matter using a compressed inert gas.

# Checking for Leaks

A common issue in mass spectrometry is keeping the system as leak-tight as possible. Air leaks may result in reduced sensitivity, tuning problems, and decreased resolution; in addition, they may reduce the lifetimes of the capillary column, filaments, turbomolecular pump, and the electron multiplier. Check the system each day for air and water leaks before you begin running acquisitions.

## Establishing Conditions Required for Leak Checks

To establish the conditions required to check for leaks, proceed as follows:

1. Activate C:\VarianWS\4000 MS Service\4000 MS (Int or Ext) Service.mth, go to segment 1 and turn the trap on.
2. Verify the column flow rate is 1.0 mL/min.
3. Set the GCMS temperatures:
  - Trap temperature to 150 °C.
  - Transfer line temperature to 270 °C.
  - Manifold temperature to 35 °C.
  - Source temperature to 150 °C.
4. Set the column-oven and injector temperatures to 100 and 230 °C respectively.



### CAUTION

Often, major air leaks are accompanied by a hissing sound. These leaks may be due to extremely loose fittings, improperly seated O-rings, or open valves. If you suspect a major leak, do not turn on the electron multiplier, RF voltage, or filament. Using the Diagnostics section, confirm that the turbomolecular pump is operating at 100% speed. If it is not, there may be a major air leak.

- If the ratio of the height of the peak of mass 18 ( $\text{H}_2\text{O}^+$ ) to mass 19 ( $\text{H}_3\text{O}^+$ ) is about 10:1, there is little water vapor in your system.
- If the ratio of peak height of mass 18 to mass 19 is less than 10:1 but greater than 5:1, additional bakeout may be necessary.
- If the ratio of the peak height of mass 18 to mass 19 is much less than 10:1, your system contains excess water vapor.

An Air/Water Spectrum Obtained from a System with No Significant Air Leaks and Little Water Vapor is indicated by:

- The peak at mass 18 ( $\text{H}_2\text{O}^+$ ) may be the base (highest) peak. This is dependent on the level of water vapor.
- The ratio of the peak height at mass 18 ( $\text{H}_2\text{O}^+$ ) to that at mass 19 ( $\text{H}_3\text{O}^+$ ) is greater than or equal to 10:1.
- The base amount value is significantly less than 500.
- The ratio of the peak height at mass 28 to that at mass 32 is about 4:1.

- If there are no air or water leaks in your system, you should obtain the following approximate values. Actual values may vary from system to system.

Base Amount	TIC	18:28 ratio	19:18 ratio	28 width
<500	<5000	≤ 1:1	10 to 15%	< 1 m/z

An air/water spectrum obtained from a system with a small air leak and little water vapor is indicated by:

- The peak height at mass 28 is noticeably greater than that at mass 18.
- The base amount value has increased to greater than 500.
- The ratio of the peak height at mass 18 to that at mass 19 is greater than or equal to 10:1.

An air/water spectrum obtained from a system with a moderate air leak and little water vapor is indicated by:

- The peak at 28 starts to overload.
- The Base Amount value may be several thousand counts.

An air/water spectrum obtained from a system with a large air leak and little water vapor is indicated by:

- The peaks at masses 18, 19, 28 and 32 are broadened. As a leak increases, all peaks broaden and eventually become undifferentiated.

## Fixing a Large Air Leak

Typical sources of large air leaks are

- Particles or damage on the manifold flange O-ring seal.
- Particles or damage on the transfer line O-ring seal.
- The transfer line brass nut is loose.
- Poor O-ring sealing between the turbomolecular pump and the manifold.



**Do not over tighten the fittings; otherwise, you may generate an even larger leak.**

If you cannot eliminate the leak, vent the system, and check the O-ring on the manifold and transfer line for particles. Wipe off both O-rings with lint-free cloth.

The turbomolecular pump will probably fail to achieve its 100% speed if there is a leak or poor seal at the turbo/manifold interface. Never attempt to operate the system under these conditions.

## Fixing a Small-To-Moderate Air Leak

You may have more trouble finding and correcting a small-to-moderate air leak than a large one. Symptoms associated with small-to-moderate air leaks include the following:

- The peak at mass 28 will have increased, becoming significantly larger than the mass 18 peak.
- The air leak will probably increase the water background, particularly in humid environments. An increase in water vapor content will likely be accompanied by a 20% or greater increase in the 19:18 intensity ratio.

## Checking GC Connections

---

NOTE: Check the GC Maintenance Section for additional information for trouble shooting leaks.

---

To identify and correct a leak at the connections between the capillary column and the injector or transfer line, proceed as follows:

- Make sure that you are using ferrules of the correct size, i.e., 0.4 mm for 0.25-mm ID columns, and 0.5 mm for 0.32-mm ID columns.
- Make sure that the ferrule on the transfer line is a graphite/Vespel mixture. Most transfer line connection leaks occur on the high vacuum side (e.g., around the transfer line O-ring).

