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OPTIC 3

High Performance GC Injector

User's Guide

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CE Declaration of Conformity

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ATAS GL International B.V. hereby declares under our sole responsibility that our OPTIC 3 High Performance Injector when installed in accordance with installation specifications is in conformity with the above Directives and Standards:

N. Gribov
R&D Manager

Date: December 4, 2002

Contents

1. Warnings and Cautions

This section explains the warnings and cautions that should be observed when installing or operating OPTIC 3.

Safety information is covered at relevant points throughout the manual. Please read this manual in its entirety before installing or operating the instrument. Should any point remain unclear, contact your supplier for assistance before proceeding.

1.1 General OPTIC 3 Warnings and Cautions

OPTIC 3 is designed for installation and use in a laboratory environment by suitably trained personnel. All relevant safety aspects should be assessed in accordance with the local regulations before installing and commissioning the instrument.

1.1.1 General OPTIC 3 warnings

OPTIC 3 is intended for use in appropriately equipped chemical analysis laboratories. In particular, provision must be made for the storage of solvents in flame-proof cabinets and personnel must wear protective clothing and eye protection at all times.

Both OPTIC 3 and the host GC must be disconnected from the mains power supply until installation of the equipment is complete. OPTIC 3 must be powered from an earthed (grounded) outlet.

There are potentially hazardous voltages present inside the OPTIC 3 control unit. Switch off the control unit and disconnect the mains power supply before removing the control unit cover.

The operation of the injector requires that it heats rapidly to high temperatures. Parts of the injector may remain hot for some time after the unit is switched off. Care must be taken when working in the area of the injector. It is especially important to ensure that the injector is cool (below 40°C) and switched off before changing injector liners.

Most organic solvents are toxic to some degree and substantial amounts of solvent vapor may emerge from the exhaust port. It is highly recommended that these vapors are ducted to a fume hood, or an active carbon trap is fitted to the port. However, the flow through this port should not be restricted as this might interfere with the analyses.

The OPTIC 3 gas controls are not designed for operation with explosive (besides hydrogen), corrosive or toxic gases.

The use of hydrogen as a GC carrier gas is potentially dangerous. It is potentially explosive. Take extreme care when using hydrogen as the GC carrier gas in a GC or GC/MS system.

1.1.2 General OPTIC 3 cautions

It is important that a good connection is made between the injector power cable and the injector. A bad connection can result in poor operation and the connection becoming excessively hot.

When leak testing connections, use a suitable electronic leak detector or a 50/50 solution of Propanol and water. Under no circumstances should a soap solution or similar be used as this will contaminate the injector.

The injector control box must not be switched on with the injector disconnected as this could cause serious damage to the instrument.

1.2 Warnings and cautions related to the OPTIC 3 cryotrap

The following warnings and cautions relate to the cryotrap fitted to OPTIC 3-SC and OPTIC 3-DC.

1.2.1 Warnings related to the OPTIC 3 cryotrap

The cryotrap uses liquid Nitrogen to produce the cold gas for low temperature operation. Personnel must be thoroughly familiar with properties and safety considerations before being allowed to handle liquid Nitrogen and the cryotrap.

Liquid Nitrogen can cause serious burns. Hand protection and goggles (not safety glasses) are to be worn at all times when handling it. Never allow any unprotected part of the body to come in contact with un-insulated tubing or parts that contain cryogenic products. The extremely cold metal will cause the flesh to stick rapidly and tear when you attempt to withdraw.

Dewar with liquid Nitrogen should be handled and stored in an upright position. Do not drop Dewar or tip it on its sides.

1.2.2 Cautions related to the OPTIC 3 cryotrap

Make sure that there is gas flowing through the tube coil of the cryotrap heat exchanger when immersing it in the liquid Nitrogen or leaving it in the Dewar for a long time. This is to avoid freezing of the water vapor present in the air, which would block the gas flow through the heat exchanger.

1. Waarschuwingen en aandachtspunten

Dit hoofdstuk beschrijft de waarschuwingen die in acht dienen te worden genomen tijdens het installeren en bedienen van de OPTIC 3.

Veiligheid informatie wordt behandeld op de relevante onderdelen in de handleiding. Lees eerst de handleiding in zijn geheel voordat u een installatie of bediening gaat uitvoeren. Blijft er een punt onduidelijk, neem dan altijd eerst contact op met uw leverancier.

1.1 Algemene OPTIC 3 waarschuwingen en aandachtspunten

De OPTIC 3 is ontwikkeld voor installatie en gebruik in een laboratorium omgeving door goed geschoold personeel. Alle lokale relevante veiligheid aspecten moeten in acht worden genomen voordat er aan de installatie van het instrument wordt begonnen.

1.1.1 Algemene OPTIC 3 waarschuwingen

De OPTIC 3 is bedoeld voor het gebruik in goed voorziene chemische analytische laboratoria. Voor de opslag van oplosmiddelen moet een vlam dovende kast aanwezig zijn en het personeel moet ten alle tijden beschermende kleding en bril dragen.

Zowel de GC en de OPTIC 3 moeten afgekoppeld zijn van de netspanning totdat de installatie is afgerond. De OPTIC 3 moet worden aangesloten op een geaarde netspanning.

In de bedieningsunit van de OPTIC 3 zijn gevaarlijke hoogspanningen aanwezig. Schakel de unit uit voordat u een van de zijplaten wilt verwijderen.

Voor de werking van de injector is het nodig dat deze snel kan verwarmen naar hoge temperaturen. De injector kan enige tijd warm blijven nadat de unit is uitgeschakeld.
Let goed op wanneer er gewerkt word in de omgeving van de injector. Het is vooral belangrijk dat de injector is afgekoeld tot een temperatuur lager dan 40°C en de unit is uitgeschakeld voordat er liners worden gewisseld.

De meeste organische oplosmiddelen zijn giftig, de een meer dan de andere. Een hoeveelheid damp van het oplosmiddel komt uit de "exhaust" port van de unit. Het is aanbevolen deze dampen af te voeren via een afzuiging of op te vangen in een actief kool filter. Hierbij is het wel van belang dat er geen restrictie optreed, waardoor de analyse wordt beïnvloed.

De OPTIC 3 gas controle unit is niet ontworpen om te werken met explosieve, corrosieve en giftige gassen (behalve Waterstof).

Het gebruik van waterstof als dragergas is kan gevaarlijk zijn vanwege de explosiviteit. Let daarom extra goed op bij het gebruik van waterstof als draaggas bij een GC of GC/MS systeem.

1.1.2 Algemene OPTIC 3 aandachtspunten

Het is van belang dat er een juiste verbinding wordt gemaakt tussen de injector power kabels en de injector zelf. Een slechte verbinding kan een slechte werking veroorzaken en de verbindingen kunnen extreem heet worden.

Tijdens het zoeken naar een lek is het van belang dat er gewerkt word met een geschikte elektronische lekdetector of met een oplossing van 50/50 -propanol en water. Onder geen enkele voorwaarde mag er een zeepoplossing of een vergelijkbare oplossing worden gebruikt, dit zal de injector vervuilen.

De injector bedieningsunit mag niet worden aangezet wanneer de injector niet is aangesloten, dit kan onherstelbare schade aan de unit veroorzaken.

1.2 Waarschuwingen en aandachtspunten voor de OPTIC 3 Cryotrap

De volgende waarschuwingen en aandachtspunten zijn gerelateerd aan de OPTIC 3-SC en OPTIC 3-DC.

1.2.1 Waarschuwingen voor de OPTIC 3 Cryotrap

De Cryotrap gebruikt vloeibare stikstof om koud gas te maken voor de lage gebruikstemperaturen. De gebruiker moet goed bekend zijn met de eigenschappen en veiligheidsregels voor het gebruik van vloeibare stikstof en de Cryotrap.

Vloeibare stikstof kan ernstige brandwonden veroorzaken. Handschoenen en een goed aansluitende veiligheidsbril moeten ten alle tijden gedragen worden wanneer er gewerkt wordt met vloeibare stikstof. Zorg er voor dat de huid nooit in contact komt met een ongeïsoleerde leiding van het cryo systeem. Het extreme koude metaal zorgt er voor dat de huid aan het metaal plakt en wanneer men probeert los te komen trekt men de huid kapot.

Dewars (vat met vloeibare stikstof) dient men rechtop te vervoeren en te plaatsen. Gooi nooit een dewar om.

1.2.2 Aandachtspunten voor de OPTIC 3 Cryotrap

Zorg ervoor dat er een gas stroom door de warmtewisselaar loopt voordat deze in de vloeibare stikstof wordt geplaatst of wanneer deze voor een langere tijd in de stikstof blijft staan. Dit is om te voorkomen dat de warmtewisselaar bevriest door de aanwezigheid van water damp in de aanwezige lucht.

1. Sicherheitshinweise und Warnungen

Dieses Kapitel enthält Erläuterungen zu den bei Installation und Betrieb von OPTIC 3 zu beachtenden Sicherheitshinweisen und Warnungen.

Sicherheitshinweise werden an allen relevanten Stellen im Handbuch gegeben. Bitte lesen Sie dieses Handbuch komplett durch, bevor Sie Optic 3 installieren oder in Betrieb nehmen. Bei etwaigen Unklarheiten kontaktieren Sie bitte vor der Ingebrauchnahme Ihren Lieferanten.

1.1 OPTIC 3 - Allgemeine Sicherheitshinweise und Warnungen

OPTIC 3 wurde für die Installation und den Gebrauch durch entsprechend geschultes Personal in Laboratorien entwickelt. Vor Installation und Ingebrauchnahme sind sämtliche relevanten Sicherheitsaspekte unter Berücksichtigung der lokalen Vorschriften zu überprüfen.

1.1.1 OPTIC 3 - Allgemeine Warnungen

OPTIC 3 ist für die Benutzung in entsprechend ausgestatteten Laboratorien für chemische Analytik entwickelt. Es ist sicherzustellen, dass Lösungsmittel in feuersicheren Behältnissen aufbewahrt werden und Mitarbeiter zu allen Zeiten Schutzkleidung und Augenschutz tragen.

Sowohl OPTIC 3 als auch der GC sind von der Hauptstromzufuhr abzutrennen, solange die Anlage noch nicht komplett installiert ist. OPTIC 3 ist an eine geerdete Steckdose anzuschließen.

Im Innern des OPTIC 3 Steuerteils befinden sich Teile mit potentiell gefährlicher Betriebsspannung. Schalten Sie den Steuerteil ab und unterbrechen Sie die Hauptstromzufuhr, bevor Sie das Gehäuse des Steuerteils entfernen.

Betrieb des Injektors erfordert, dass er schnell auf hohe Temperaturen erhitzt wird. Teile des Injektors können noch heiß sein, nachdem das Teil abgeschaltet wurde. Beim Arbeiten in der Nähe des Injektors ist Vorsicht geboten. Es ist sicher zu stellen, dass der Injektor (unter 40°C) abgekühlt und ausgeschaltet ist, bevor die Injektor-Liner ausgewechselt werden.

Fast alle organischen Lösungsmittel sind bis zu einem gewissen Grad giftig. Da aus dem Auslassport hohe Mengen an Lösungsmitteldämpfen austreten können, wird dringend empfohlen, diese Dämpfe in eine Abzugshaube zu leiten oder einen Aktivkohlenfilter am Port anzubringen. Dadurch darf jedoch der Fließvorgang durch diesen Port nicht beeinträchtigt werden um korrekten Verlauf der Analyse zu gewährleisten.

Die OPTIC 3 Gaskontrollen sind nicht für Betrieb mit Explosivstoffen, (abgesehen von Hydrogen), Schadgasen oder toxischen Gasen gedacht.

Der Gebrauch von Hydrogen als GC-Trägergas ist gefährlich. Beim Gebrauch von Hydrogen als GC-Trägergas in einem GC- oder GC/MS-System besteht Explosionsgefahr – daher ist äußerste Vorsicht geboten.

1.1.2 OPTIC 3 - Allgemeine Warnungen

Es ist wichtig, dass eine gute Verbindung zwischen dem Injektor-Niedrigspannungskabel und dem Injektor hergestellt wird, da eine schlechte Verbindung die Leistung herabsetzen und die Verbindung extrem heiß werden lassen kann.

Beim Überprüfen der Verbindungen auf Lecks sollte ein geeigneter elektronischer Leckdetektor oder eine 50/50 Lösung aus -Propanol und Wasser benutzt werden. Keinesfalls Seifenlauge oder Ähnliches benutzen, da dies den Injektor kontaminiert.

Der Steuerteil des Injektors darf nicht angeschaltet werden, solange der Injektor nicht angeschlossen wurde, da dies das Instrument schwer beschädigen kann.

1.2 Sicherheitshinweise und Warnungen betreffend die OPTIC 3 Kühlfalle

Die folgenden Sicherheitshinweise und Warnungen betreffen die an OPTIC 3-SC und OPTIC 3-DC angebrachte Kühlfalle.

1.2.1 Warnungen betreffend die OPTIC 3 Kühlfalle

Die Kühlfalle wird mit flüssigen Stickstoff betrieben um das kalte Gas für Niedrigtemperatur-Betrieb zu erzeugen. Vor der Benutzung ist sicherzustellen, dass das Bedienungspersonal mit den Eigenschaften flüssigen Stickstoffs und den Sicherheitsanforderungen betreffend die Kühlfalle eingehend vertraut ist.

Flüssiger Stickstoff kann schwere Verbrennungen verursachen. Beim Umgang damit sind grundsätzlich Handschutz und Vollsichtbrillen (keine Schutzbrillen) zu tragen. Ungeschützte Körperteile dürfen keinesfalls mit nicht-lierten Leitungen oder Teilen in Kontakt kommen, die Tieftemperaturprodukte enthalten, da die Haut unverzüglich am extrem kalten Metall kleben bleibt und beim Zurückziehen abgerissen wird.

Dewarbehälter mit flüssigem Stickstoff sind senkrecht zu hantieren und zu lagern. Dewarbehälter nicht fallen lassen oder umdrehen.

1.2.2 Warnungen betreffend die OPTIC 3 Kühlfalle

Stellen Sie sicher, dass Gas durch die Rohrschlange des Kühlfallen- Wärmetauschers fließt, wenn sie ihn in den flüssigen Stickstoff tauchen oder für lange Zeit im Dewarbehälter lassen um Vereisung des Wasserdampfes in der Luft zu verhindern, was den Gasfluss durch den Wärmetauscher unterbrechen würde.

1. Mises en garde et précautions d'emploi

Cette section expose les mises en garde et les précautions d'emploi à observer lors de l'installation ou du fonctionnement d'OPTIC 3.

Les informations relatives à la sécurité sont présentées en différents endroits du manuel lorsque ce point s'impose. Nous vous prions donc de lire le manuel dans son intégralité avant d'installer ou de faire fonctionner l'appareil. Si des points manquent de clarté, veuillez solliciter l'aide de votre fournisseur avant de procéder à la mise en marche de l'appareil.

1.1 Mises en garde et précautions d'emploi générales concernant OPTIC 3

OPTIC 3 est conçu pour être installé et utilisé en laboratoire par un personnel formé à cet effet. Tous les aspects de la sécurité doivent être déterminés en fonction des règles en vigueur dans les lieux concernés avant d'installer et de mettre en service l'appareil.

1.1.1 Mises en garde générales concernant OPTIC 3

OPTIC 3 est destiné à l'utilisation dans des laboratoires d'analyses chimiques correctement équipés. Des dispositions doivent être notamment prises pour que les solvants soient entreposés dans un lieu de rangement ininflammable et pour que le personnel porte en permanence des vêtements et des lunettes de protection.

OPTIC 3 tout comme le GC hôte doivent être déconnectés du réseau électrique tant que l'installation de l'appareil n'est pas terminée. OPTIC 3 doit être alimenté à partir d'une prise de terre.

Il peut y avoir des voltages dangereux présents dans l'unité de commande d'OPTIC 3. Eteignez l'unité de commande et débranchez-la avant d'enlever le couvercle.

Le fonctionnement de l'injecteur implique son réchauffement rapide afin d'atteindre des températures élevées. C'est pourquoi des éléments de l'injecteur peuvent rester très chauds une fois celui-ci éteint. Il faut donc faire attention lorsqu'on travaille à proximité de l'injecteur. Il est particulièrement important de s'assurer que l'injecteur est refroidi (au-dessous de 40°C) et éteint avant de procéder à toute opération de remplacement.

La plupart des solvants organiques sont dans une certaine mesure toxiques et une quantité non négligeable de vapeur de solvant peut sortir de la soupape d'échappement. Il est fortement recommandé de faire en sorte que ces vapeurs soient canalisées vers une hotte d'aération ou un filtre à charbon actif adapté à la soupape. Il ne faut toutefois pas restreindre l'évacuation qui se fait par cette soupape, ceci risquant de fausser les analyses.

Les commandes de gaz d'OPTIC 3 ne sont pas conçues pour un fonctionnement avec des gaz explosifs (à l'exception de l'hydrogène), corrosifs ou toxiques.

L'utilisation d'hydrogène en tant que gaz vecteur GC peut être dangereux puisqu'il s'agit d'un gaz potentiellement explosif. Vous devez prendre des mesures de précautions extrêmes lorsque vous utilisez de l'hydrogène en tant que gaz vecteur dans le système GC ou GC/SM.

1.1.2 Précautions d'emploi générales concernant OPTIC 3

La qualité du raccordement entre le câble électrique de l'injecteur et l'injecteur et très importante. Une mauvaise connexion peut entraîner un mauvais fonctionnement ainsi qu'un échauffement extrême de celle-ci.