In the case of a graphite/Vespel ferrule, tighten each ferrule one-half turn beyond finger tightness. In the case of a graphite ferrule, tighten each ferrule three-quarters of a turn beyond finger tight.

- Leaks at the septum may arise from loose injector nuts or overuse of the septum. Regularly change the septum as part of your routine GC preventive maintenance program. To reduce the level of air bleeding into the system and any background from the septum material, use good quality, low bleed septa.
- Air leaks in the GC pneumatics are the most difficult leaks to detect and eliminate because detection gases are not particularly effective for this purpose. In general, you should tighten all fittings.
- Saturated filters on the GC may produce an increase in the air/water background. Replace the filters at regular intervals and whenever moisture or other background from the GC becomes a problem.

## Troubleshooting Air Leaks Using Leak Detection Gas

You may use a leak detection gas such as difluoroethane to locate leaks. For example, difluoroethane is sold commercially under the name Dust-Off. A leak at the transfer line (the high vacuum side) should produce an immediate response. If, on the other hand, the leak is coming from the GC injector, it will take about 90 sec to register a response. (It takes about that length of time for the gas molecules to travel through the capillary column.) If you discover a leak at the injector, you can correct the problem without venting the system; however, be sure to wait until all GC zones are cool before beginning. If the leak is coming from a transfer line O-ring seal, you will have to shutdown the GC/MS system and vent the system before fixing it.

---

NOTE: Use the Leak segment of the C:\VarianWS\4000 MS Service\4000 MS (Int or Ext) Service.mth method in Manual Control. If necessary, edit the mass range as appropriate for the detection gas selected.

NOTE: Do not spray indiscriminately around the fittings. Typical leak detection gases such as Freon or argon diffuse very rapidly from the fitting you are testing toward a true leak. This could lead you to identify mistakenly the fitting that you are testing as the leak source.

---

Check for leaks:

- Spray a fine stream of detection gas on the transfer line closest to the analyzer.
- Examine the monitor for a response. If a peak at an appropriate mass for the gas selected does not appear, there is no leak at the transfer line seal.
- If a peak appears, there is a leak. The transfer line O-ring may have particles on its surface. Shut down the system and check the O-ring.

Also, check the following gaskets and fittings for leaks. (Tighten the fittings and/or flanges as needed. Wait a few seconds between subsequent applications of leak detection gas.)

- Pneumatics manifolds
- Vent valve fitting
- Vacuum manifold flange
- Transfer line nut
- Injector nut
- Septum nut

---

## Fixing High Water Levels

The presence of excess water vapor may be due to

- Failure to bakeout for a sufficient length of time (i.e., at least two hours, when you vent the system).
- Introduction of water vapor when you clean the ion trap.
- Introduction of water vapor when you replace the capillary column.
- Water vapor in the carrier gas tank.
- An atmospheric air leak in the system. This problem most often occurs under conditions of high relative humidity.
- In external mode, the helium getter is expended.

You will often observe high water backgrounds after venting the system, and especially after cleaning the trap. Several hours of bakeout may be required for the water vapor to desorb from surfaces in the vacuum system, and for the water level to drop to a stable level. Never operate your system if the mass 18 and 19 peaks are the same height. After the system has baked out sufficiently (e.g., overnight), the presence of excess water is due to contamination in the carrier gas tank, moisture collecting in cold spots, or an air leak.

Saturated filters on the GC may produce an increase in the air/water background. Replace the filters at regular intervals, and whenever moisture or other background from the GC becomes a problem.

---

## GC Troubleshooting

---

**NOTE:** Please refer to the GC Operator's Manual for information about GC troubleshooting and diagnostics procedures not described in this section.

---

This section describes chromatographic troubleshooting, with particular emphasis on GC/MS applications. You will be able to investigate most of the problems addressed in this section by running the COLTEST mixture (03-920273-00).

The following procedure describes the chromatographic conditions and the expected results when running the COLTEST sample with a 30-m vf5ms column (0.25 mm ID, 0.25 µm film thickness).

---

## Using the COLTEST Sample for Troubleshooting

---

The Coltest sample provides a good mechanism for identifying a variety of chromatography problems. A COLTEST method can be found in the C:\VarianWS\4000 MS Service directory of the software.

### Running the COLTEST Sample

#### ***Flow Pressure Conditions***

Use a constant flow of 1.0 mL/min.

#### ***Injector Conditions***

- 1177 Injector:

Use an isothermal temperature of 240 °C.

Set up the following split program conditions:

Time	Split State 1	Split Ratio
initial	On	100
0.01	Off	Off
1.00	On	100

#### ***MS Temperature Conditions***

1. Set the transfer line temperature to 250 °C.
2. Set the trap temperature to 150 °C.
3. Set the manifold temperature to 40 °C.
4. Set the source temperature to 150 °C if the 4000 MS is in external mode.

The COLTEST test mixture contains the following compounds at levels of 1 to 5 ng/ $\mu$ L.

No.	Compound	Formula	Integer Weight	Quantitation Mass
1	Decane	C <sub>10</sub> H <sub>22</sub>	142	57
2	1-octanol	C <sub>8</sub> H <sub>18</sub> O	130	69
3	Undecane	C <sub>11</sub> H <sub>24</sub>	156	71
4	Nonanal	C <sub>9</sub> H <sub>18</sub> O	142	67
5	2,6-dimethylphenol	C <sub>8</sub> H <sub>10</sub> O	122	107
6	2-ethylhexanoic acid	C <sub>8</sub> H <sub>16</sub> O <sub>2</sub>	144	73
7	2,6-dimethylaniline	C <sub>8</sub> H <sub>11</sub> N	121	106
8	decanoic acid, methyl ester	C <sub>11</sub> H <sub>22</sub> O <sub>2</sub>	186	74
9	undecanoic acid, methyl ester	C <sub>12</sub> H <sub>24</sub> O <sub>2</sub>	200	87
10	Dicyclohexylamine	C <sub>12</sub> H <sub>23</sub> N	181	138
11	dodecanoic acid, methyl ester	C <sub>13</sub> H <sub>26</sub> O <sub>2</sub>	214	143
12	Hexachlorobenzene	C <sub>6</sub> Cl <sub>6</sub>	282	284

You can also effectively separate the individual components in the mixture for subsequent data manipulation, e.g., library searches and quantitation.

## Troubleshooting Common Chromatographic Problems

The COLTEST mixture includes polar or active compounds such as 1-octanol, 2,6-dimethylphenol, and 2,6-dimethylaniline. Also present are some non-polar or inactive compounds such as decane and dodecane at approximate levels of 1 ppm in hexane. Analysis of the mixture yields information about solvent tailing, column efficiency, dead volume, active sites in the injector/column, etc. You can use the analysis to troubleshoot common chromatographic problems. The following table identifies many of the problems, and proposes solutions.

## Correcting Solvent Tailing or Broadening Problems

Possible Cause	Solution
Poor column installation resulting in dead volume in the injector	Reinstall the column in the injector. Make sure you have a good cut on the column by examining the column under magnification.
Solvent flashing in hot injector.	Reduce the injection speed. If possible, reduce the injector temperature. If you are using sandwich injection, reduce the solvent plug to 0.5 $\mu$ L.
Septum purge line is plugged	Check that the septum purge flow is 3.5-4.5 mL/min with a 10-psi head pressure. If necessary, adjust the valve setting.
Injector is not purged properly following splitless injection	For a splitless injection, the vent flow should be at least 70 mL/min. The injector should be switched to the split mode 30 to 90 sec after the injection.

## Correcting Tailing Sample Peaks for Particularly Active Components

Possible Cause	Solution
Active sites in the injector insert or liner	Change or clean the injector insert. If necessary, silanize it.
Active sites or degraded phase present in the column	Remove the front 15 cm of the column and reinstall it. If the retention times are changing, or if cutting the column does not fix the problem, replace the column.

## Correcting Low Response and Severe Tailing with High Boiling Point Compounds

Possible Cause	Solution
Injector not hot enough to vaporize high boilers	Increase the temperature of the injector
High levels of column bleed masking component peaks	Condition the column at 30 °C below its maximum operating temperature. Switch to a high temperature column if conditioning does not help.
High levels of silicone or other contamination are coated on the ion trap surfaces	Clean the ion trap as outlined in the Maintenance section.
Insufficient vaporization of the higher boiling point components	Raise the injector temperature and lower the injection speed.
Trap temperature is too low	Increase the trap temperature in increments of 20 °C.

## Correcting Leading Sample Peaks (Reverse Tailing)

Possible Cause	Solution
Column overhead due to injection of excessive amounts of a component	Dilute the sample, or perform a split injection.
Degradation of the stationary phase	Change the column.
Carrier gas velocity is too low	Increase the carrier flow rate.

## Correcting Poor Resolution<sup>1</sup>

Possible Cause	Solution
Column temperature or program is not optimized	Modify the method (e.g., slow the column ramp rate) to improve the separation
Carrier gas flow is not optimized	Decrease the carrier gas linear velocity to improve the resolution.
Column cannot separate certain species, (e.g., those with similar boiling points)	Use a more polar column.
Column stationary phase is degraded, resulting in poor efficiency	Replace the column.

<sup>1</sup>Peaks are not well separated, e.g., 2,6-dimethylphenol and 2-ethylhexanoic acid in the COLTEST mixture.