Pour les tests de détections de fuites sur les connexions, utilisez un détecteur de fuite électronique approprié ou une solution 50/50 d'propanol et d'eau. Il ne faut en aucun cas utiliser une solution savonneuse ou une solution semblable qui contaminera l'injecteur.

La boîte de commande de l'injecteur ne doit pas être allumée lorsque l'injecteur est déconnecté sous peine de grandement endommager l'appareil.

1.2 Mises en garde et précautions d'emploi concernant le piège cryogénique d' OPTIC 3

Les mises en garde et précautions d'emploi suivantes concernent le piège cryogénique dont sont équipés OPTIC 3-SC et OPTIC 3-DC.

1.2.1 Mises en garde concernant le piège cryogénique d'OPTIC 3

Le piège cryogénique utilise de l'azote liquide pour produire le gaz froid nécessaire aux opérations à basses températures. Le personnel devra connaître parfaitement les propriétés et les mesures de sécurité avant d'être autorisé à manipuler l'azote liquide et le piège cryogénique.

L'azote liquide peut causer de graves brûlures. Il faut donc porter des gants et des lunettes à pourtour étanche (et non pas de simples lunettes de protection) tout le temps qu'on utilise ce produit. Il ne faut jamais laisser une partie non protégée du corps entrer en contact avec des tuyaux non lés ou avec des éléments contenant des produits cryogéniques. La température extrêmement froide du métal fera que la peau y adhérera et se déchirera lorsqu'on tentera de se dégager.

Les dewars contenant de l'azote liquide devront être manipulés et entreposés en position verticale. Il ne faut pas laisser tomber les dewars ou les basculer sur le côté.

1.2.2 Précautions d'emploi concernant le piège cryogénique d'OPTIC 3

Assurez-vous que le gaz circule bien dans le serpentin de l'échangeur thermique du piège cryogénique lorsque vous l'immergez dans l'azote liquide ou lorsque vous le laissez longtemps dans le dewar. Ceci afin d'éviter que la vapeur d'eau présente dans l'air ne givre ce qui bloquerait la circulation du gaz dans l'échangeur thermique.

1. Advertencias y precauciones

En esta sección se explican las advertencias y precauciones que deben observarse a la hora de instalar o manipular el OPTIC 3.

A lo largo del manual existen distintos apartados que contienen información relacionada con la seguridad. Así, pues, le recomendamos que lea detenidamente todo el manual antes de instalar o manipular el instrumento. Si tras su lectura tuviese alguna duda, le rogamos que se ponga en contacto con su proveedor.

1.1 Advertencias y precauciones generales relacionadas con el OPTIC 3

OPTIC 3 está diseñado para que se instale y utilice en un laboratorio por personas convenientemente capacitadas. Antes de instalarlo o ponerlo en funcionamiento es imprescindible que se valoren los aspectos relativos a la seguridad en el contexto de las normas locales.

1.1.1 Avs generales en torno al OPTIC 3

OPTIC 3 debe utilizarse en laboratorios de análisis de sustancias químicas debidamente equipados y, en este sentido, es necesario tomar medidas para almacenar los dlventes en armarios refractarios y para que el personal lleve ropa protectora y gafas en todo momento.

Hasta que finalice la instalación es imprescindible desconectar de la toma eléctrica tanto el OPTIC 3 como el GC huésped. En cuanto al OPTIC 3 es necesario que reciba la corriente de una toma con conexión a tierra.

La unidad de control del OPTIC 3 contiene varias tensiones potencialmente peligrosas, por lo que antes de retirar la cubierta es necesario desconectar la unidad de control y también la fuente de alimentación eléctrica.

Para que el inyector funcione adecuadamente es necesario que se caliente rápidamente y alcance altas temperaturas. Se prestará mucha atención cuando se trabaje en la zona del inyector pues sus piezas podrían seguir estando calientes tiempo después de haber desconectado la unidad. Es especialmente importante asegurarse de que el inyector esté frío (por debajo de los 40°C) y desconectado antes de cambiar los revestimientos del inyector.

La mayoría de los dlventes orgánicos son relativamente tóxicos y es posible que salgan vapores por el orificio de escape. Se recomienda encarecidamente dirigir dichos vapores a una campana de humos o instalar un separador de carbono activo en el orificio. Por otro lado, conviene no restringir el flujo a través del orificio puesto que algo así podría interferir en los análisis.

Los controles de gases del OPTIC 3 no están pensados para trabajar con gases explosivos (aparte de hidrógeno), corrosivos o tóxicos.

La utilización de hidrógeno como gas portador de GC es, en principio, peligroso dada su explosividad. Se debe prestar sumo cuidado y atención cuando se utilice hidrógeno como gas portador de GC en un sistema GC/MS.

1.1.2 Precauciones generales OPTIC 3

Es importante que la conexión entre el cable eléctrico del inyector y el inyector sea buena, de lo contrario, el funcionamiento no sería óptimo y, además, la conexión podría calentarse en exceso.

Cuando se realicen pruebas de fugas en las conexiones se utilizará un detector de fugas electrónico adecuado o una solución al 50% de propanol y agua. En ningún caso se utilizará solución jabonosa o un producto similar ya que contaminaría el inyector.

No debe conectarse la caja de control del inyector cuando éste esté desconectado dado que se dañaría gravemente el instrumento.

1.2 Advertencias y precauciones relacionadas con el crioseparador OPTIC 3

Las advertencias y precauciones siguientes guardan relación con el crioseparador instalado en el OPTIC 3 y el OPTIC 3-DC.

1.2.1 Advertencias relacionadas con el crioseparador del OPTIC 3

El crioseparador utiliza nitrógeno liquido para producir el gas frío necesario para que el instrumento funcione a baja temperatura. Es esencial que el personal esté debidamente familiarizado con las propiedades y las consideraciones en torno a la seguridad antes de que manipulen el nitrógeno líquido y el crioseparador.

El nitrógeno líquido puede provocar graves quemaduras, de ahí que sea necesario portar en todo momento cuando se manipule gafas protectoras (no gafas de seguridad) y guantes. Nunca permitir que zonas desnudas del cuerpo entren en contacto con tuberías no aisladas o piezas que puedan contener productos criogénicos. El metal extremadamente frío hará que la piel se pegue rápidamente, rasgándola cuando se intente separarla.

Los frascos Dewar con nitrógeno liquido se manipularán y almacenarán en posición vertical. No verterlos ni volcarlos sobre los costados.

1.2.2 Precauciones relacionadas con el crioseparador del OPTIC 3

Es necesario asegurarse de que el gas esté fluyendo a través de la bobina del tubo del termointercambiador del crioseparador cuando se sumerja o saque del nitrógeno líquido. Con esta medida se pretende evitar que se congele el vapor de agua presente en el aire, lo cual bloquearía el flujo de gas a través del citado termointercambiador.

1. Avvertenze e precauzioni

In questo paragrafo vengono esposte le avvertenze e le precauzioni da osservare per l'istallazione e il funzionamento dell' OPTIC 3.

Lungo tutto il manuale vengono trattati gli aspetti essenziali dell'informazione per la sicurezza. Si prega gentilmente di leggere tutto il manuale prima di procedere all'istallazione e al funzionamento dell'apparecchio. Se qualche punto dovesse rimanere poco chiaro, si prega di prendere contatto col vostro fornitore per dei chiarimenti prima di procedere.

1.1 Avvertenze e precauzioni generali dell' OPTIC 3

L'OPTIC 3 é stato progettato per essere istallato e usato in un ambiente di laboratorio da personale adeguatamente preparato. Tutti gli aspetti relativi alla sicurezza devono essere in accordo alle norme locali prima che si proceda all'istallazione e alla commisione dell'apparecchio.

1.1.1 Avvertenze generali dell' OPTIC 3

L'OPTIC 3 é stato creato per essere usato in laboratori di analisi chimica appropriatamente equipaggiati. In particolare, devono essere presi dei provvedimenti riguardo al magazzinaggio dei solventi che vanno conservati in mobili anti-infiammabili e al personale che deve sempre indossare degli indumenti e degli occhiali di protezione.

Sia l'OPTIC 3 che l'host GC non devono essere collegati ai principali generatori di corrente fino a ultimazione dell'istallazione dell'apparecchiatura. L'OPTIC 3 deve essere alimentato da una presa interrata.

All'interno dell'unità di controllo dell'OPTIC 3 sono presenti dei voltaggi potenzialmente pericolosi. Prima di rimuovere il coperchio dell'unità di controllo spegnere l'unità di controllo e disinserire i principali alimentatori di corrente.

La messa in funzione dell'iniettore richiede un riscaldamento rapido fino al raggiungimento di temperature elevate. Parti dell'iniettore potrebbero rimanere calde anche molto tempo dopo lo spegnimento dell'unità. Bgna procedere con cautela quando si lavora attorno all'area dell'iniettore. Prima del cambio dei rivestimenti dell'iniettore (liners) bgna assicurarsi che questi si sia raffreddato (al di sotto dei 40°C) e che sia spento.

La maggior parte dei solventi organici sono relativamente tossici e una notevole quantità di questi vapori potrebbero fuoriuscire dall'orificio di scarico. Si consiglia vivamente di canalizzare questi vapori in un cappuccio del vapore, oppure di

applicare una presa del carbonio attivo all'orificio di scarico. Ad ogni modo il flusso attraverso l'orificio di scarico non deve essere impedito in quanto cio potrebbe interferire con le analisi.

I sistemi di controllo dei gas dell'OPITIC 3 non sono stati progettati per procedimenti con esplosivi (eccetto l'idrogeno), gas corrosivi o tossici.

L'uso dell'idrogeno come gas trasportatore GC é pericoloso in quanto per sua natura potenzialmente esplosivo. Procedere con estrema cautela durante l'uso dell'idrogeno come gas trasportatore GC in un sistema GC o GC/MS.

1.1.2 Precauzioni generali dell' OPTIC 3

E' importante collegare bene il cavo elettrico dell'iniettore all'iniettore. Un collegamento sbagliato puo causarne il cattivo funzionamento e un surriscladamento dello stesso collegamento.

Quando si effettua una prova per la rilevazione di perdite dei collegamenti, fare uso di un rivelatore di perdite elettronico oppure di una soluzione al 50/50 di -propanolo e acqua. In nessun caso va usata una soluzione detergente o dello stesso tipo in quanto cio contaminerebbe l'iniettore.

La cassetta di controllo dell'iniettore non deve essere accesa quando l'iniettore é staccato in quanto cio potrebbe danneggiare seriamente la strumentazione.

1.2 Avvertenze e precauzioni relative al cryotrap dell'OPTIC 3

Le seguenti avvertenze e precauzioni riguardano il cryotrap inserito all'OPTIC 3-SC e all'OPTIC 3-DC.

1.2.1 Avvertenze relative al cryotrap dell'OPTIC 3

Il cryotrap fa uso di azoto liquido per la produzione di gas freddo per operazioni a basse temperature. Il personale addetto deve essere a conoscenza delle proprietà e delle considerazioni di sicurezza prima di essere autorizzato all'uso dell'azoto liquido e del cryotrap.

L'azoto liquido puo causare delle gravi bruciature. Indossare sempre delle protezioni per le mani e degli occhiali di protezione (non usare degli occhiali di sicurezza) durante il suo maneggiamento. Non permettere a nessuna parte del corpo non protetta di entrare in contatto con la tubazione non lata o delle parti che contengano prodotti criogenici. La temperatura estremamente fredda del metallo causa l'aderenza immediata della pelle ad esso e lo strappo nel tentativo di rimuoverla.

I dewars di azoto liquido dovrebbero essere maneggiati e conservati in posizione eretta. Non lasciar cadere i dewars o inclinarli di lato.

1.2.2 Precauzioni relative all'OPTIC 3 cryotrap

Assicurarsi che vi sia del gas che scorre lungo la bobina del tubo dello scambiatore di calore del cryotrap quando lo si immerge nell'azoto liquido o lo si lascia nel dewar per

molto tempo. Tale precauzione va presa per evitare il congelamento del vapore acqueo presente nell'aria, che bloccherebbe la fuoriuscita del gas attraverso lo scambiatore di calore.

2. System overview

Most of the gas chromatographs (GC) are fitted with basic injectors offering few operating features. OPTIC 3 is a highly advanced injector with sophisticated temperature and gas flow control that can be used for the most demanding GC analyses.

A standard OPTIC 3 system consists of the injector, the control unit and the control software. The control software has to be installed on a PC running MS Windows 98 (SE), NT, 2000, XP or Vista.

OPTIC 3 can be delivered in one of the five configurations. The basic model is OPTIC 3-S. OPTIC 3-D has an auxiliary gas control channel. OPTIC 3-SC and OPTIC 3-DC feature a cryotrap, which is covered by a separate section of this manual. OPTIC 3-PTV is a simplified version of OPTIC 3-S, delivered without any gas control channel.

The main part of this guide contains information, which you are likely to refer to regularly. Information on setting up the system and other information which is only occasionally referred to is included in the appendices.

2.1 High Performance

OPTIC 3, when correctly set up, offers a minimum of sample discrimination. Reproducibility is comparable to on-column injection, but offers excellent tolerance of "dirty" samples. Thermal degradation is less severe than with conventional hot split and splitless injectors and the analyst has much greater control over the way in which the sample is introduced into the gas chromatograph.

2.2 Advanced Gas Control

One of the features that sets OPTIC 3 apart from other injectors is its sophisticated gas control system. OPTIC 3-S and OPTIC 3-SC have main channel gas control only, while OPTIC 3-D and OPTIC 3-DC also have an auxiliary gas channel.

Main gas channel has a flow sensor and electronic flow controller to control the total carrier gas through the system. It also has a pressure controller to control the injector pressure. Together, they are used to control the split flow and the column flow. Since technically it is extremely difficult to control the column flow directly, it is programmed using the pressure controller. This is based on the measured injector pressure, carrier gas properties, column parameters, and GC oven temperature. Unlike many other injector systems, OPTIC 3 has its own GC oven temperature sensor to increase the accuracy of the flow calculation.

Auxiliary gas channel can be controlled on the basis of flow or pressure in a part of the system selected by the user.

2.3 Improved Flexibility

OPTIC 3 can be used for hot-split, hot-splitless, cold-split, cold-splitless and on-column injections. These techniques are discussed in greater details in Section 5 "Defining and Running Methods". It also offers features like the patented At-Column injection mode,

which are not provided by conventional injectors. Facilities such as large volume sampling, thermal desorption, and pyrolysis are standard features.

OPTIC 3 offers far greater control over the fate of the sample in the injector than conventional systems. When the sample is injected, the analyst can select the temperature, the column flow and the split flow. It is even possible to inject under static gas flow conditions. Consequently, it is possible to develop many novel approaches to sample injection. OPTIC 3 also features interfaces for the GC and autosampler and auxiliary connections for interfacing to other equipment.

2.4 Compatibility

OPTIC 3 is designed to be compatible with most popular makes and models of GC and GC-MS. Once installed, OPTIC 3 should be fully compatible with an auto-sampler. Please visit our web site at www.atasgl.com or contact your local OPTIC 3 distributor if you have any questions concerning compatibility.

The interfaces are discussed in greater detail in the hardware and configuration sections of this manual, and the appendices.

3. Hardware

The OPTIC 3 hardware consists of a bench-top control unit, the injector and optional cryogenic trap that are installed on the host gas chromatograph.

3.1 Control Unit

The control unit contains a microprocessor, injector temperature and gas flow control systems, user interface (keypad and display), power supply, interfaces to the PC, GC, autosampler and auxiliary equipment, and optional cryotrap control (OPTIC 3-SC and 3-DC only).

3.1.1 Keypad

OPTIC 3 has a multi-functional keypad on the front panel of the unit. The eight keys are used for local control (running methods, and setting certain parameters). More extensive control is provided by the Evolution Workstation software running on a host PC.

A diagram of the front panel is shown in Figure 3.1.

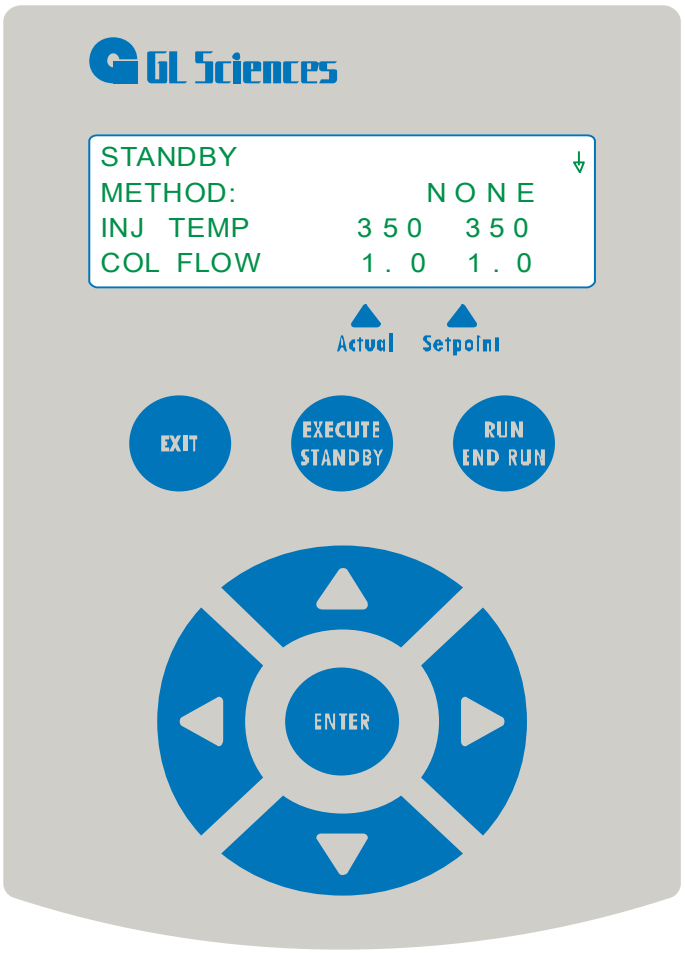


Figure 3.1 Front panel

3.1.2 Local Display

Information is presented to the user on a 4-line LCD display above the keypad. A typical display example is shown in Figure 3.1.