## Lack of Peak Size Reproducibility

Possible Cause	Solution
Leaking or partially plugged syringe	Visually check that the syringe is pulling up the sample. Check that the nut is tight. Flush the syringe with solvent. Replace the syringe.
Leak at the septum	Replace the septum regularly and ensure that the septum nut is tight.
Improper installation of column in the injector, or a leak at the column inlet	Check the installation of the column in the injector. Tighten the capillary column nut.
Sample is being absorbed by active surfaces in the injector or column	Change the injector insert. Remove the front 15 cm of the column, or replace the column.
Incomplete vaporization of sample in the injector	Increase the injector temperature.
Injector splits too soon.	Confirm that the switch time is chromatographically optimized.

## Correcting Peak Splitting (Particularly for Low Boilers)

Possible Cause	Solution
Sample flashing in injector simulating two injections	Lower the injection temperature.
Column is cracked	Re-cut and install the column.
A piece of septum is stuck in the injector insert.	Replace the insert and septum.

## Correcting Extra, Unexpected Peaks in the Chromatogram

Possible cause	Solution
Septum bleed	Use high-temperature, low-bleed septa. Make sure that the septum purge flow is set correctly.
Impurities from the sample vials (e.g., plasticizers present)	Confirm that this is indeed the case by running a solvent blank with a new syringe. Use certified sample vials, and keep the samples refrigerated.
Impurities from the carrier gas present	Install or replace the carrier gas filters.
Injector or GC pneumatics contaminated	Remove the column from the injector and bake it out at elevated temperature, (e.g., 350 °C) using a split vent flow of at least 20 mL/min.
Impurities present in the sample	Confirm that this is indeed the case by running a blank or standard.
Solvents are extracting impurities from the septum.	Switch to a new septum type, lower the injection temperature, or reduce the injection volume.
Impurities present in syringe wash solvent	Use high purity grade solvents.

## Correcting Retention Time Differences Between Runs

Possible Cause	Solution
Unstable carrier gas flow controller/regulator	Check the pneumatics for leaks. If necessary, replace the flow controller/ regulator.
Column contamination or degradation	Condition or replace the column.
Injector leaks	Replace the septum at regular intervals. Check that the septum nut and capillary column nut are tight.



### WARNING

If the equipment is used in a manner not specified in this manual, the protection provided by the equipment may be impaired.



# Miscellaneous Procedures and Instructions

---

## Other Documents

Other documents that you may wish to consult regarding 4000 MS operation include the following:

- 4000 GC/MS Internal Ionization Users Guide (03-954032-00)
  - 4000 GC/MS External Ionization Users Guide (03-954033-00)
  - 4000 GC/MS Hybrid Ionization Users Guide (03-954034-00)
  - 4000 MS Data Handling Users Guide (03-954038-00)
  - 4000 GC/MS Software Operation Manual (03-914999-00)
  - 4000 GC/MS Pre-installation Instructions (03-914997-00)
- 

## Site Requirements

### Site Preparation

The 4000 MS has been designed to operate reliably under carefully controlled environmental conditions. It is the responsibility of the purchaser to provide a suitable location, a power source of acceptable quality, and a suitable operating environment. Operating a system or maintaining it in operational condition outside of the power and operating environment limits listed below could cause failures of many types. The repair of such failures is specifically excluded from the standard warranty and service contract coverage.

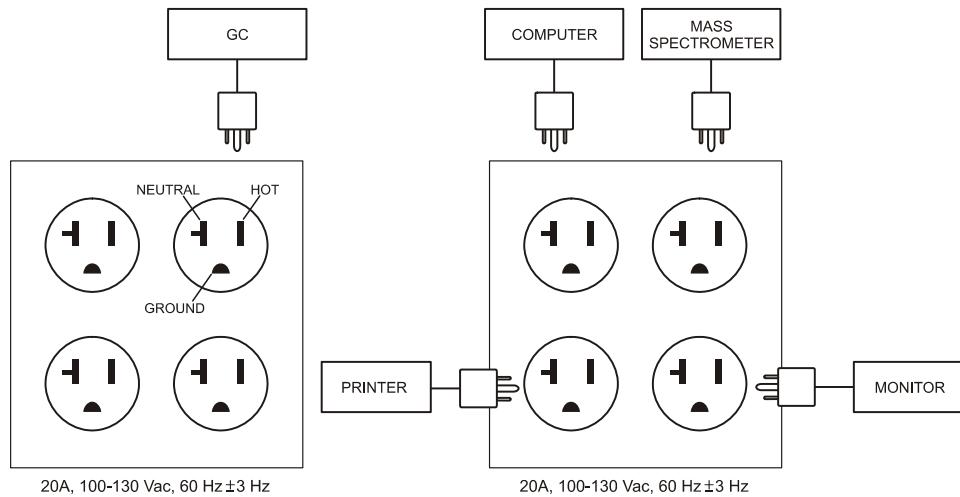
For additional information, please request specific pre-installation support directly through your local Varian Sales/Service Center.