3.1.3 Rear panel

The rear panel (Figure 3.2) provides gas and electrical connections for the injector, PC, host GC, autosampler, optional auxiliary equipment and mains power. The mains inlet is integrated with the mains fuse.
The "**Carrier In**" coupling is connected to the carrier gas supply. The "**Carrier Out**", and "**Split**" couplings are connected to the corresponding lines of the injector.

The "**Aux Gas In/Purge In**", "**Aux Gas Out/Purge Out**" couplings on the OPTIC 3-D and OPTIC 3-DC are used for the second (auxiliary) gas channel. This channel can be used, for example, for septum purge flow control. "**Aux Gas In/Purge In**" should be connected to the septum purge line of the injector in this case.

The "**Air In**" coupling is connected to the cooling air supply for the injector. The "**To Injector**" coupling is connected to the cooling pipe of the injector, using the PTFE tubing supplied.

The "**Exhaust**" coupling is the vent from the gas control system and should be left open. Alternatively, the exhaust flow can be ducted to an extraction system, but this should not restrict the flow.

OPTIC 3-SC and OPTIC 3-DC have a "**Cryo Gas In**" coupling for the cooling gas supply, and a "**Cryo Gas Out**" coupling to connect it to the cryotrap. "**Cryotrap Thermocouple**" and "**Cryotrap Heater**" connections are used to connect the trap to the control unit.

The "**Injector Thermocouple"**, "**Injector Heater**" and "**Solvent Monitor**" connections are used to connect the control unit to the injector.

The "**GC Oven Thermocouple**" connection is used for the thermocouple (supplied with OPTIC 3) to measure the GC oven temperature.

The OPTIC 3 control unit communicates with the PC running the control software using either a serial or LAN (Ethernet) connection, as selected in the Configuration Menu accessed from the control unit keypad or the control software (see Appendix B "Software Installation and Configuration" and Appendix C "Local Control Menus").

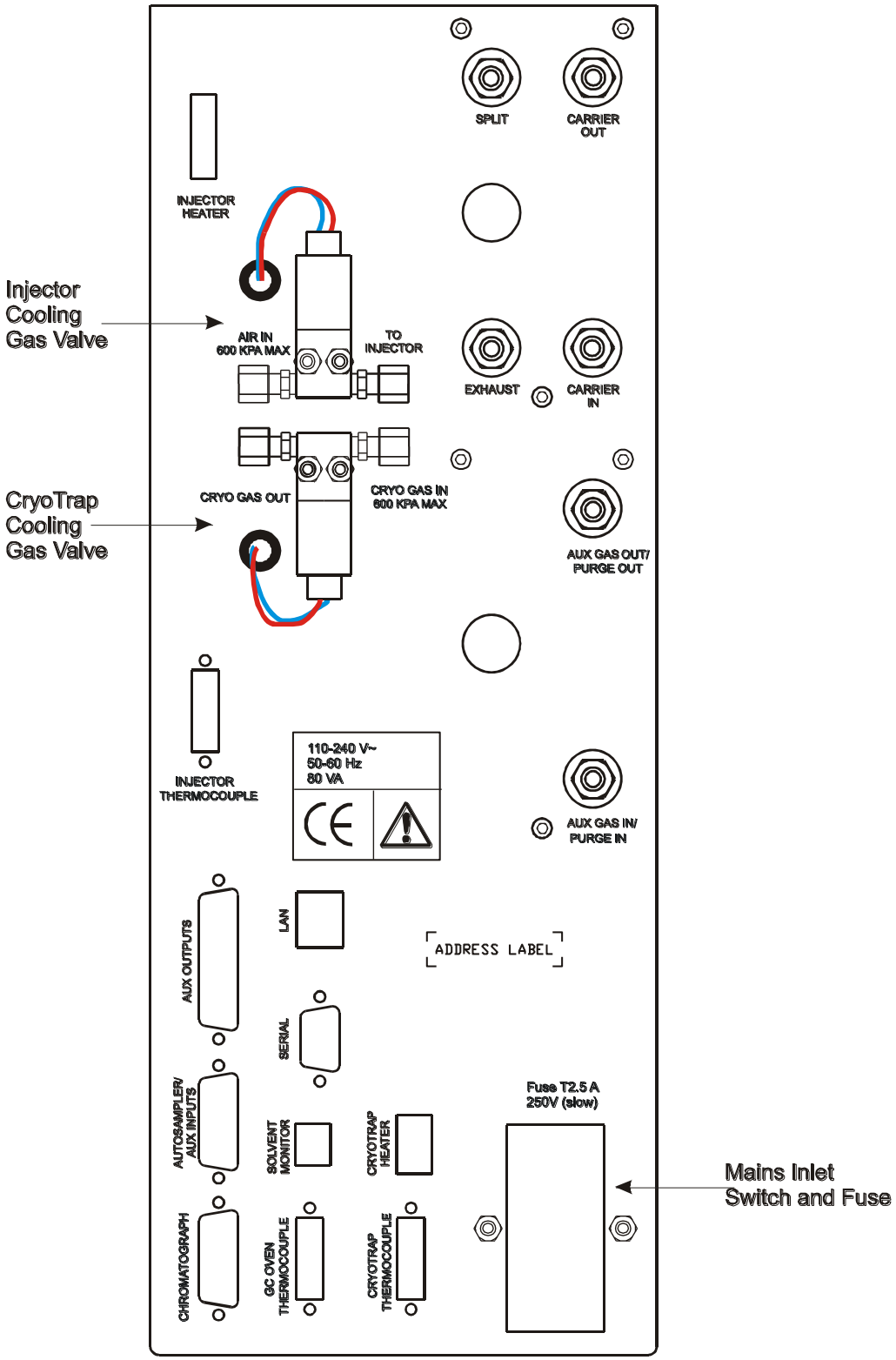


Figure 3.2 OPTIC 3-DC rear panel. Only the D-models have auxiliary gas connections. Only the C-models have cryotrap connections.

There are also connections for the gas chromatograph and autosampler, and auxiliary inputs and outputs. These are configured in the **Configuration Menu** and **I/O Menu** accessed from the control unit keypad or with the control software on the PC (see Appendix C "Local Control Menus", and the help file for the control software). The pin-outs of the connectors are described in Appendix E "Rear Panel Connections".

3.1.4 Gas Control System

Figure 3.3 shows a schematic diagram of the OPTIC 3 gas control system. Only OPTIC 3-D and OPTIC 3-DC have the auxiliary gas control system.

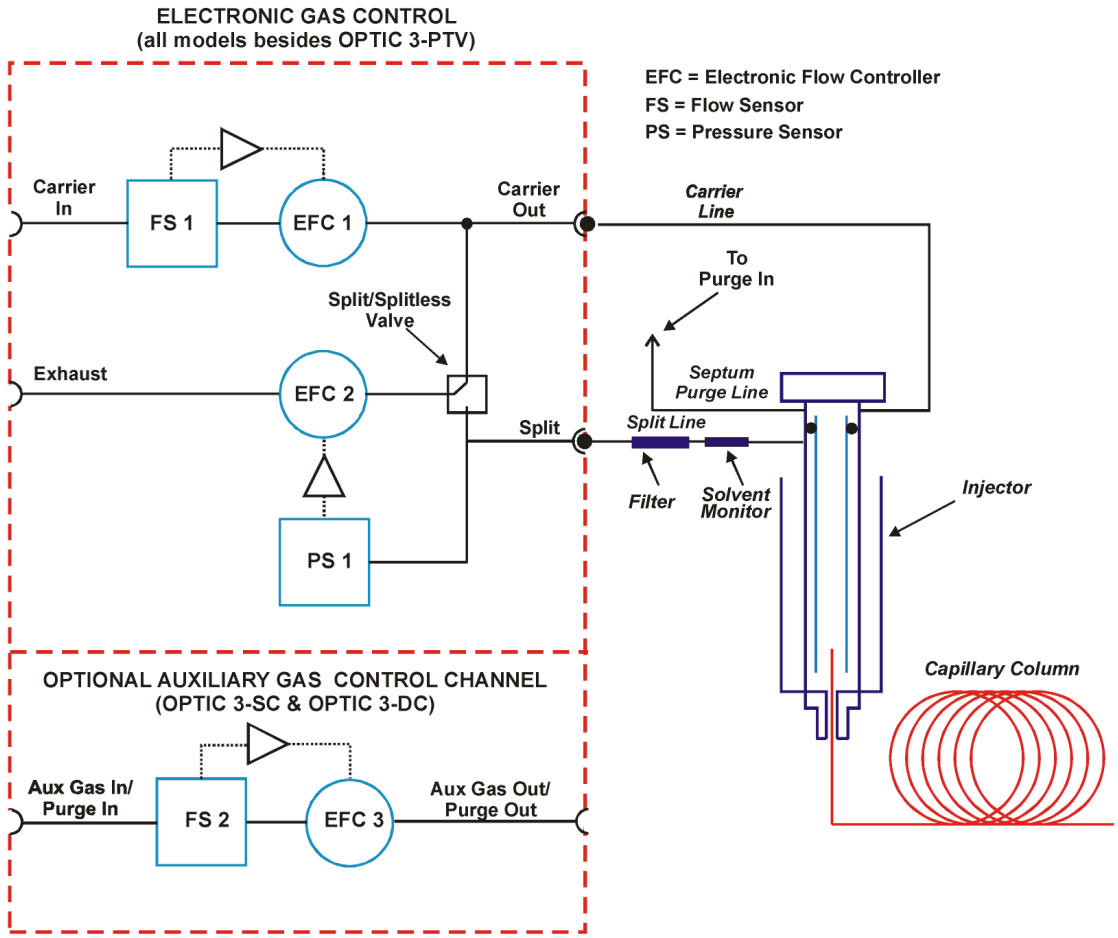
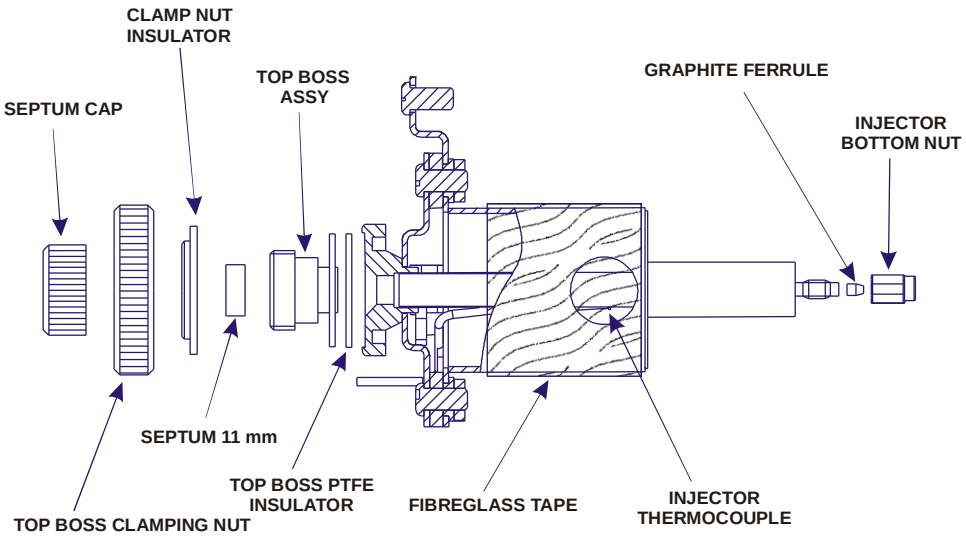


Figure 3.3 OPTIC 3 Gas Control System

3.2 Injector Hardware

The OPTIC 3 injector assembly is installed on the host GC and is connected to the control unit by gas lines and electrical cables. The installation of the injector on the host GC is described in Appendix A "Hardware Installation", and Figure 3.4 shows the details of the injector.

Side view



Top view

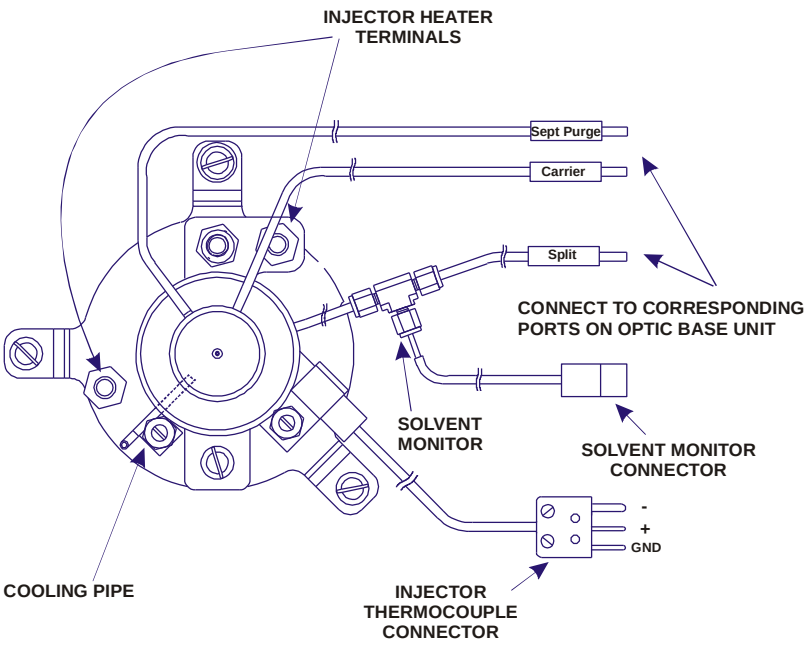


Figure 3.4 Injector diagram

3.3 Cryogenic Trap (optional)

The OPTIC 3 cryogenic trap, supplied with OPTIC 3-SC and OPTIC 3-DC, comprises the trap, the heat exchanger, and the liquid Nitrogen Dewar. The trap is installed in the oven of the host GC and is connected to the control unit with gas lines and electrical cables. The details of the cryotrap hardware and its installation are described in the separate section of the manual supplied with the trap.

3.4 Local Control and Menus

OPTIC 3 is locally controlled through a menu structure. For more extensive control and defining methods, the Evolution Workstation software running on a PC connected to the OPTIC 3 control unit is required.

The keypad on the OPTIC 3 control unit provides basic control to:

- Access the basic functions and configuration;
- Run methods downloaded from the PC;
- Define and run sequences.

When power is applied to OPTIC 3, a system initialization sequence is performed. During this time the LCD display will read: **Initializing OPTIC 3**.

On completion of the initialisation sequence, the "Standby Menu" option of the Top Level Menu is displayed. The Top Level Menu contains the following options:

- Standby Menu: standby parameter configuration;
- Method Menu: selecting and running one of up to 10 methods uploaded from the PC where they were defined with the control software;
- Sequence Menu: selecting and defining up to 3 sequences containing the uploaded methods;
- Configure Menu: general instrument and PC communication configuration;
- I/O Menu: GC and autosampler interface input/output configuration;
- Column Parameters Menu: setting the GC column parameters, carrier gas and column outlet pressure.

3.4.1 OPTIC 3 Keypad

The eight keys of the OPTIC 3 local keypad perform the following functions:

- Enter: enter the lower level menu, confirm the reset of parameters;
- Exit: accept the current entry and return to the higher level menu;
- Execute/Standby: start or interrupt method;
- Run/End Run: run or stop method;
- Left/Right: select menus or menu items;
- Up/Down: select the required value of the parameter.

There is no cancel function, so if you do not want to enter a new value, you have to use the up/down cursor keys to restore the previous value before exiting.

3.4.2 Status display

The highest level of the OPTIC 3 local menu structure is a status display. It can be accessed from any level by pressing 'Exit' repeatedly. To return to the control menus from the status display, press 'Enter', after which you can use the left/right buttons to select the required menu.

The status display displays the actual values and set points of the following parameters:

- Line 1: Instrument status (Standby, Equilibrating, Wait for Ready In, Wait for Run In, Running OPTIC Method, Running GC & OPTIC Methods);
- Line 2: Active method;
- Line 3: Injector temperature;
- Line 4: Column flow;
- Line 5: Injector pressure;
- Line 6: Split flow;
- Line 7: Solvent monitor reading;
- Line 8: Auxiliary flow (if enabled);
- Line 9: Cryotrap temperature (if enabled).

Only four first lines are displayed on the status display when it is first accessed. Press the 'Up/Down' keys to see lines 5 to 9.

3.4.3 Control menus

The following top-level control menus are available: Standby, Method, Sequence, Configuration, and Input/Output.

Standby Menu

The Standby menu is used to enter parameters such as timeout, injector temperature, column flow, split flow and others (see the complete list of parameters in Appendix D "OPTIC 3 Default Parameters") which are set when OPTIC 3 goes into the standby state.

Method Menu

The Method menu is used to select a method stored in OPTIC 3 internal memory. Pressing the "Execute" button can then start the method.

Sequence Menu

The Sequence menu is used to define and activate sequences (a sequence is a series of methods previously downloaded from the PC and stored in the OPTIC 3 memory).

Configuration Menu

The Configuration menu is used to define general system parameters, including communication with the PC running the Evolution Workstation software.

I/O Menu

The Input/Output menu is used to define the communication with the host GC, autosampler and any auxiliary equipment.

Column Parameters Menu

The Column Parameters menu is used to define the GC column parameters like internal diameter and column length and also to set the carrier gas type and column outlet pressure.

The local control menu structure is described in detail in Appendix C "OPTIC 3 Local Menus".

3.4.4 Master/Slave Mode

Depending on the way it is connected to the GC and autosampler, OPTIC 3 can be set to act as the system master or system slave in the I/O menu.

OPTIC 3 Master

When OPTIC 3 is configured as the master then once the initial conditions of the method are reached, it will wait for a "Ready In" (1) signal from the GC (Figure 3.5).

When the "Ready In" signal is received, OPTIC 3 waits for the equilibration time to elapse and then sends the "Ready Out" (2) signal to the autosampler. After the injection is completed, OPTIC receives a "Run In" (3) signal from the autosampler and OPTIC then sends "Run Out" (4) to the GC and performs the actions defined in the method profile.

Figure 3.5 OPTIC 3 state diagram in master mode

The operator can manually give the "Run In" signal from the keypad or control software at any time, in which case OPTIC ends all current actions and starts the method run.

OPTIC 3 Slave

When OPTIC 3 is configured as the slave then once the initial conditions of the method are reached, it does not wait for a "Ready In" signal from the GC (Figure 3.6) but immediately enters the equilibration state.

Figure 3.6 OPTIC 3 state diagram in slave mode

When the equilibration time has elapsed, OPTIC sets the "Ready Out" (1) signal to the GC. After the injection is completed, OPTIC receives a "Run In" (4) signal from the GC (autosampler) and performs the actions defined in the method profile.

3.5 Remote Control from PC

OPTIC 3 can be controlled remotely from a PC using the Evolution Workstation software supplied with the instrument. Furthermore, methods can only be defined using this software, and then be transferred to the OPTIC 3 control unit where they may then be activated locally.

Note: EZChrom Elite OPTIC 3 Control module can also be used for the remote instrument control. For more details please visit www.atasgl.com

4. Defining and Running Methods

4.1 Introduction

OPTIC 3 methods are defined using the Evolution Workstation software running on a PC connected to the OPTIC 3 control unit. They can then be initiated from the PC. Alternatively, up to ten methods can be uploaded to the control unit where they can then be initiated under local control, without a further need for the connection to the PC.

See Evolution Workstation on-line help for more information about defining and running OPTIC 3 methods.

4.2 Method Types

OPTIC 3 supports the following methods:

- Split
- Splitless
- Large Volume Injection
- LINEX-TD (Thermal Desorption)
- LINEX-DMI (Difficult Matrix Introduction)
- At-Column
- Hot Split
- Hot Splitless
- Standard TD (Thermal Desorption)
- Expert (full control of all parameters including auxiliary inputs)

See Appendix G "Examples of OPTIC 3 Method Profiles" for method diagrams.

4.2.1 Split Method

OPTIC 3 can be used as a conventional hot split injector (Hot Spit method) but also for cold split injection (Split method). In virtually all cases, better results will be obtained using the cold split approach. This means starting with the injector cold (i.e. below the boiling point of the solvent) and temperature programming the injector to a temperature usually 50 ºC above the maximum elution temperature of the sample. This has the following advantages:

- Syringe needle discrimination is eliminated, thus ensuring that the fraction of the sample, which enters the column, is truly representative of the sample as a whole.

- The milder temperature regime experienced by the sample reduces thermal degradation (especially when lower injector temperature ramp rates are used).

- Better run-to-run reproducibility.

4.2.2 Splitless Method

OPTIC 3 can be used for conventional hot splitless injection (Hot Splitless method) or for the cold equivalent (Splitless method). In virtually all cases, better results will be obtained using the cold injection technique for reasons identical to those described above. The quality of results obtained should be comparable to on-column injection in all respects but with the added advantage of preventing involatile and particulate matter from reaching the column. Cold splitless injection is therefore better suited to the analysis of "dirty" samples.

4.2.3 Large Volume Injection Method

In the past, analysts have been used to injecting samples of no more than 2 μl into the GC. Since split/splitless injectors generally have an internal capacity of approximately 2 ml, and 1 μl of solvent gives rise to approximately 1 ml of vapour, the total amount of sample that can be injected into a split or splitless injector is around 2 μl.

When an injection is made into a temperature programmable injector, the evaporation of the solvent is a controllable process and therefore restrictions on the volume of solvent that can be injected are much less severe.

The OPTIC 3 large volume method is designed to implement the rapid large volume technique. This enables injections up to 150 μl to be made routinely, at-once, and with a minimum of optimization. The technique is compatible with most auto-samplers and is very robust, making it suitable for routine applications. Other large volume sampling techniques, such as speed programmed injection, may also be accommodated.

In large volume injection, the sample is introduced with the injector at near ambient temperature. The solvent is allowed to evaporate into the carrier stream and pass out through the split vent. At an appropriate point, the split vent is closed and the injector heated. The sample is then transferred to the column. Simple optimisation of the parameters enables components with a very wide volatility range to be analysed by this technique.

The advantages of this method are very significant:

- An improvement in detection limits of up to 100 times that of conventional splitless injection.

- Much greater flexibility in the design of sample preparation procedures.

- Easier on-line interfacing with sample preparation accessories (such as automated solid phase extraction instruments).

4.2.4 LINEX-TD and LINEX-DMI Methods

OPTIC 3 can be used for the thermal desorption of gas and solid samples (LINEX-TD) and also real world samples with complex dirty matrixes (LINEX-DMI).

When analyzing the gas phase samples, the samples can be collected off-line using ATAS GL TD liners or adsorption tubes packed with a suitable sorbent. Alternatively, on-line analysis is possible with the sample being introduced into the liner while it is still within the injector.

DMI enables sample introduction into GC column to be performed from a disposable container (microvial) placed inside the injection liner. This has a great advantage over traditional injection because large volumes (up to 30 µl) of dirty sample extracts or even row samples can be introduced directly into GC or GC/MS. Using the principle of selective exclusion, the volatiles and semi-volatiles are thermally desorbed and transferred onto the column, while non-volatile residues are retained in the microvial, which is disposed after analysis. The DMI method permits also the use of the built-in solvent monitor, facilitating the optimization of the solvent vent time.

Thermal desorption using the OPTIC 3 TD or DMI methods is distinguished from conventional thermal desorption by the way the liner with the sample or the adsorption tube is closely coupled to the column. This has the following advantages:

- The analysis of very active components is possible since the opportunities for adsorptive losses are greatly reduced.

- The analysis of components with a very high boiling point (such as polymer additives) is possible.

- The very short time-cycle associated with desorption makes it possible to obtain results quickly.

4.2.5 At-Column Method

At-Column is a new large volume concentrating technique, which is a standard feature of OPTIC 3. It uses a special, open (not-packed) liner, which means that the sensitive compounds of interest are not decomposed or strongly retained in the injector. Instead, the solvent is evaporated in the liner and the target compounds are concentrated at the inlet of the capillary column. Consequently, there is no need for a long pre-column or accurate control of the injection rate.

The concept behind the At-Column method is to create a liner temperature gradient around the solvent boiling point. As a result, the solvent stops flowing through the liner at the point where the carrier pressure is equal to the solvent vapor pressure. To create such a gradient, the injector temperature is set **below** the solvent boiling point and the GC oven temperature is set **above** the solvent boiling point. The outlet of the liner is connected to the capillary column by a short capillary of de-activated fused silica (retention gap). To vent the evaporated solvent to the split line, the liner has a small hole in its wall. The position of the hole is such that a sufficient amount of the sample can be injected. To prevent the solvent flowing into the capillary column, a glass bead is placed at the bottom of the liner.

The OPTIC 3 At-Column method has a built-in calculator, which facilitates the optimisation of the method parameters. When the initial injector pressure and the solvent are entered, the calculator automatically sets the initial injector temperature

and suggests the initial GC oven temperature. If necessary, the initial injector temperature can be manually set to a different value.

For more details about the At-Column method please visit www.atasgl.com

4.2.6 Expert Method

The six modes of operation described above are designed to cover the most common ways in which OPTIC 3 will be used. The Expert mode is designed to cater for the many other possibilities.

In an expert method, up to nine segments for the injector temperature profile can be programmed including segments with a negative temperature ramp when the injector heater is switched off and cooling is on. Up to nine segments for column flow as well as split flow profiles can be set. In addition, seven external channels of timed events can be programmed in any sequence.

Flow or pressure control of an auxiliary gas supply channel is also possible.

Some of the special features of these methods are introduced in Section 6 "Advanced OPTIC 3 features".

4.3 Method Definition

To define a method:

- Select **New** from the **File** menu.
- Select **Method** from the list box and press **OK**.
- Select the required method type and press **OK**.
- Set up the method parameters within the method window.
- Save the method for the later use or execute it by selecting **Method - Execute**. A method that is activated can be edited, but the changes will not have any effect on the running method. To use the changed method, execute it again.

When saving a method, you can give it a name of at most 11 characters. By default, methods are saved in the 'Methods' subdirectory of the Evolution Workstation directory.

Method parameters can be changed in the parameter list and in the graph. The values of some parameters depend on the value of other parameters, e.g. the value of the **Final Injector Temperature** cannot be less than the value of the **Initial Injector Temperature** and in the Split method the **End Time** cannot be less than the **Transfer Time**.

Note that times must be entered in seconds and are displayed in min:sec format in the graph.

Parameters can be changed in the graph by dragging the control points. Some points, e.g. the end points, cannot be dragged, and some can only be dragged vertically or horizontally. While dragging the parameter value in the parameter list will be updated.

Note: When a parameter is selected in the Parameter View, the background of the name of the parameter is greyed. Even though the cursor is not visible, values can be entered. To confirm the value, press 'Tab' or 'Enter'. In order to restore the old value press 'ESC'. Changing drop-down list box values can be done with the 'Arrow Right' and 'Arrow Left' keys.

Evolution Workstation has an extensive Help function, which includes much of the information contained in this section of the manual.

4.4 Method Parameters

The functions of all parameters are explained below. When a new method is created, the parameter tables are filled with the default parameters. The default parameters are also listed separately for each method type in Appendix D "OPTIC 3 Default Parameters". The parameter tables for each method only include the parameters relevant to that method.

Note: This section of the manual addresses only method parameters. The general configuration and standby parameters of OPTIC 3 are discussed in Appendix B "Software Installation and Configuration".

4.4.1 General Method Parameters

Method Name

This parameter identifies the method. The user enters it when the method is first saved. Until the user enters a name, Evolution Workstation will use a temporary name (e.g. Method1). A method name can be at most 11 characters long.

Equilibration Time

This parameter is used to ensure the stability of the initial method conditions prior to OPTIC 3 entering the **Ready** state.

End Time

This parameter determines the time between the start and end of the GC method run. It should be set to the total run-time for the GC analysis, so that the cooling of the injector and resetting of column and split flow parameters coincide with the end of the GC run.

4.4.2 Temperature Parameters

Initial Temperature

This parameter determines the injector temperature at the start of the method run.

Ramp Rate

This parameter determines the rate of increase of the injector temperature.

Final Temperature

This parameter determines the injector temperature at the end of the temperature ramp.

Hold Time

Hold Time is time, during which the injector temperature is maintained constant. If the Temperature Control is set to 'Floating' then the injector temperature stops being controlled at the end of the thermal segment.

Temperature Control

This parameter determines if the injector temperature is maintained to the end, or it is allowed to float at the end of the Hold Time.

Solvent Cooling Effect

This parameter determines whether or not a solvent cooling effect (rapid cooling during the solvent evaporation) should be used during solvent elimination (Expert Method only). For more information about this feature you are referred to Section 6.2 "Solvent cooling effect".

Cooling Valve Mode

This parameter determines if injector cooling is switched on or off during the method run time (Expert Method only). When it is set to "No", cooling is switched off and the injector temperature is maintained by the injector heater controller.

4.4.3 Column Flow Parameters

Carrier Control Mode

This parameter determines the carrier gas control mode. When it is set to "Pressure", the carrier gas flow through the column is controlled using the injector pressure as a parameter. In case of "Flow", the column volumetric flow is used as the main parameter to determine the carrier gas flow.

Transfer Time

This parameter determines the time during which the transfer flow is maintained after the start of the method run.

Transfer Column Flow/Injector Pressure

This parameter enables the user to set the column flow (injector pressure) used to transfer the sample from the injector onto the column (a higher flow (injector pressure) gives a faster transfer).

Start Column Flow/Injector Pressure

This parameter determines the carrier gas flow (injector pressure) after the transfer time has elapsed, i.e. the column flow (injector pressure) at the start of the actual analysis segment.

End Column Flow/Injector Pressure

This parameter determines the carrier gas flow (injector pressure) at the end of the method run.

Sample Sweep Column Flow/Injector Pressure

This parameter determines the column flow (injector pressure) used to sweep the sample with carrier gas prior to desorption (LINEX-TD and LINEX-DMI) or transfer (LVI).

Desorption Column Flow/Injector Pressure

This parameter determines the flow during desorption time in LINEX-TD and LINEX-DMI methods.

Desorption Time

This parameter determines the length of the desorption segment.

4.4.4 Solvent Venting Parameters

Vent Mode

This parameter determines whether solvent venting is carried out during a user-specified fixed time or using the built-in solvent monitor (SM). The solvent monitor vent time control can be based on the absolute (SM Threshold, 0 - 4095 arbitrary units) or relative (SM Level, 1 - 90%) output signal. Both solvent monitor output modes are discussed in greater detail in Section 6.1 "Vent mode and solvent monitor".

Vent Time

In the Fixed Time mode, this parameter determines the vent time during split injection.

Solvent Monitor Threshold

In the absolute vent mode, this parameter determines the solvent monitor level (expressed in arbitrary units) below which split valve closes.

Solvent Monitor Level

In the relative vent mode, this parameter determines the solvent monitor threshold relative to the measured maximum of the SM signal (expressed in %) below which split valve closes.

4.4.5 Split Flow Parameters

Vent Flow

This parameter determines the split flow during solvent venting.

Split Flow

This parameter determines the split flow after a sample transfer. It can be set lower than the Split Flow to reduce the carrier gas use.

Septum Purge Flow

This parameter determines the septum purge flow. It is only available if the auxiliary gas channel is installed (OPTIC 3-D and OPTIC 3-DC) and is designated as the septum purge option in System Configuration.

4.4.6 At-Column Calculator

Evolution Workstation offers the possibility to optimise some of the parameters of an At-Column method. After you choose the solvent and initial injector pressure, the software will set the initial injector temperature and suggest the initial GC oven temperature. You can then modify the initial injector temperature if required. For further details you are referred to section 4.2.5 "At-Column method".

Initial Injector Pressure

This parameter determines the injector pressure when eliminating solvent in an At-column method.

Solvent

This drop-down box gives you a choice of a number of solvents commonly used in gas chromatography.

4.4.7 Auxiliary Outputs and Inputs

Any of the auxiliary outputs can be used in any of the seven OPTIC 3 method types. Each output can switch at nine user-specified times during a method run. Furthermore, in an Expert method, the auxiliary outputs can be controlled by the state of the auxiliary inputs.

To keep the Evolution Workstation screens uncluttered, the auxiliary outputs are only shown in the method parameter tables if they have first been selected in **Configuration - System configuration - Auxiliary Outputs**.

The four auxiliary inputs can only be used in Expert methods. These inputs can only be used if they have first been enabled by selecting **Configuration - System Configuration - Auxiliary Inputs**.

4.4.8 Expert Method Parameters

Expert methods allow the user to set the profiles for any method parameter with up to nine individually configured segments. This makes it possible to develop advanced temperature and gas flow profiles.

*Note: The profiles in the Expert method could contain up to nine time segments. To add a time segment, click the right mouse button pointing on the profile line where the segment should start. Select **Add Step**, the segment will be added the graph and the parameters in the parameter list will be updated. To remove a segment, press the right mouse button and select **Remove Step**.*

The relevant parameters are:

- **Injector Temperature:** Delay Time (the time delay used to ensure the equilibration of the gas flow parameters due to a rapid injector pressure change after the injection); Ramp Rate 1 - 9; Hold Temperature 1 - 9; Hold Time 1 – 9. Note that both positive and negative temperature ramps can be set (see also Section 6.4 "Negative temperature ramps in Expert Methods").

- **Column Flow:** Start Column Flow 1 - 9; Column Flow Time 1 - 9; End Column Flow 1 – 9;

- **Split Flow:** Initial Split Flow; Split Flow 1 - 9; Split Flow Time 1 – 9.

- **Auxiliary Flow/Pressure:** Start Auxiliary Flow/Pressure 1-9, Auxiliary Gas Control Time 1-9, End Auxiliary Flow/Pressure 1-9.

Any of the auxiliary outputs can be used in an Expert method. Each output can switch at nine user-specified times during a method run. Furthermore, an Expert method can use auxiliary inputs 1 - 4 to control auxiliary outputs, the 'Run ' and 'Ready' outputs to the GC, and the 'Ready' output to the autosampler.

4.5 Performing a Method

Figure 4.1 illustrates the states that OPTIC 3 goes through as it prepares to run a method.