### Power

You are responsible for providing two dedicated fourplex single-phase power sources with earth grounds hard-wired to the main power panel ground. Within North America or Japan these power sources must be 20A, 90-130 Vac, 60 Hz  $\pm 3$  Hz, and outside North America they must be 10A, 180-260 Vac, 50 Hz  $\pm 3$  Hz. One of these fourplex power sources is for the mass spectrometer, computer, monitor, and printer. The other fourplex power source is for the gas chromatograph and (optional) autosampler. If you have additional sample

preparation devices or test equipment, we recommend a separate dedicated power source for their operation.

NOTE: Do not use the free outlet for equipment that draws more than 2 amps.



*Interconnect Diagram for the 4000 MS*



**CAUTION**  
Avoid using power supplies from sources that may be subject to RF interference, such as electric motors and elevators.

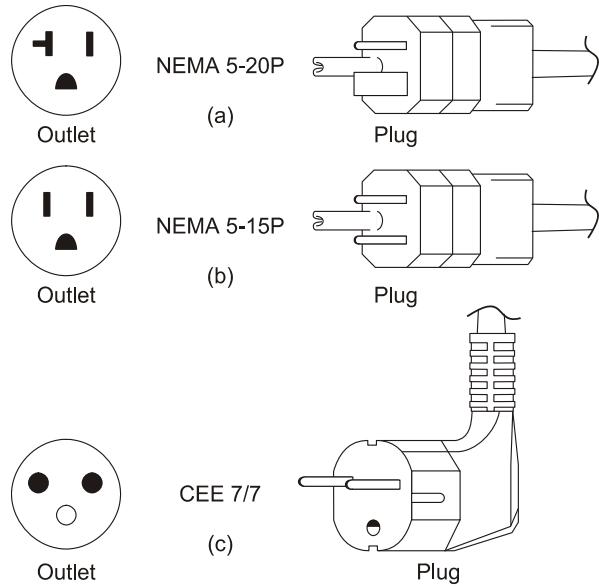
Care must be taken to ensure that sources of radio frequency interference (RFI) and electromagnetic interference (EMI) are not placed on the same power line, or share the same ground plane, since this can degrade the performance of the GC. Equipment such as motors, solenoids, fluorescent light fixtures, and radio communication transmitters should be isolated from the instrument and connecting cables as much as possible.

The power cable from the GC is approximately 2m (6 ft) long and fitted with National Electronics Manufacturers Association (NEMA) 5-20P power plugs. The NEMA 5-20P power plug and corresponding outlet are shown in Figure (a). NEMA 5-20P plugs are rated at 20A and 120 Vac.

The power cable from the mass spectrometer is approximately 2.5m (8 ft) long and fitted with US Standard National Electronics Manufacturers Association (NEMA) 5-15P power plugs. The NEMA 5-15P power plug and corresponding outlet are shown in Figure (b). NEMA 5-15P plugs are rated at 15A and 120 Vac.

Systems shipped outside the United States, Canada or Japan are fitted with CEE 7/7 plugs; these are rated at 16A and 230 Vac. The CEE 7/7 plug and outlet are shown in Figure (c).

The power cables for the computer, monitor, and printer are approximately 2m (6 ft) long. They are fitted with NEMA 5-15P plugs. The power cable from the 8400 AutoSampler is about 2m (6 ft) long, and is fitted with a NEMA 5-15P plug rated at 120V.



*NEMA 5-20P, NEMA 5-15P, and CEE 7/7 Power Plugs and Outlets*

With a 120V power source, the maximum amperage requirements for each of the 4000 MS components are as follows:

Component	Amperes
Mass Spectrometer	12
Gas Chromatograph	20
Varian 8400 AutoSampler	0.5
Computer	3
Monitor	2
Laser Printer	3-4

---

NOTE: With a 230V power source, the maximum amperage requirement of each of the above components is one-half of the amperage given above.

---

Never plug the mass spectrometer and the gas chromatograph into the same power source; otherwise, you may overload the fourplex power source. The Interconnect Diagram for the 4000 MS shows the five power cables of a typical installation. Never use the free outlet on each of the power sources for equipment that draws more than 2A.

## Quality of Power

The quality of the power supplied to your 4000 MS is very important. The power must be 90 -130 Vac, 60 Hz  $\pm$ 3 Hz (180-260 Vac, 50 Hz  $\pm$ 3 Hz), and it must be stable. It must be free of fluctuations due to slow changes in the average voltage or to changes resulting from surges, sags, or transients.

- Slow average changes are gradual, long-term changes in the average root mean square (RMS) voltage level, with typical durations greater than 2 seconds.
- Sags and surges are sudden changes in average RMS voltage level, with typical durations between 50  $\mu$ sec and 2 seconds.
- Transients (or impulses) are brief voltage excursions of up to several thousand volts with durations of less than 50  $\mu$ sec.

Constant high line voltage or surges in voltage can produce overheating and component failures. Constant low line voltage or sags in voltage may cause the system to function erratically or to stop functioning. Transients, even of a few microseconds duration, may cause electronic devices to fail catastrophically or degrade sufficiently to significantly shorten their lives. Therefore, it is important to establish the quality of the line power in your laboratory before you install your 4000 MS.