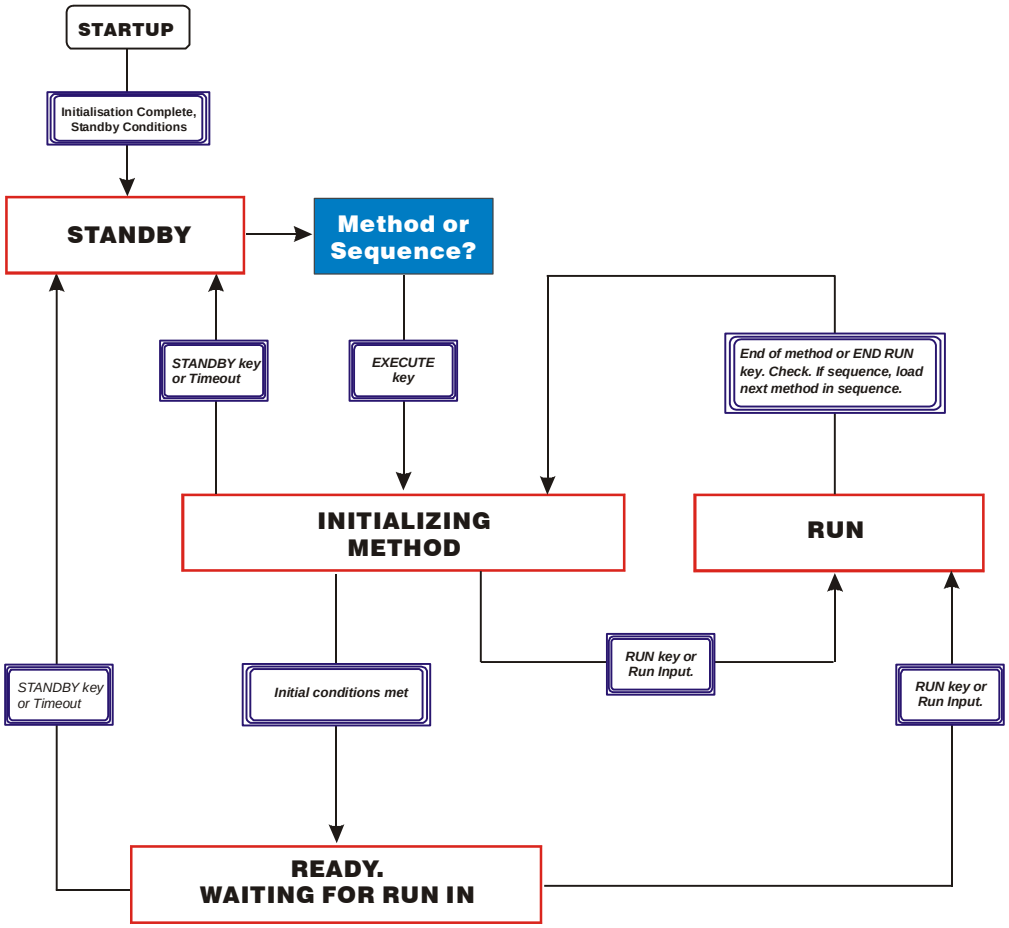


Figure 4.1 OPTIC 3 state transition diagram

When you select **File - New - Method** - **[method type]** the relevant parameters and their default values will be displayed for the selected method type. You can now adjust the parameters as required and save the method with a name of at most 11 characters.

4.5.1 Running Method from Evolution Workstation

Once a method has been saved, you can select **Method - Execute** to send it to the OPTIC 3 control unit. It will start the method running on OPTIC 3. If you use manual injection, wait for OPTIC 3 to enter the **Waiting for Run In** state, then inject the sample

and select **Method - Run**. The Evolution Workstation status graph will reflect the instrument parameters when the state changes to **Equilibrating**.

4.5.2 Stopping Method from Evolution Workstation

While the Instrument is in the **Initialize Method, Waiting for Ready In, Equilibrating** or **Waiting for Run In** state the method can be stopped by selecting **Method - Standby**. The Instrument will return to the Standby state. If the Instrument is in the **Running OPTIC Method** or the **Running OPTIC & GC Methods** state, the current run can be ended by selecting **Method - End Run**. The current run is stopped and the Instrument will return to the **Initialize Method** state and starts a new run with the same method.

4.5.3 Uploading a Method to OPTIC 3

Ten methods can be uploaded to the OPTIC 3 where they can be run and included in sequences defined on the control unit. Methods uploaded to the instrument and sequences created locally cannot be activated from the control software, but only from the control unit keypad.

To upload the method to OPTIC 3, select **File - Open**, then select **Method - Upload** to transfer it to the control unit.

4.5.4 Deleting a Method Stored in OPTIC 3

The methods stored in the OPTIC 3 control unit can only be deleted with the Evolution Workstation software. Select **Method - Delete**, choose the method to be deleted, and confirm.

Note: Method used in a local sequence cannot be deleted from OPTIC memory until it is removed from the sequence.

4.5.5 Listing Methods Stored in OPTIC 3

The methods stored in the OPTIC 3 control unit can be displayed with the Evolution Workstation software. Select **Method - List Uploaded Methods**.

4.6 Running a Method Locally on OPTIC 3

Once a method has been uploaded from the Evolution Workstation to the OPTIC 3 control unit, it can also be run under local control.

If OPTIC 3 shows the Status Display, press Enter to go to the Standby Menu. From the Standby Menu, use the left/right cursor keys to go to the Method Menu. In the Method Menu you can select Method 0 - 9 (the method names and types are displayed). To start the selected method, press the Execute button.

4.7 Optimizing a Method

Some method parameters may have to be optimized by varying a parameter value within a certain range. The Evolution Workstation makes it possible to create a sequence of methods to perform such an optimization. The optimization sequence is created automatically after a sequence name, parameter to be optimized, its minimum and maximum values and a step size in the sequence are defined.

To create an optimization sequence:

- Select **Method – New Optimization Sequence**.
- Enter the name of the sequence and the directory it should be stored in.
- Choose the parameter to be optimized, its minimum and maximum values and the step size.
- Select 'OK' to save the sequence.

The Sequence window will then be displayed where you can modify the sequence as required. For more information about sequences you are referred to Section 5 "Defining and running sequences".

4.8 Converting a Method to Expert Method

Any type of method, besides At-Column can be converted to an Expert Method if you want greater control over the method parameters. Select **Method - Convert to Export Method**.

5. Defining and Running Sequences

A sequence is a combination of a number of methods. Each method can be run automatically a predefined number of times within a sequence. Sequences can be defined and run using the Evolution Workstation software. Up to three sequences can be created locally on OPTIC 3.

Note: Sequences created locally cannot be run from the PC.

5.1 Defining Sequences Locally

Use the cursor keys to locate the **Sequence menu** and press **Enter**. Use the cursor keys to select Sequence 1, 2 or 3. Select the methods to be included in the sequence. These methods must have been created with the Evolution Workstation software, and then uploaded to OPTIC 3. For each method the number of executions can be defined. After entering the last method in the sequence, press **Exit**.

5.2 Running Sequences Locally

Once a sequence has been created, it can be selected in the Sequence menu and started by pressing **Execute**. If **Run/End Run** is pressed while running a sequence, OPTIC 3 will terminate the current method and advance to the next method in the sequence. To stop the entire sequence, press **Execute/Standby**.

5.3 Defining Sequences using Evolution Workstation Software

Select **File, New, Sequence**. Click **Insert** to insert any previously defined method. You can also specify the number of times it should be executed. This is done by clicking the method and then using the **Repeat** up/down arrows on the right of the screen. Once the number of repeats has been selected, press **Apply** to confirm. To move a method up or down in the sequence, click it to select it and then click **Up** or **Down**. Similarly, you can remove a method from the sequence. The sequence can also be looped by checking the relevant box. Once it has been created the sequence is saved by selecting **File, Save**.

5.4 Running Sequences using Evolution Workstation Software

Once a sequence has been created with the software, it can be activated, executed, stopped, etc. by selecting **Sequence** and the relevant option.

Select **Execute** to upload and start the first method in the sequence on OPTIC 3.

6. Advanced OPTIC 3 Features

6.1 Automated Solvent Vent Mode

One of the most frequently used gas chromatography injection techniques is a large volume injection. In this technique the sample is injected with the split line open at an injector temperature below the solvent boiling point. After the injection, the solvent front passes across the head of the capillary GC column and is vented through the split line. This solvent elimination step is largely completed in approximately 5 to 45 seconds, depending on the solvent type and injection volume. The optimization of the vent time is straightforward but can be time-consuming. OPTIC 3 has a solvent monitor (SM) built in the split line of the injector, which makes it easier to determine the solvent elimination time during the split transfer. Depending on the value of Vent Mode parameter the vent time is determined manually (Vent Mode = Fixed Time) or automatically by the solvent monitor (Vent Mode = SM Threshold or SM Level). The diagram below illustrates the automatic absolute and relative modes.

In the absolute mode (Vent Mode = SM Threshold) the split line is closed when the solvent monitor signal drops below the SM Threshold set point defined by the user (Fig. 6.1). In the relative mode (Vent Mode = SM Level) the system calculates the SM Threshold at which the split line is closed. For this purpose, OPTIC 3 measures the solvent monitor zero (baseline) level (point A in the diagram) and the maximum value of the solvent peak ΔR. The SM Threshold is then calculated as $\Delta R \times$ SM Level.

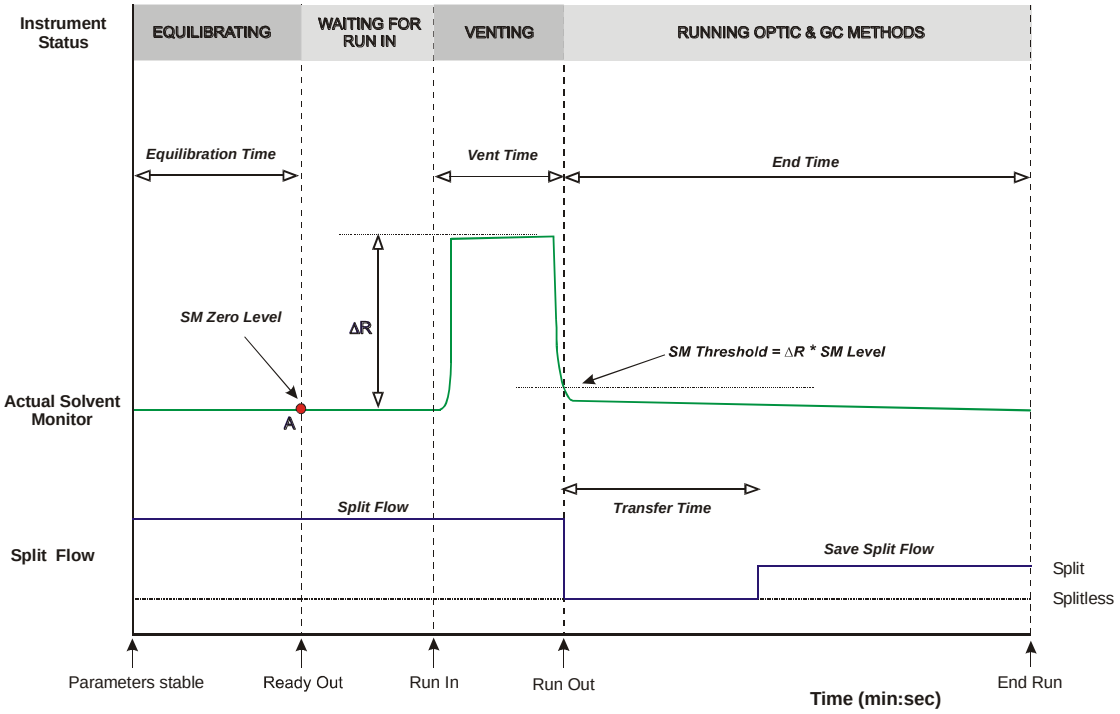


Figure 6.1 Solvent monitor signal in absolute and relative vent modes

6.2 Solvent Cooling Effect

During the solvent split injection, the solvent passes across the head of the capillary GC column and is vented through the split line. The evaporation of solvent can cause considerable cooling of the injector liner. Cooling occurs because the heat capacity of the packing of the liner and the liner itself is low and the heat transfer from the liner to the evaporation site is slow. For some applications the solvent cooling could be beneficial. OPTIC 3 has the option of switching the injector heater off while eliminating the solvent to exploit the effect. This option is available in all standard OPTIC 3 methods apart from At-Column. It can be enabled by setting the 'Solvent Cooling Effect' parameter to Yes. The diagram below illustrates a possible modification (dashed line) of a method temperature profile resulting from the solvent cooling effect.

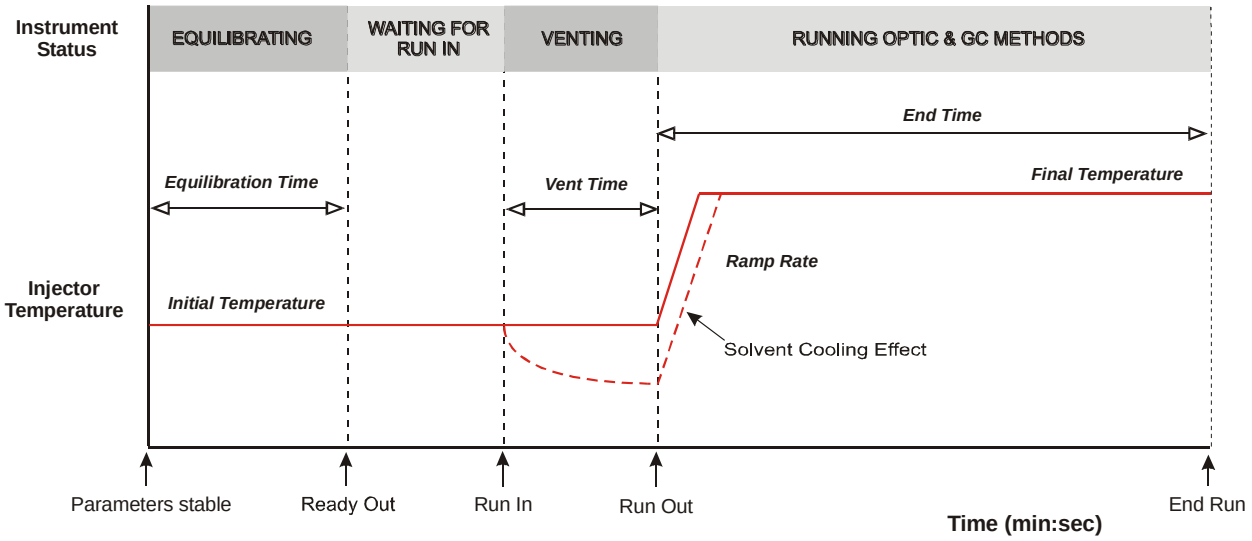


Figure 6.2 Injector temperature - Solvent-cooling effect

6.3 Floating Final Temperature

By default, in all OPTIC 3 methods the final injector temperature is maintained at a constant value until the end of the method. OPTIC 3 has the option of setting the final temperature to 'floating' after a specified interval (Hold Time) has elapsed. It is enabled by setting the Temperature Control parameter to 'Floating' and the Hold Time to an appropriate value, smaller than the End Time. The diagram below shows an example of an injector temperature profile with a floating segment (dashed line).

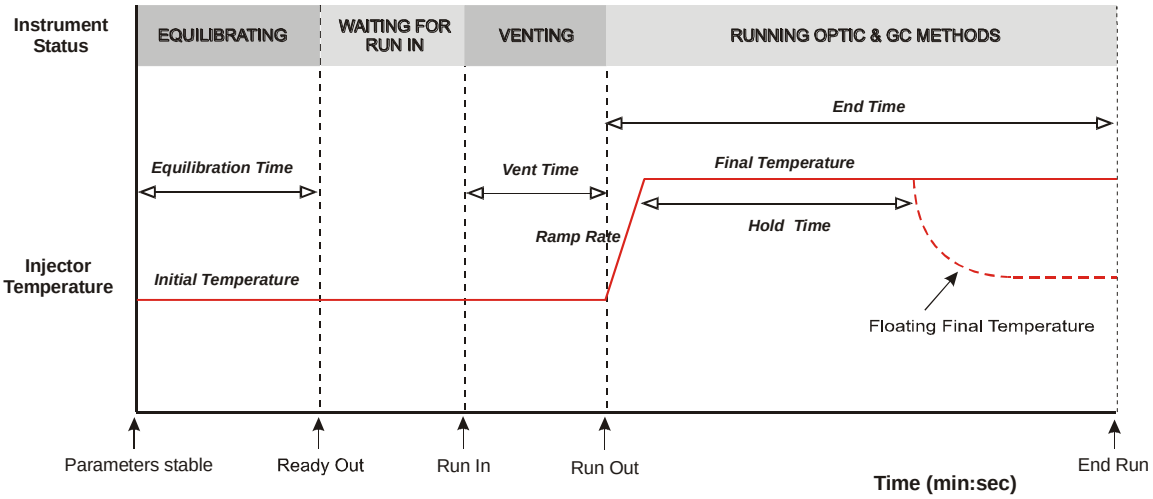


Figure 6.3 Injector temperature - Floating final temperature

6.4 Negative Temperature Ramps in Expert Method

The OPTIC 3 Expert Methods make it possible to set both positive and negative temperature ramps for the injector temperature profile (see default Expert Method profiles). The ramp rate can only be specified for positive temperature ramps, not for negative ramps. Furthermore, the Hold Time for negative ramp segments is also set differently than for positive segments. The specified temperature should be above the expected GC oven temperature during the method run.

6.5 Cooling Valve Mode

OPTIC 3 has the option of switching injector cooling on and off during the method run time. The method parameter Cooling Valve Mode can be set to Yes or No. When it is set to No, cooling is switched off and the injector temperature is maintained by the injector heater controller.

6.6 Auxiliary Outputs

OPTIC 3 has seven auxiliary outputs. These are normally open relays under software control for external devices. Any of the auxiliary outputs can be used in any of the seven types of the OPTIC 3 methods. To keep the Evolution Workstation screens uncluttered, the auxiliary outputs are only shown in the methods if they have first been selected in **Configuration-System Configuration-Auxiliary Outputs**.

6.7 Auxiliary Inputs

OPTIC 3 has four auxiliary inputs, which respond to contact closures or openings. When an input signal is received, OPTIC 3 can respond appropriately when executing an Expert method. The inputs first have to be selected in **Configuration -System Configuration-Auxiliary Inputs.** Further details are included in Section 4 "Defining and running methods", under "Expert Method".

6.8 Cryotrap Control

OPTIC 3-SC and OPTIC 3-DC have control facilities for an ATAS GL Cryogenic Trap. The trap can be used to collect the sample in a narrow band at the head of the capillary GC column by cooling a short section of the column. The electronic control board and the trap itself are designed for rapid cooling and subsequent re-heating to ensure an excellent peak shape and reproducibility. The trap can be used with any of the OPTIC 3 standard method types. An example of the cryotrap temperature profile, incorporated in a method, is shown below.

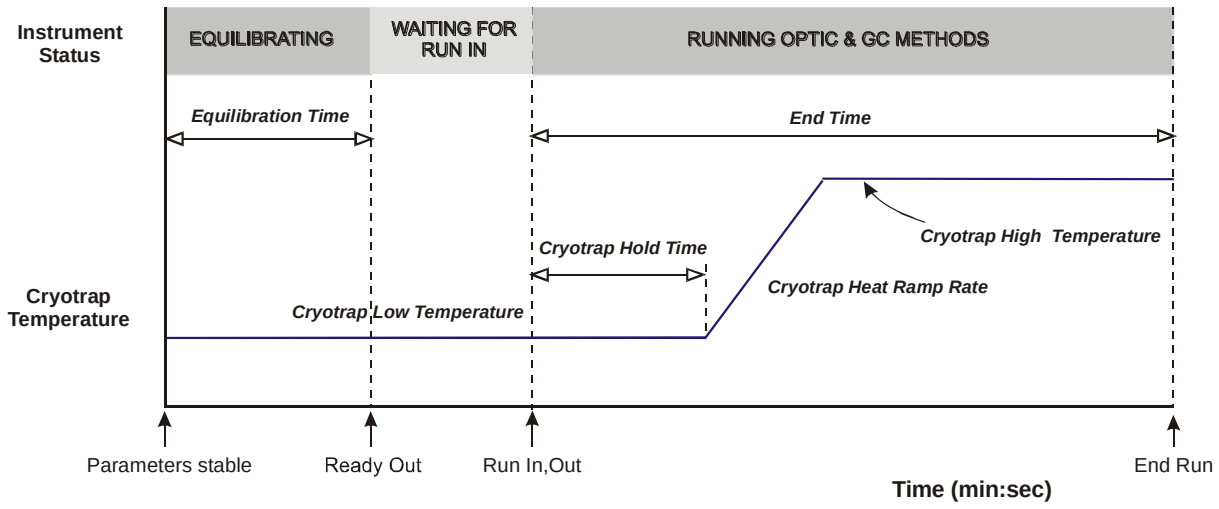


Figure 6.4 Cryotrap temperature profile.

6.9 Technical Notes

For a range of technical notes about the OPTIC 3 injector and other ATAS products, please visit www.atasgl.com

7. Maintenance

OPTIC 3 needs little routine maintenance, other than replacing consumables. In some cases, the injector may be affected by contamination and require cleaning. If there are any problems with the operation of the system, please see Section 8 "Troubleshooting and diagnostics".

7.1 Routine Maintenance

OPTIC 3 does not need any routine maintenance other than that for normal GC equipment, i.e.:

1. The septum should be changed regularly.

2. The liner should be inspected regularly and, if contaminated, should be re-packed or replaced.

7.2 Cleaning

If carryover or background contamination is evident in the chromatograms, and the problem has been traced to the OPTIC 3 injector, it may need to be cleaned as described below.

1. Remove the column and insert a clean empty liner. Enter the following parameters from the **Standby Menu** on the control unit or by selecting **Configuration - Direct Control** in the Evolution Workstation software:

   - Split valve state: Split
   - Injector temperature: 600 °C
   - Column flow: 0.5 ml/min
   - Split flow: 200 ml/min

   Activate these parameters and purge the injector for one hour.

2. If a hot air gun is available, heat the carrier and split lines (and the septum purge line, if used) to help remove the contamination. Be careful with the solvent sensor.

3. Repeat steps 1 and 2 with the bottom of the injector blocked with a solid (no hole) ferrule or paper clip.

4. Test the system to see if the carryover has disappeared.

5. If carryover is still present, remove the column and OPTIC 3 liner. Disconnect the carrier, split and septum purge lines at the rear panel of the control unit.

6. Position a large beaker under the open injector base to collect waste and flush each line and the injector itself with solvent to remove contamination. Use cotton wool buds moistened with solvent to remove any deposits from the internal walls of the injector.

7. Repeat steps 1 to 4.

8. If carryover is still present contact your supplier.

7.3 Servicing

The OPTIC 3 system does not normally require any servicing. If there are problems, which cannot be solved by replacing consumables, cleaning the injector, or following the troubleshooting instructions, then please contact your supplier for further support.

8. Troubleshooting and diagnostics

All OPTIC 3 error messages are displayed on the control unit. Warnings are displayed on the control unit display and also, if a PC is connected, on the status line of the Evolution Workstation software. An intermittent beep will sound when an error or warning message is displayed.

8.1 Errors and Warnings

8.1.1 Injector Thermocouple Fault

The error message **INJ TC FAULT** is generated when the injector thermocouple loses its connection to the body of the injector, if the thermocouple fails, or if the connection to the OPTIC 3 control unit is lost. This error is displayed on the local display if the problem exists for two seconds or more. The heater is switched OFF. This fault is non-recoverable i.e. the fault must be addressed and the instrument powered off and then on again.

8.1.2 Injector Temperature above Maximum

The error **INJ TEMP ABOVE MAX** message is generated when the injector temperature exceeds the maximum temperature of 650°C. This error is displayed on the local display if the injector temperature is above 650°C and is rising. The heater is switched OFF. This fault is non-recoverable i.e. the fault must be addressed and the instrument powered off and then on again.

8.1.3 Cryotrap Thermocouple Fault

This error **CRYO TC FAULT** message is generated when the cryotrap thermocouple loses its connection to the body of the trap, if the thermocouple fails, or if the connection to the OPTIC 3 control unit is lost. This error is displayed on the local display if problem exists for two seconds or more. The cryotrap heater is switched OFF. This fault is non-recoverable i.e. the fault must be addressed and the instrument powered off and then on again.

8.1.4 Cryotrap Temperature above Maximum

The error **CRYO TEMP ABOVE MAX** message is generated when the cryotrap temperature exceeds the maximum of 450°C. This message is displayed on the local display (status line) if the cryotrap temperature is above 450°C and rising. The cryotrap heater is switched OFF. This fault is non-recoverable i.e. the fault must be addressed and the instrument powered off and then on again.

8.1.5 Injector Temperature Warning

The **INJ TEMP WARNING** message is generated when injector temperature setpoint cannot be reached during the method initialization, method run or standby. This message is displayed on the local display and the status line of the Evolution

Workstation software. The software checks that the ramp is following its course and maintained correctly. If the actual temperature is ±50°C from the set point then **INJ TEMP WARNING** is displayed. This is a warning and is therefore recoverable. However once the warning has been acknowledged (by pressing "ENTER") it should not occur again until the instrument is next powered on.

8.1.6 Cryotrap Temperature Warning

The **CRYO TEMP WARNING** message is generated when cryotrap temperature set point cannot be reached during the method initialization, method run or standby. This message is displayed on the local display and the PC. The consequences are the same as for 'Injector Temperature warning'.

8.1.7 Injector Pressure Warning

The **INJ PRES WARNING** message is generated when the injector pressure (column flow) set point cannot be reached during the method initialization, method run or standby. This message is displayed on the local display and the PC. The software checks that the pressure (column flow) is maintained at the set point. This message is generated if the actual pressure differs by ±25 kPa (±3.6 psi) from the calculated setpoint (which is required to set the column flow) for two minutes. This is a warning and is therefore recoverable. However once the warning has been acknowledged (by pressing "ENTER") it should not occur again until the instrument is next powered on.

8.1.8 Auxiliary Pressure Warning

The **AUX PRES WARNING** message is generated when the auxiliary pressure set point cannot be reached during the method initialization, method run or standby. This message is displayed on the local display and the PC. The software checks that the pressure is maintained at the set point. This warning message is generated if the actual pressure differs by ±25 kPa (±3.6 psi) from the set point for two minutes.

8.1.9 Auxiliary Flow Warning

The **AUX FLOW WARNING** message is generated when the auxiliary flow set point cannot be reached during the method initialization, method run or standby. This message is displayed on the local display and the PC. The software checks that the auxiliary flow is maintained at the set point. This warning message is generated if the actual flow differs by ±20% or ±2 ml/min (whichever is higher) from the set point for two minutes.

8.2 Troubleshooting

If there are any problems during the installation or operation of OPTIC 3, please check the following issues. If this information does not help to solve the problem, then please contact your supplier for further support.

8.2.1 No Power

If the instrument fails to power up, check the following:

1. Is the power lead fully inserted into the power socket?
2. Is the mains plug fuse intact (UK only)?
3. Is the fuse in the instrument power socket intact?

8.2.2 Injector Does Not Heat Up

If the instrument powers up, but the injector does not heat up, check the following:

1. Is the power lead to the injector damaged?
2. Is there a high resistance contact between the injector power lead and the injector? (Refer to 8.3)

8.2.3 Injector Heats Up Slowly

If the display temperature takes an excessive time to reach the set temperature, check the following:

1. Is there a high resistance contact between the injector power lead and the injector? (Refer to 8.3)

8.2.4 Injector Cools Down Slowly

If the injector takes an excessive time to cool down to the initial temperature set in the method, check the following:

1. Is the cooling air supply pressure set to 500 kPa (80 PSI)?
2. Is the cooling pipe blocked?

8.2.5 Column Flow Set Point can Not be Reached

If the column flow set point can not be reached and **INJ PRESSURE WARNING** is displayed, check the following:

1. Is the carrier gas supply pressure set to at least 500 - 600 kPa (80 PSI)?
2. Is there a leak in the system?

8.3 Injector Power Lead Connection

If there is a high resistance contact between the injector power lead and the injector, carry out the following procedure:

1. Ensure that power to the instrument is switched OFF.

2. Remove the power lead and inspect the terminals. Any pitting or discoloration can be removed by careful use of very fine emery-cloth.

3. Clean the terminals and reconnect them securely to the injector.

9. Technical Specifications

General

- Dimensions: 43 cm × 15 cm × 43 cm (h × w × d)
- Weight: 11 kg
- Ambient temperature range: 10 - 40°C
- Ambient operating humidity: 5 - 90%
- Mains power: 100 ÷ 240 V AC, 50/60 Hz
- Power consumption: 80 VA
- Conforms to safety standards:
  The European Low Voltage Directive 73/23/EEC,
  The Electromagnetic Compatibility Directive 89/336/EEC
- Storage of ten methods and three sequences

Injector

- Full electronic pressure/flow control
- Maximum operating temperature: up to 600°C at a GC oven temperature of 35°C
- Cooling: air (down to 35°C), LCO_2 (down to -50°C), LN_2 (down to -180°C)
- Temperature ramp rates: 0.2 - 30 °C/sec
- Up to nine temperature program ramps including negative
- Compatible with Merlin MicroSeal septum
- Compressed air for cooling: moisture and oil free air at a pressure of 500 kPa (80 PSI)

Electronic Gas Control

- Two EGC channels
- Full electronic control of column, split and septum purge flows
- Pressure range: 2 - 100 PSI (14 -700 kPa)
- Total Flow range: 6 - 300 ml/min He (main channel, all models), 2 - 100 ml/min He (auxiliary channel, OPTIC 3-D and DC only)
- Pressure sensors:
  - accuracy : ± 2% full scale
  - repeatability: ± 0.2% full scale
- Flow sensors:
  - accuracy : ± 2% full scale
  - repeatability: ± 0.2% of full scale
- Up to nine flow program ramps
- He, N_2 or H_2 carrier gas at a maximum pressure of 100 PSI (700 kPa)
- Solvent sensor in the split line (optional)

External Communication

- LAN
- RS-232 (115,200 baud max.)
- Seven auxiliary relay outputs (30V/500 mA max.)
- External 5 V and 24 V supply connections (250 mA max.)
- Remote start/stop to GC and autosampler
- Four auxiliary inputs

Evolution Workstation Software

- Real-time system status and parameter display
- Method definition and development
- Automatically generated optimization sequences
- Direct control of the instrument
- System run log file
- Password protection with two access levels
- On-line help

Cryotrap

- Operating temperature range : -150°C to +350°C
- Cooling: 1) Cold gas (N_2, He or dry air) produced by LN_2 heat exchanger;
  2) Direct LN_2 from pressurized vessel;
- Temperature ramp rates: 1.0 - 50 °C/sec
- Low LN_2 consumption
- Compatible with any GC

10. OPTIC 3 Cryotrap

The OPTIC 3 cryotrap is designed to solve problems with peak tailing and band broadening in capillary gas chromatography. Its cold trap can be used to focus the sample in a narrow band at the head of the column by cooling a short section of the column. Rapid cooling and heating ensures an optimum peak shape and reproducibility. The cryotrap is fitted to OPTIC 3-SC and OPTIC 3-DC and is available as an accessory for other OPTIC 3 models.

10.1 Hardware

The cryotrap consists of the control board inside the OPTIC 3 control unit, the trap, the heat exchanger, and the liquid Nitrogen Dewar. The heart of the trap is a small heater, which can be cooled to sub-ambient temperatures and heated up to 350°C. The trap is cooled by gas flowing through the heat exchanger immersed in the Dewar with liquid Nitrogen. After trapping, the sample is released by a rapid resistive heating. The cooling and heating of the trap are controlled by the OPTIC 3 control unit.

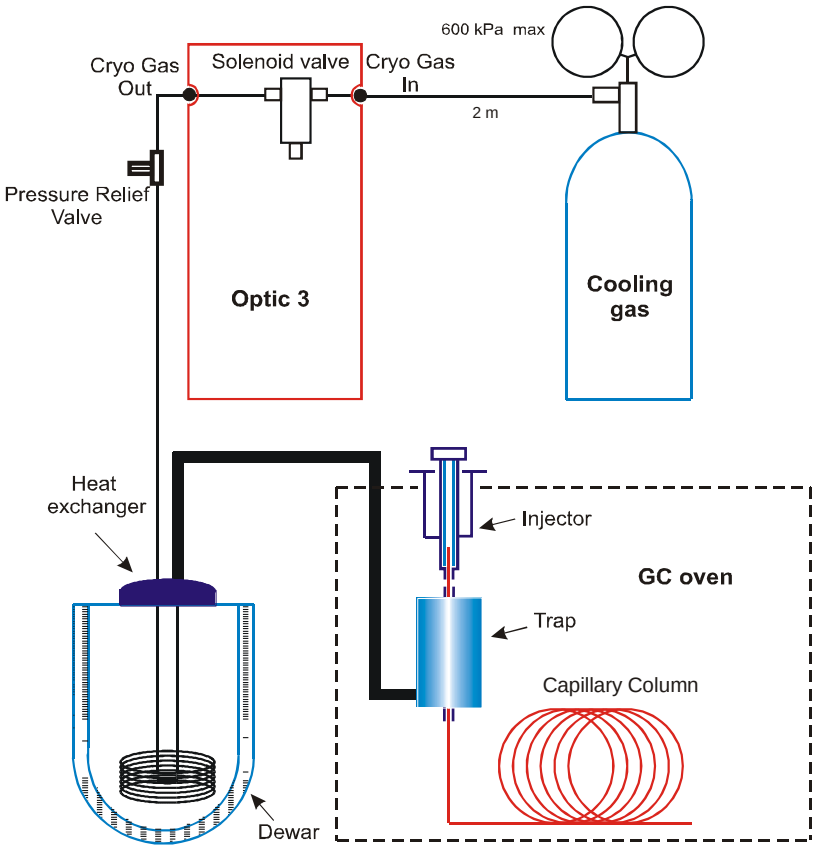


Figure 10.1 Cooling gas supply system of OPTIC 3 cryotrap.

The OPTIC 3 cryotrap cooling gas supply system is shown in Figure 10.1. Nitrogen, dry air or another gas can be used as the cooling medium for the trap. It can be supplied either from a gas cylinder or from the laboratory gas supply system. The laboratory air dryer can be used to produce the dry air for the cryotrap cooling as well.

10.2 Rear Panel Connections

The OPTIC 3 rear panel (Figure 10.2) has connections for the trap cooling gas system, trap heater, and the temperature sensor.

The "**Cryo Gas In/Purge In**" port of the cryotrap cooling solenoid valve is connected to the cooling gas supply, and the "**Cryo Gas Out/Purge Out**" port to the inlet of the heat exchanger inserted in the Dewar. The external manual flow controller supplied with the cryotrap kit should be installed in parallel with the solenoid valve (see Figure 10.1). It is used to set a bypass flow, to avoid blockage of the heat exchanger by ice while it is immersed in the liquid Nitrogen during periods when the trap is not being cooled.

The "**Cryotrap Thermocouple**" connector is used for the trap temperature sensor.

The " **Cryotrap Heater**" connector is used for the trap heater.

10.3 Cryotrap Installation

This section should be read carefully, in conjunction with any specific instructions supplied with the cryotrap installation kit. Should you intend to install the cryotrap onto an instrument for which a dedicated installation kit is not available, then please follow these instructions with particular care. The installation should only be carried out by personnel familiar with the relevant safety regulations and cryogenic operations.