Occasionally, you may encounter line power sources of unacceptable quality; such power sources may adversely affect the operation of the 4000 GC/MS. The 4000 GC/MS is tested under EMC Standard 61326-A1 + A2. If voltage conditions exceed those standards, additional power conditioning or surge protection is advised. You may want to contact a specialist in power conditioning services.

To protect against power failures, an Uninterruptible Power Supply (UPS) can be used. The amount of power drawn depends on instrument operating conditions but 4KVA should be sufficient under typical acquisition conditions, at normal line voltage. Greater power may be drawn during system power up or bakeout. The UPS should have a switchover time of 20 ms. or less.

---

## Operating Environment

It is your responsibility to provide an acceptable operating environment. Attention paid to the operating environment will ensure continued high performance of your 4000 MS. Expenditures for air conditioning will be more than offset by good sample throughput and a reduction in repair costs.

### Temperature

The laboratory temperature must be held between 15 and 30 °C (59 and 86 °F).



**CAUTION**

As the laboratory temperature increases, system reliability decreases. All electronic components generate heat while operating. This heat must be dissipated to the surrounding air if the components are to operate reliably.

The turbomolecular pump's temperature cutoff protects the bearing and prolongs its lifetime. If the laboratory temperature is significantly above 30 °C (86 °C), the pump cutoff temperature could be reached, and if so reached, would result in the pump being shutdown.

There must be a good flow of air around the system, and the air conditioning must be capable of maintaining a constant temperature (within operational limits) in the immediate vicinity of the system. Using demanding GC methods the average steady-state heat load of the 4000 MS is 6000 BTUs.

## Humidity

The relative humidity of the operating environment must be between 40 and 80%, with no condensation. Operating a 4000 MS at very low humidity will result in the accumulation and discharge of static electricity; this will shorten the life of electronic components. Operating the system at high humidity will produce condensation and result in short circuits. High humidity will also block the filters on cooling fans and accelerate wear of the heads in the diskette drives.

Varian recommends that your laboratory be equipped with a temperature/humidity monitor. This will ensure that your laboratory is always in conformance with temperature and humidity specifications.

## Exhaust System

It is your responsibility to provide an adequate exhaust system. Much of what is introduced into the mass spectrometer will eventually be exhausted from the mechanical pump, along with the small amounts of oil vapor that these pumps characteristically emit. Therefore, the pump outlets should be connected to a fume exhaust system. Consult local regulations for the proper method of exhausting the fumes from your system.

---

# Gas Requirements

## Helium - GC Carrier Gas

Minimum 99.998% ultra-high purity with less than 1.0 ppm each of water, oxygen, and total hydrocarbons. One 257-ft<sup>3</sup> tank with Matheson regulator #3104-580, or equivalent tank and regulator.

---

NOTE: The presence of >1 ppm oxygen or water in the carrier gas supply may significantly affect the performance of the 4000 MS, and it may damage such components as the capillary column, filaments, and multiplier. Varian recommends that its customers verify that their gas suppliers use controlled tanks; this will ensure that purity standards are maintained. If you purchase pure gases in contaminated tanks, you may end up with a contaminated system requiring costly and time consuming repair.

---

## **Methane, Isobutane, Ammonia - CI Reagent Gases (with CI option only)**

99.99% purity. One gas cylinder with a two-stage pressure regulator that has a stainless steel diaphragm and maximum inlet pressure of 30 psi (200 kPa).

## **Cryogenics**

Systems equipped with SPI/1079 injectors or column oven cryogenics require one of the following:

- Liquid CO<sub>2</sub> at 850-1000 psig
- Liquid N<sub>2</sub> at 20-50 psig

If you are not sure which one of these cryogenic options you ordered, check your purchase order.

---

## **How to Install the 4000 MS**

To install the 4000 MS, proceed as follows:

1. Connect the GC to a helium source, and then purge the system filters and columns for 15 minutes.
2. Feed the capillary column and nut through the side of the GC. Connect the column to the transfer line.
3. Slide the 4000 MS toward the GC until the transfer line is protruding into the GC oven.
4. Connect vacuum tubing from the rear of the 4000 MS to the foreline pump with a clamping ring.
5. Connect the power cord from the foreline pump to the rear of 4000 MS (J2 label pump power only).
6. Connect the GC Ethernet cable to the Ethernet port on the computer. There should be a tee connector and a terminator at each end of the 50-ohm coax cable.
7. Connect the USB cable to the 4000 MS and the computer.
8. Plug in the GC, ms-spectrometer, and data-system power cords.
9. Switch on the power to the 4000 MS, the GC, and the computer.
10. Bring up System Control on the computer.
11. Select Diagnostics.
12. Check the turbomolecular pump speed. The pump speed should reach the 100% value within 30 min of turning on the power to the mass spectrometer.
13. Bake-out the trap (250 °C) and manifold (120 °C) for at least 2 hours before you tune it.