The installation of the trap includes four steps:

1. Installation of the cryotrap board.
2. Installation of the trap in the gas chromatograph oven.
3. Connections of the trap to a gas supply.
4. Making the electrical connections.

10.3.1 Installation of the Cryotrap Board

Normally OPTIC 3-SC or OPTIC 3-DC Injection Systems are supplied with the cryotrap board installed. If an OPTIC 3-S or OPTIC 3-D is to be extended with the cryotrap option, refer to the instructions supplied with the cryotrap installation kit for installation details. Please follow these instructions with particular care and do not hesitate to contact the supplier should you have any questions.

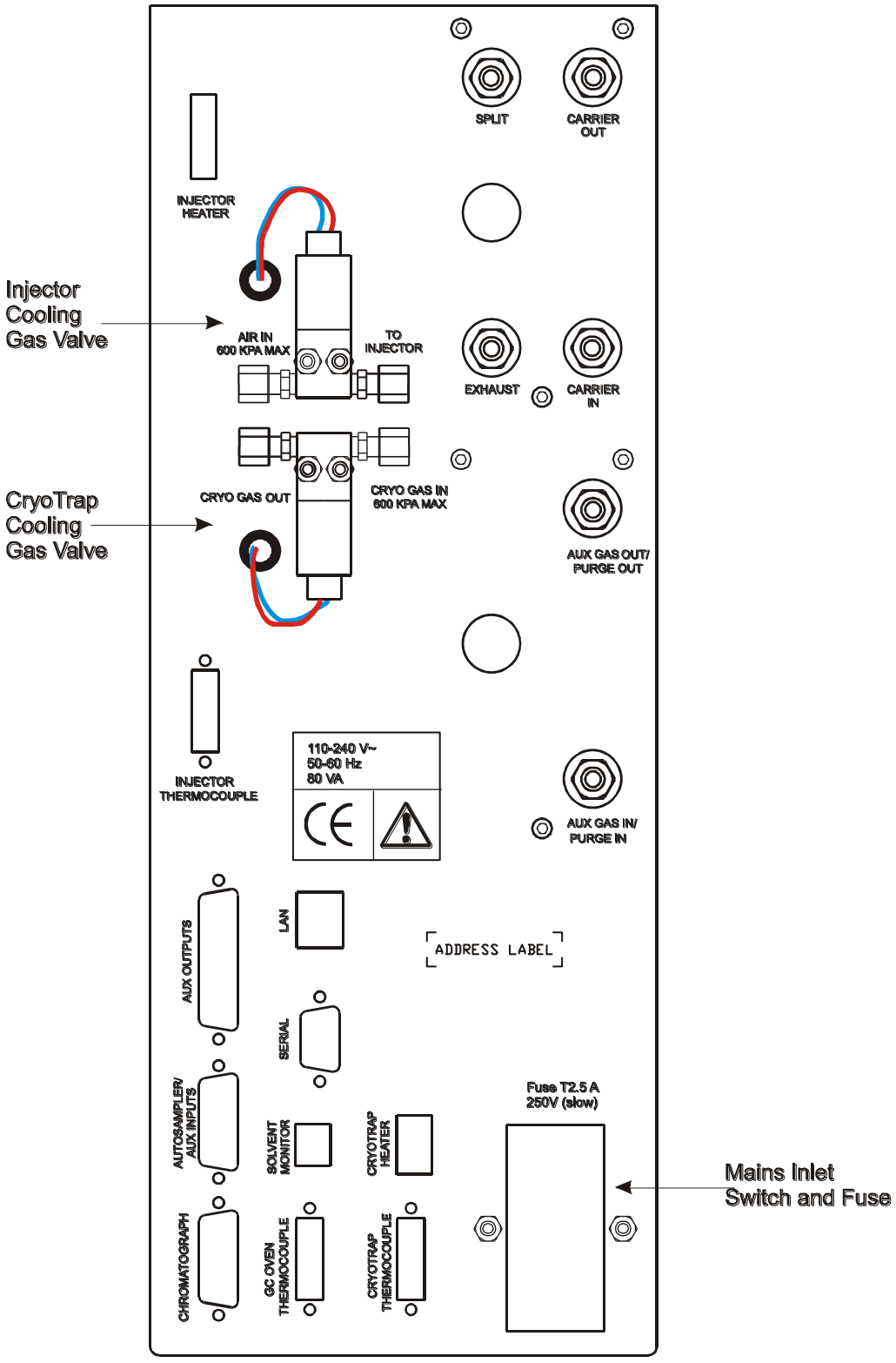


Figure 10.2 OPTIC 3-DC rear panel. OPTIC 3-SC does not have the auxiliary gas channel.

10.3.2 Installation of the Cryotrap in the GC Oven

WARNING: It is essential that both the OPTIC 3 control unit and the host GC are disconnected from the mains supply until the installation is completed.

The trap should be mounted vertically in the gas chromatograph oven, below the injector body. A mounting kit is supplied with the instrument. Ideally, the trap is installed in a location, which makes it possible to run the trap cooling line through existing holes (e.g. for a second injector) in the inner and outer panels of the oven. If there are no such holes, they will have to be made by competent personnel after choosing their location carefully.

After completing the installation, it should be inspected by a qualified person.

10.3.2.1 Mounting the Trap

1. Determine the location for the trap and, if necessary, remove or relocate the second injector (if fitted).

2. If necessary, make the holes in the oven walls.

3. Install the mounting bracket in a corner of the chromatograph oven, close to the injector to be used with the cryotrap.

4. Carefully guide the cooling gas inlet pipe together with the thermocouple and heater cables through the holes in the GC oven wall.

5. Secure the trap to the mounting bracket. Make sure that there is enough space to install the column.

For more details, refer to the instructions supplied with the cryotrap installation kit for installation details.

10.3.2.2 Connecting the GC Column

The column installation depends upon the role that the cryotrap and the GC are to perform. See Appendix A "Hardware Installation" for further details about column installation.

1. Guide the end of the column through the central tube of the trap.

2. Fit a graphite ferrule, of an appropriate internal diameter for the column to be used, to the nut at the base of the injector. Avoid the use of Vespel ferrules, as these are likely to cause leaks.

3. If standard Pyrex, quartz or thermal desorption liners are used, insert the tip of the column to a depth of 10 to 21 mm depending on the liner used. If fritted liners are

used, insert the column until the tip touches the underside of the frit and then withdraw the column approximately 1 mm.

4. Tighten the nut at the base of the injector until the column is retained within the injector.

CAUTION: Do not tighten the base nut excessively. The injector base is very fragile and can be damaged easily.

5. When installation is complete, all connections should be leak tested with an electronic gas leak detector or a 50/50 solution of -propanol and water. Under no circumstances should a soap solution or similar be used, as this would contaminate the injector.

10.3.2.3 Adjusting the Trap Position

The trap position should be adjusted to ensure the best performance. Preferably, the central tube of the trap should be aligned with the injector axis.

CAUTION: When adjusting the position of the trap, special care should be taken not to break the column.

1. Align the trap in all three planes such that its central tube is aligned with the vertical axis of the injector.

2. Move the trap up along the mounting rod so that the upper end of the central tube is approximately 5-10 mm from the injector nut and secure the trap.

3. Inside the column oven, pack mineral wool insulation tightly around the cooling gas inlet pipe to ensure that hot air from the oven cannot leak out around the pipe.

10.3.3 Connecting the Gas Lines

The cryotrap uses a heat exchanger immersed into a Dewar with liquid Nitrogen to produce cold gas for cooling the trap. The gas lines diagram of the cooling system is shown in Figure 10.1.

10.3.3.1 Connecting the Trap to a Gas Supply

Connect a gas cylinder or a laboratory gas outlet to the "Cryo Gas In" port, as shown in Figure 10.1. For optimum cooling efficiency the cryotrap needs a supply of cooling gas at 45 – 60 PSI (300 – 400 kPa). A lower pressure can be used, but the cooling rate will be correspondingly lower.

Nitrogen, dry air or another gas can be used as the cooling medium for the cryotrap. While analytical grade gas is not needed, the gas supply should be free of moisture.

10.3.3.2 Installing the Heat Exchanger

Connect the "Cryo Gas Out" port of the OPTIC 3 control unit to the heat exchanger inlet using the PTFE tubing supplied with the cryotrap kit. Cut the tube approx. 10-15 cm from the solenoid valve. Install the pressure relief valve into the line (Fig. 10.1). Connect the heat exchanger outlet to the trap using the thermally insulated hose.

10.3.4 Electrical Connections

Connect the trap assembly to the OPTIC 3 control unit using the heater and thermocouple cables supplied with the instrument.

WARNING: The control unit should be disconnected from the mains supply until all the electrical connections have been made.

For the stand alone CryoFocus 3 system external logic connections refer to the Appendix A.

10.4 Operating the cryotrap

10.4.1 Configuring the Cryotrap

The cryotrap can be used with any of the OPTIC 3 standard method types. Prior to that, it should be enabled in the system configuration. This can be done either through the Evolution Workstation software or the local OPTIC 3 control keypad.

Ensure that the OPTIC 3 control unit is switched on and communicating with the Evolution Workstation software on the PC. Select **Configuration - System Configuration - General.** Check the cryotrap option. Click on OK to confirm the configuration.

To enable the cryotrap via the local control keypad, go to **Configuration Menu - Cryotrap**. Using 'Up/Down' keys set the option to **Yes**. Press 'Exit' to confirm.

10.4.2 Cryotrap Temperature Profile

When the cryotrap is enabled, the trap temperature profile will be added to any method you create. An example is shown in Section 6 "Advanced OPTIC 3 features".

The following parameters can be set in the profile:

Cryo Low Temperature:

This is the initial temperature, or cryofocussing temperature, to cold-trap the analytes of interest. Any temperature between –150 °C and +350 °C may be set but this temperature will normally be in the range –150 °C to 0 °C.

Cryo Hold Time:

This is the time required after receiving a run signal for the sample to be fully transferred from the injector to the cryotrap.

Note: *If the sample is introduced via a splitless injection system the Cryo Hold Time would be a bit longer than the Transfer Time. The time range available is 00.00 to 120:00 minutes but it is usual to set a time in the range 1:00 to 5:00 minutes.*

Cryo Heat Ramp Rate:

This is the rate in degrees Celsius per second (°C/sec) at which the trap heats from the **Cryo Low Temperature** to the **Cryo High Temperature**. Currently the range available is 1 to 100 °C/sec although in practice the rates achievable are 1 to around 60 °C/sec. It is recommended that a rate of 50 °C is used to achieve the most efficient transfer to the column.

Cryo High Temperature:

This is the final temperature, or desorption temperature, to transfer the analytes from the trap to the column. Any temperature between -150 °C and +350 °C may be set but it is usual to set a temperature in the range +100 °C to +350 °C.

Note: *It is important not to set this temperature above the maximum operation temperature of the capillary column used otherwise a permanent damage to the column will result and column bleed will be seen in the resultant chromatogram.*

10.4.3 Cooling the Heat Exchanger

Before the method using the cryotrap can be started, the heat exchanger coil should be cooled down to subambient temperature. This is done by inserting it in the Dewar filled with liquid Nitrogen.

Secure the Dewar filled with the liquid Nitrogen on the floor or on the bench, next to the GC. Remove the cap from the Dewar. Carefully insert the heat exchanger coil into the Dewar. Introduce it slowly, especially when the heat exchanger coil enters the liquid. This is to prevent sudden boiling of the liquid Nitrogen and thus ejecting it from the Dewar. Insert the heat exchanger fully into the Dewar. The cryotrap is now ready to operate.

CAUTION: **Whenever the heat exchanger coil is immersed in liquid Nitrogen there should be a flow of gas through it. This is to avoid blockage of the heat exchanger by ice.**

10.4 Fast Splitless Transfer

The cryotrap can be used for Fast Splitless Transfer (FST). This requires the OPTIC 3 optional auxiliary gas control channel, fitted to OPTIC 3-DC. Alternatively, an external solenoid-operated valve controlled by OPTIC 3, connected in series with the manual needle valve can be used instead. The diagram in Figure 9.3 shows the gas connections required for FST. A T-splitter is installed on the column, at the outlet of the trap. This split flow can be routed to the '**Aux Gas In/Purge In**' port on the rear panel of OPTIC 3 so that a higher flow rate can be used when transferring analytes from the

sample inlet to the trap. This speeds up the transfer and reduces the likelihood of sample degradation inside the injector, making the system ideal for thermally labile sample components.

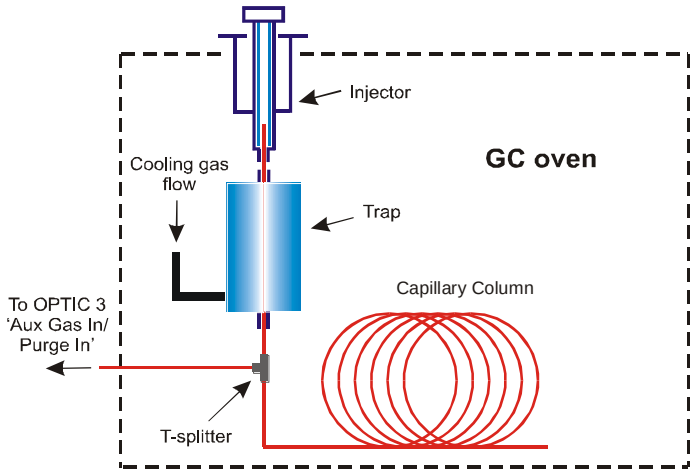


Figure 10.3 OPTIC 3 cryotrap Fast Splitless Transfer gas connections

If the auxiliary gas control channel is available, the system is expanded for Fast Splitless Transfers as follows:

1. Install a T-splitter on the column, at the outlet of the trap.

2. Connect the T-splitter to the "**Aux Gas In/Purge In**" port on the rear panel of the OPTIC 3 control unit.

3. Set the transfer flow with "Auxiliary Flow" parameter in an OPTIC 3 Expert method.

If the OPTIC 3 is not equipped with the auxiliary gas control channel then refer to the instructions supplied with the installation kit for the installation details of the external FST valve and its control. Please follow these instructions with particular care and do not hesitate to contact the supplier should you have any questions.

Appendix A: Hardware Installation

This section should be read carefully, in conjunction with any specific instructions supplied with the installation kit. Please follow these instructions with particular care if you are installing the OPTIC 3 on an instrument for which a dedicated installation kit is not available, and do not hesitate to contact the supplier should you have any questions.

The installation of OPTIC 3 on the GC can be divided into the following steps:

- Installation of the injector in the chromatograph oven.
- Connection of the injector to the gas controls.
- Electrical connections.
- Installation of an appropriate liner and column.
- Installation of the cryogenic trap (OPTIC 3-SC and OPTIC 3-DC).

WARNING: It is important that both the OPTIC 3 and the host GC are disconnected from the mains supply until the installation is complete.

A.1 Installing the Injector

To achieve the best results with OPTIC 3, the injector should be installed directly into the oven insulation, rather than into an injector block. Fitting OPTIC 3 in this way ensures that it cools rapidly to the required initial temperature.

A.1.1 Location

If OPTIC 3 is to be used for manual injection only, it can be fitted anywhere on the top of the oven away from the injector and detector blocks. The most convenient location is an existing hole in the column oven's inner skin. If OPTIC 3 is to be used with an autosampler, extra care is needed in deciding upon the location. In many cases the removal or relocation of an existing injection port will be required. Ensure that the new injector is aligned in all three axes such that its septum cap is co-incident with that of the standard injector.

OPTIC 3 must be grounded to the GC with the cable provided, or with the base plate, depending on the installation kit.

Note: The removal of an injection port entails the removal of the injector heater and temperature sensor. Some GCs will treat the absence of the sensor as a fault. In such cases a resistor has to be fitted in place of the sensor (refer to the installation kit for details) and the GC's injector control must be switched off at all times.

A.1.2 Installation

1. Determine the location for the OPTIC 3 injector and if necessary remove or relocate the existing injection port.

2. Fit the injector body directly to the GC (Agilent 6890) or to the mounting plate provided with the installation kit.

3. Identify the injector gas lines by their labels and decide on the best routes for them. Uncoil the pipes as well as all the electrical leads.

4. Inside the column oven, pack mineral wool insulation tightly around the injector in order to ensure that hot air from the oven cannot leak out around the newly installed injector.

5. Ensure that un-insulated part of the solvent monitor is not in electrical contact with anything.

6. Connect the injector thermocouple to the "Injector Thermocouple" connector on the rear panel of the OPTIC 3.

7. The OPTIC 3 solvent monitor and filter should be installed in a pneumatic T-piece in the split line. Connect the solvent monitor cable to the "Solvent Monitor" connector on the rear panel.

8. Install the oven thermocouple supplied with the OPTIC 3 installation kit in the GC oven, as close as possible to the GC's own oven thermocouple. Connect the thermocouple to the "GC Oven Thermocouple" connector on the OPTIC 3 rear panel.

9. OPTIC 3-SC and OPTIC 3-DC only: the installation and connection of the cryotrap are described in Section 10 "OPTIC 3 Cryotrap".

10. The connection of the OPTIC 3 to the host GC and autosampler is described in Section 1.3 of this appendix and the notes supplied with the installation kit.

WARNING: The completed installation should be checked by a competent person before use.

A.2 Gas Line Connection

All gas connections to the OPTIC 3 control unit rear panel are made using 1/8" Swagelok fittings. A set of PTFE 1/8"-1/16" reducing ferrules is supplied with the instrument for the connection of 1/16" injector gas lines.