## How to Move the 4000 MS

To move the 4000 MS proceed as follows:

1. Using the shutdown procedure, shut down the GC and mass spectrometer.
2. Turn off the GC and computer. Then unplug the GC, mass spectrometer, and data system power cords.
3. Keep an eye on the capillary column inside the GC as you gently slide the mass spectrometer away from the GC. Be sure not to bend or kink the capillary column.
4. Use the alignment tool to prevent the transfer line from turning while you loosen the brass capillary nut connecting the column to the transfer line.
5. Cap the transfer line with a capillary nut and no-hole ferrule.
6. Place the capillary column and nut inside the GC oven. This will protect them from damage.
7. Turn off the carrier gas, and then disconnect the helium gas line that is connected to the GC filter.
8. Cap the filters with Swagelok® plugs or caps.
9. Move the 4000 MS to its new location. Be sure the new location satisfies the power and environmental requirements.

# Parts and Supplies

## Electronics

Part Number	Description
03-925305-02	Assy, Chassis Fan, Analyzer Side
03-931410-01	Assy, Transferline Heater
03-931417-01	Assy , Cable, Power, Turbo
03-932403-01	Cable, Flat, 4000 EFC
03-930102-04	Valve, Solenoid,2-Way,BUNA-N W/Pins
03-930106-01	Valve, Solenoid,2-Way, Manifold Mount, Chemrez Seals
03-930107-03	Valve, Solenoid,3- Way , Manifold Mount, Vitron Seals
03-931325-01	Assy, Flex Circuit, Heaters
03-931420-01	Assy, Flex Circuit, Filament
03-931425-01	Assembly, Adaptor, Int Ion Source
03-931437-01	Assy, Cable, GC/4000MS Start

## Pneumatics

Part Number	Description
03-931124-91	Kit, Getter Replacement
03-930100-01	Valve, Needle, Parker, CAL-GAS
03-932641-01	He EFC Assembly
03-931772-01	Tube, CI IN, Pneumatic Blk/Needle Blk
03-930101-01	Valve, Needle , Parker , CI-GAS
03-930107-02	Valve, Solenoid, 3-way, Manifold Mount, Vitron Seals
03-925707-00	Rivet, Solid, 1/8 X ¾

## Analyzer, Attached to Top Flange

Part Number	Description
03-931739-01	Assy, Gate, Int Ion Source, Clean
03-931738-01	Assy, BASE, Int Ion Source, Clean
03-931737-01	Ring, Center, Int Ion Source
03-931736-01	Plate, Retaining, Int Ion Source, Clean
03-931020-01	Assy, Source, Internal Ionization
03-930535-01	Spacer, Quartz Clean, Not Coated
03-930535-02	Spacer, Quartz, Clean, Silco Coated
03-931018-01	Assy, Trap
03-931611-01	Isolator, Lens and Screw
03-931622-01	Shield, Flex Circuit
03-931671-01	Spacer, Magnet/Oven

<b>Part Number</b>	<b>Description</b>
03-931672-01	Thumbscrew, Trap Oven
03-931028-01	Trap Oven Half, Entrance (see Note below)
03-931028-02	Trap Oven Half, Exit (see Note below)
03-931027-03	Assy, Trap Heater, External Source
03-931677-01	Structure, Magnet, External, w/Magnet Holes
03-931677-02	Structure, Magnet, External, No Magnet Holes
03-920174-01	Filament, 4000MS, Internal Ionization
03-931675-93	RF Electrode, Silco Stl Coated, Cleaned
03-931644-93	Assy, Silco End Cap With Plug
03-931712-01	Internal Transfer line Tip, Cleaned
03-930605-01	Spring Gold Plated, Trap
03-931670-01	Block, External Source
03-931678-01	Magnet Holder
03-931679-01	EI Volume
03-931680-01	Gasket, Ext. Source
03-931681-01	Retainer, Lens, Pins, External Source
03-931683-01	Ring, Center, Trap
03-931684-01	Assy, Lens 1
03-931685-01	Assy, Lens 2
03-931686-01	Assy, Lens 3
03-931610-01	Assy, External Filament, Base w/Posts, Filament and Screws
03-931017-01	Assy, External Source
03-931758-01	Spring, External Source, CI Volume Retract
03-931761-01	Disc, Magnet
03-931711-01	Tip, Transfer line, External
03-931607-01	Holder, CI Volume
03-931608-01	Volume, CI

NOTE: If your oven has either a solid aluminum or solid black anodized surface, you must order both halves of the oven in (part number 03-931139-91).

If the front and back of the oven are black anodized and the sides are aluminum, then order only the half you need.

The ovens include a heater and a temperature sensor.

## **Analyzer, Attached to Manifold**

<b>Part Number</b>	<b>Description</b>
03-931012-01	Assy, Transfer line, 4000 MS
03-931640-01	Clamp, Turbo
03-931689-01	Assy, Vent Stem
03-931691-01	Electrode, Conversion, Dynode
03-931751.01	Multiplier, Channel, Model CEM 4755
03-931753-01	Strap, High Voltage, Multiplier
03-931757-01	Inlet, Helium, Manifold-Trap, Polyimide
03-931762-01	Knob, Vent
21-719935-00	Spring, COMP, 0.210 OD, 0.026 DIA, 0.380L, SST
21-709266-00	Spring, COMP, .720 OD, .055 Wire, 3.0L, SH.587
03-931774-01	CI Gas Inlet , Manifold
03-931726-01	Elbow, Vacuum, 4000 MS

## **Chemical Ionization**

<b>Part Number</b>	<b>Description</b>
03-930555-01	CI Manifold
03-931790-01	CO Block Frit Spacer
03-930556-01	CI Plate
03-931774-01	CI Gas Inlet, Manifold
12-221106-24	6-32 x 1½ Screw

## **Vacuum**

<b>Part Number</b>	<b>Description</b>
03-931119-91	Kit, Turbo Replacement, V301
88-299538-00	GP Oil
27-101002-00	Oil Mist Cartridge Replacements pk. of 2
03-930660-01	DS102 115V
03-930661-01	DS102 230V
03-930662-01	DS102 100-120V, 200-240

## O-Rings

Part Number	Description
03-930109-25	O-ring, 1.176 ID, .070 DIA, Viton, Clean
03-930109-20	O-ring , 2-135, 1.925ID, 0.103 DIA, Viton (transfer line)
03-930109-24	Viton O-ring , Top Flange PCB, 2-148, 7.484 ID, Quad
03-930109-10	BUNA O-ring Clean 0.125
03-930109-07	O-ring , 2-108, 0.237 ID, 0.103 DIA, Viton
03-930109-27	O-ring , 1.049 ID, 0.103 IDA, Viton, Clean
03-930109-18	O-ring , Baked, Quad-X Seal, 1.112 ID
03-930109-28	0.145 ID Viton O-ring
03-930109-11	0.239 ID Viton O-ring
03-905960-09	0.348 OD O-ring

## Miscellaneous/Other

Part Number	Description
28-693976-00	Union 1/16 SST for PID, ELCD (HALL)
28-247071-00	1/8" Brass Plug
03-917084-00	1/8" Capillary Column Nut
03-931783-01	He Getter Mounting Clip
88-299440-00	Vacuum Grease
03-917142-00	Viton Ferrule
03-917157-00	Viton Ferrule Washer
22-119650-00	Cable Tie
28-849792-00	Fitting, Screw Plug, 10-32 Brass NI Plated
28-158923-00	Polyurethane Tubing Clear
28-993060-00	1/8" Clutch Clamp
28-158611-00	Tubing, Poly, 1/8" X 1/16" Red
28-158603-00	Tubing poly, green
28-849793-00	Fitting, 10-32 THD, Male Tube, Brass NI
03-931805-01	Tool, Internal Column Length
03-930604-01	Alignment Tool Wrench, Saturn 2000
29-900077-00	Key, Hex, 6 mm
03-931411-03	Cable, USB 2.0, 3 Meter Long
03-931699-01	Holder Trap Service
03-931103-91	Kit, Standard Accessory, 4000 MS

## Test Samples

Part Number	Description
03-930652-01	Perf. Eval. Std. GC/MS (Internal EI & CI)
03-930127-01	Test Std. 4000 MS In External EI (2 pg/ $\mu$ L OFN)
03-920305-00	Benzophenone External CI Sensitivity Sample (50 pg/ $\mu$ L)
03-931130-01	Test Std, 4000MS In External NCI (1 pg / $\mu$ L DFB)
03-920353-00	Calibration Compound/Haz
03-920273-00	GC/MS Column Test Mix/Haz

---

## Varian Service

If you have a problem with your 4000 MS that you are unable to resolve using the procedures described, you may want to call a Varian Customer Support Representative. When you call, please be prepared to provide the following information:

- 4000 MS serial number (located on the front panel)
- Installed options.
- Diagnostics test results

If you are having problems with the gas chromatograph, please be prepared to provide the following information:

- GC model
- Autosampler model, if any
- Type of injector you are using
- Cryogenics
- Information about your GC column, (i.e., the manufacturer, bonded phase, film thickness, ID, and length)

If you are having problems with your computer and/or software, please be prepared to provide the following information:

- Computer manufacturer and model
- Windows version
- Mouse driver version
- Printer manufacturer and model
- Network configuration
- Printouts of your Autoexec.bat and Config.sys files
- MS Workstation software version

In addition, you should observe the following guidelines when describing the problem to the Customer Support Representative:

- Tell the service representative which part of the software, System Control, Manual or Acquisition, for example, you were using when the problem occurred.