A standard OPTIC 3-S control unit has the following gas line connections on the rear panel:

- Carrier Gas In (from cylinder or other gas supply)
- Carrier Gas Out (to injector)
- Split
- Exhaust

- Cooling Air In
- Cooling Air Out
- Purge In
- Purge Out

OPTIC 3-D has the following additional gas connections:

- Auxiliary Gas In
- Auxiliary Gas Out

The auxiliary gas channel could, for example, be used for the septum purge flow control.

OPTIC 3-SC and 3-DC have the following additional gas connections for the cryotrap:

- Cryo Gas In
- Cryo Gas Out

A.2.1 Carrier Gas Line

OPTIC 3 needs a carrier gas supply which conforms to the requirements in the Technical Specifications (Section 9 of this manual). This can be provided by a cylinder and regulator or laboratory gas supply system.

1. Connect the carrier gas supply to the "**Carrier In**" coupling on the OPTIC 3 rear panel.

2. Connect the injector's carrier gas line to the "**Carrier Out**" on the OPTIC 3 rear panel, using a Swagelok coupling and reducing ferrule.

A.2.2 Split Line

The split line is sleeved with insulation. When OPTIC 3 is switched on, the split line is electrically live (3V AC). While this does not represent a risk of electric shock, it is important, for the correct operation of the injector, that the split line (including the solvent monitor T-piece and the charcoal filter) is **electrically** isolated from the rest of the chromatograph and OPTIC 3 control unit. A PTFE reducing ferrule in the charcoal filter and/or in the solvent monitor t-piece is used to isolate the split line from the chassis of the control unit.

1. Determine the place for the Solvent Monitor and Filter installation. Cut the pipe using a tube cutter.

2. Using a sharp blade cut the insulation around the pipes and remove the unwanted insulation, leaving approx. 20 mm of the line un-insulated. Secure the ends of insulation with the shrink sleeve.

3. Install the Solvent Monitor and Charcoal Filter. Connect the injector split line to the 1/8" coupling on the 1/8" coupling of the Solvent Monitor inserting the pipe

into the reducing ferrule of the coupling. The insulation should butt up against the fitting leaving no un-insulated pipe exposed. Tighten the fitting.

WARNING: Do not insert the pipe too far into the fitting to avoid electrical contact between the split line pipe and the Solvent Monitor T-piece.

4. If Filter is supplied separately from the Solvent Monitor, connect the remaining port of the Solvent Monitor to the inlet port of the Filter.

5. Connect the remaining part of the split line between the Filter outlet and the "Split" coupling on the OPTIC 3 rear panel, inserting the pipe into the reducing ferrule of the Swagelok coupling. Tighten the fitting.

A.2.3 Septum Purge Line

The standard OPTIC 3 does not offer electronic control of the septum purge flow. The septum purge line can be connected either to fixed flow restrictor (1-2 ml/min at 100 kPa), which is mounted on the rear panel ("**Aux Gas In/Purge In**" port) or blocked with the blanking nut supplied. OPTIC 3-D and OPTIC 3-DC have an auxiliary gas control channel which can be used for septum purge flow control.

To use the septum purge facilities do the following:

1. Connect the injector's septum purge line to the "**Aux Gas In/Purge In**" coupling on the OPTIC 3 rear panel.

2. Leave the "**Aux Gas Out/Purge Out**" coupling open.

A.2.4 Exhaust

The exhaust port always vents carrier gas and also part of the sample during sample injection. To ensure correct operation of the instrument, the "**Exhaust**" coupling should not be blocked. If necessary, the port may be ducted to an exhaust system, but this should not restrict the flow.

A.2.5 Cooling Air Connection

WARNING: When replacing or adjusting the cooling pipe, ensure that the unit is switched off. Before switching on ensure that no part of the cooling pipe is in contact with either the body of the injector or the power connections at the top of the injector.

For optimum cooling efficiency, OPTIC 3 requires a supply of the compressed air at 500 kPa (80 PSI). A lower pressure supply can be used, but the injector cooling will be slower.

While analytical grade air is not needed, if air is supplied from a compressor or air line, the air supply should be filtered to be free of moisture, oil and particulates.

1. Connect the air supply to the "**Air In**" coupling of the solenoid valve on the OPTIC 3 rear panel.

2. Connect the PTFE cooling air hose to the "**To Injector**" coupling on the OPTIC 3 rear panel. Push the other end onto the cooling air stub of the OPTIC 3 injector.

A.3 Electrical Connections

A.3.1 Injector connections

1. Connect the injector power leads to the injector. Use only flat washers supplied with the injector. Screw the connections tightly.

CAUTION: It is important that a good connection is made between the injector power cable and the injector. A bad connection can result in poor operation and the connection becoming excessively hot.

2. The OPTIC 3 solvent monitor and charcoal filter assembly is installed in a pneumatic T-piece in the split line. Connect the solvent monitor cable to the "**Solvent Monitor**" connector on the rear panel. Ensure that uninsulated part of the solvent monitor is not in electrical contact with anything.

3. Connect the injector thermocouple to the "**Injector Thermocouple**" connector on the rear panel of the OPTIC 3.

4. Install the oven thermocouple supplied with the OPTIC 3 installation kit in the GC oven, as close as possible to the oven thermocouple. Connect the thermocouple to the "**GC Oven Thermocouple**" connector on the OPTIC 3 rear panel.

CAUTION: Do not confuse the GC oven thermocouple with the injector thermocouple. This can lead to a damage of the injector.

A.3.2 External Logic

OPTIC 3 has a full range of external logic interfaces and control functions which permit control to be integrated with the operation of the host GC, autosampler and other equipment. The connections are provided on D-type connectors on the rear panel of the control unit. The pin-outs are specified in Appendix E "Rear Panel Connections". Instructions for the connection of OPTIC 3 to the host GC and autosampler are included in the dedicated installation kit for your GC.

A.3.3 Connection to the Host PC

The OPTIC 3 control unit has to be connected to a PC running the Evolution Workstation software. This software is used to define the methods, which are then uploaded to the OPTIC 3 control unit or executed directly from the PC. The uploaded methods can be initiated locally on the control unit. Either a serial or LAN connection may be used. By default the communication is set to LAN. For remote communication to be established make sure that the communication interface parameters of the OPTIC 3 control unit match the parameters set in the Evolution Workstation software on the PC. The communication port configuration is described in Appendix B "Software

Installation and Configuration". Once methods have been uploaded to the control unit the PC may be disconnected if required.

Note: If you use your own serial cable instead of the one supplied in the installation kit, make sure it is a crossed (null modem) cable. If OPTIC 3 is connected to a hub, a standard network cable should be used. If it is connected directly to the PC, a crossed network cable should be used.

A.4 Liner and Column Installation

The ways, in which the column should be installed and the type of the injection port liner required depend upon the application of the OPTIC 3.

A.4.1 Liner Installation

A liner is installed in the injector prior to delivery. To change the liner, follow the procedure below.

CAUTION: During this operation, care should be taken not to stress any of the gas lines.

1. Cool the injector to below 40°C.

2. Switch off OPTIC 3.

3. Unscrew the largest knurled nut (top boss clamping nut) which joins the injector top assembly to the injector body and lift the top assembly aside. Care should be taken not to lose the flat PTFE insulating washer (top boss insulator).

5. Take out the existing liner with the O-ring in place.

6. Insert the new liner with O-ring.

7. Replace the top assembly and tighten the nut finger-tight. Do not use a spanner or wrench, as this is likely to damage the injector. Care should be taken to ensure that the flat PTFE insulating washer is fitted correctly.

8. Switch OPTIC 3 back on.

A.4.2 Column Installation

1. Install a new column using the proper ferrule and the column mounting tool. Push the column through the nut and graphite ferrule (Figure A1). The cone of the ferrule should go into the nut. Avoid the use of Vespel ferrules as these are likely to cause leaks.

2. Insert the end of the column in the column mounting tool so that it protrudes approx. 10 mm from the end. Tight the nut by hand and cut the protruding end of the column flash with the end of the tool.

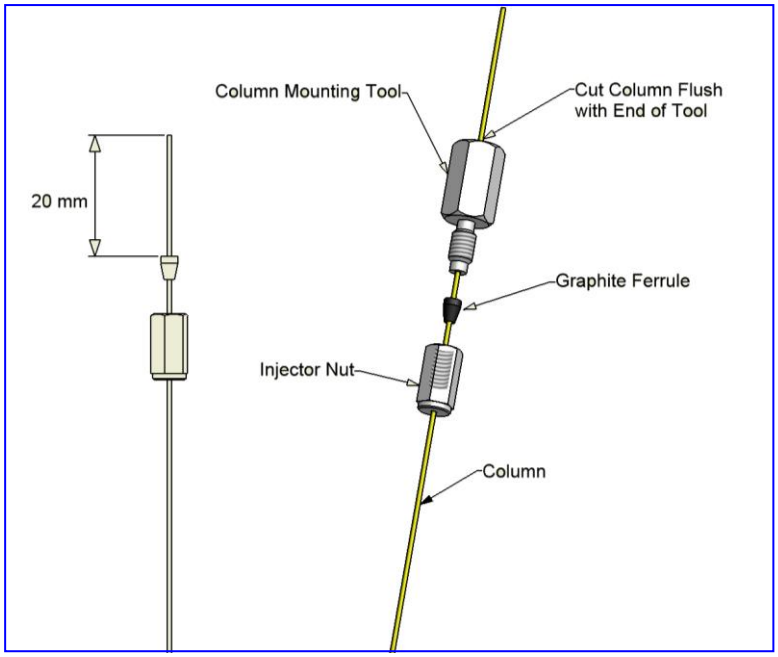


Figure A.1

3. Remove the capillary column from the tool. Insert the column into the injector and secure it by hand tightening the nut. Use the wrench to turn it additional half turn.

4. On-column liners are fitted with the restriction towards the top of the injector and the column should be introduced into the injector until the column tip engages in the bottom of the restriction. Tighten the nut at the base of the injector until the column is retained within the injector. Use the wrench supplied.

CAUTION: Do not to tighten the base nut excessively. The injector base is very fragile and can be damaged easily.

5. When installation is complete, all connections should be leak tested with an electronic gas leak detector or a 50/50 solution of propanol in water.

CAUTION: Under no circumstances should a soap solution or similar be used, as this would contaminate the injector.

A.5 Cryotrap Installation

The installation of the cryotrap (OPTIC 3-SC and OPTIC 3-DC) is described in Section 10 of this guide.

Appendix B: Software Installation and Configuration

B.1 Introduction

The Evolution Workstation software (also referred to in this manual as 'control software') is an integral part of the OPTIC 3 injection system. It is designed to perform the following basic functions:

- Instrument configuration and control.
- Method and sequence definition and development.

In addition, Evolution Workstation offers the following features: automatic generation of the parameter optimisation sequences, direct control of the instrument in the standby state, At-Column calculator, and system run log file. If required, the software can be password protected and offers two levels of access ('User' and 'Manager'). The software includes an extensive Help function.

The methods defined on the PC can be either executed from the PC or uploaded to the control unit to be initiated locally. The uploaded methods can be also used to create up to three sequences to be executed locally.

Note: The run time parameters are displayed in the graph of the instrument status view in the real time without any filtering. The only limiting factor is the precision with which the data are represented. Due to this limitation the curves can sometimes show steps, especially if the displayed parameter range is small. Even in that situation, the displayed parameter irregularities are within the technical specifications of the instrument.

B.2 PC Requirements

Most modern PCs will meet the minimum requirements for the Evolution Workstation software:

- MS Windows 98 (SE), 2000, NT (service pack 4 or later), XP, or Vista
- 64 MB of RAM
- 20 MB free hard disk space
- Serial or Ethernet port for connection to the OPTIC 3 control unit

B.3 Installing Evolution Workstation Software

- Place the CD-ROM in the CD-ROM drive.
- Select the CD-ROM drive in Windows Explorer.
- Double click setup.exe.
- The InstallShield wizard will guide you through the installation process, and you can specify the location where the program should be installed. Please note that it should not be installed on a network drive.

The ATAS program directory contains two subdirectories for the method (*.mth) and sequence (*.seq) files.

If you want, you can create a shortcut to Evolution Workstation.exe on the desktop using the normal Windows procedure.

B.4 Configuring Communication Parameters

After setting up the OPTIC 3 hardware and installing the PC software, they must first be configured to communicate. Either a serial connection or a LAN (Ethernet) may be used. Obviously, both the control unit and the control software must have identical communication configurations.

To set the communication configuration on the control unit:

- Press the left/right cursor key to select the Configuration Menu
- Press the Enter button
- Press the left/right key cursor key to select the Communication Interface

To set the communication configuration in the control software, select **Configuration/ External Communication** in the main menu bar.

When using serial communications, the COM port and the baud rate must be selected. When using the LAN connection, the TCP/IP address must be specified.

Note: If the communication configuration within OPTIC 3 control unit was changed, the instrument should be switched OFF and ON for the new settings to take effect.

B.5 General Configuration

It is probably a good idea to start Evolution Workstation on your PC and go through all the configuration options as you are reading this manual. Under **Configuration** on the task bar at the top of the Evolution Workstation window, you will find:

- Standby parameters: timeout (the time that OPTIC 3 remains waiting for Ready or Run after which it enters the Standby state), standby temperature, column flow, split flow, etc. (the complete list of parameters is included in Appendix D "OPTIC 3 Default Parameters").

- System Configuration: configurations of parameters, such as hardware access control, maximum injector temperature, pressure units, GC column parameters, etc.

- Auxiliary outputs: enable/disable the outputs used in the method, name the auxiliary outputs.

- External Communication: configuration of serial or LAN interfaces with the OPTIC 3 control unit.

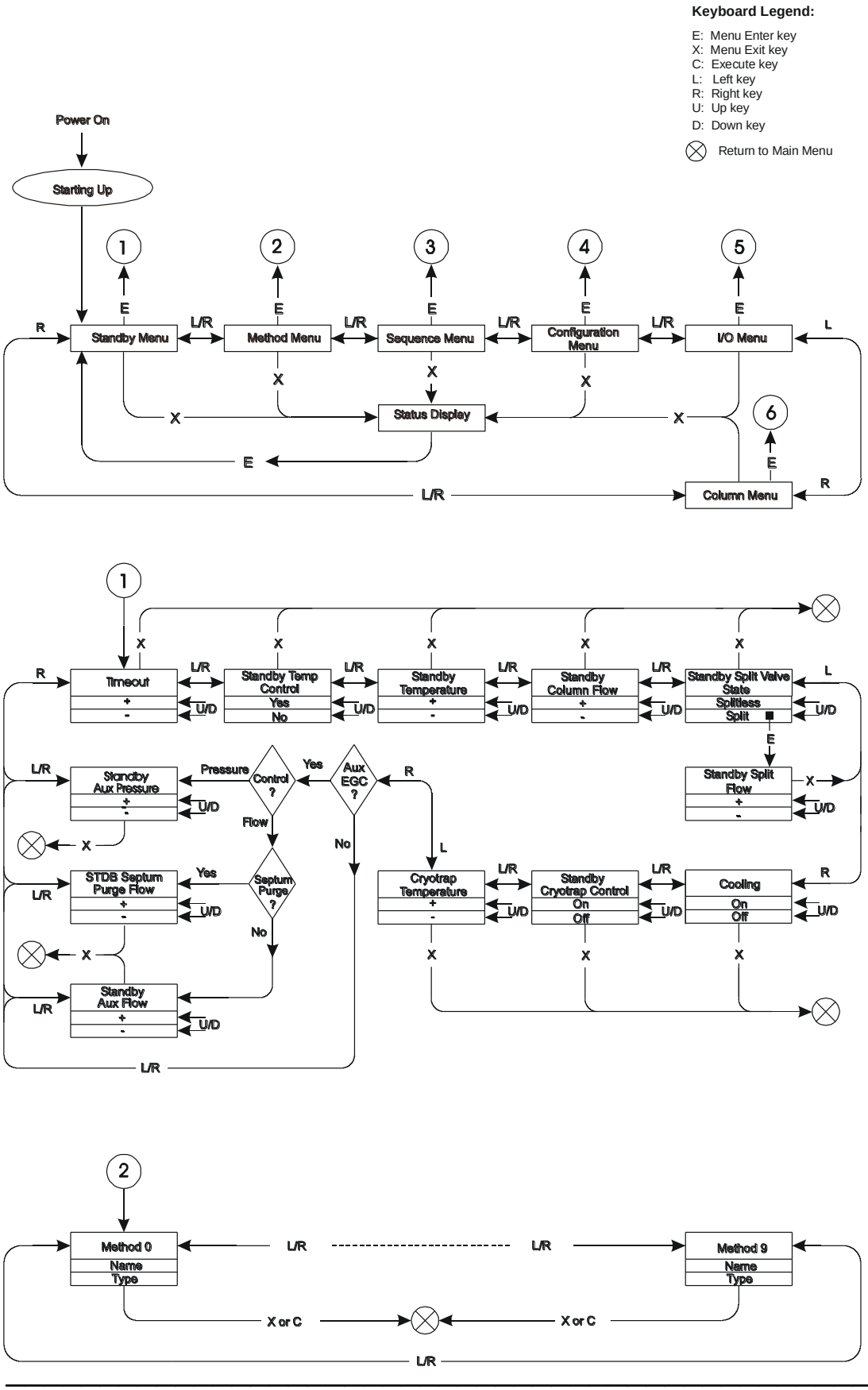
- Access Control: activating access control for the Evolution Workstation software and entering passwords for 'Users', who can only start and stop methods and sequences, and 'Managers', who can define methods, and have access to all functions of the control software.

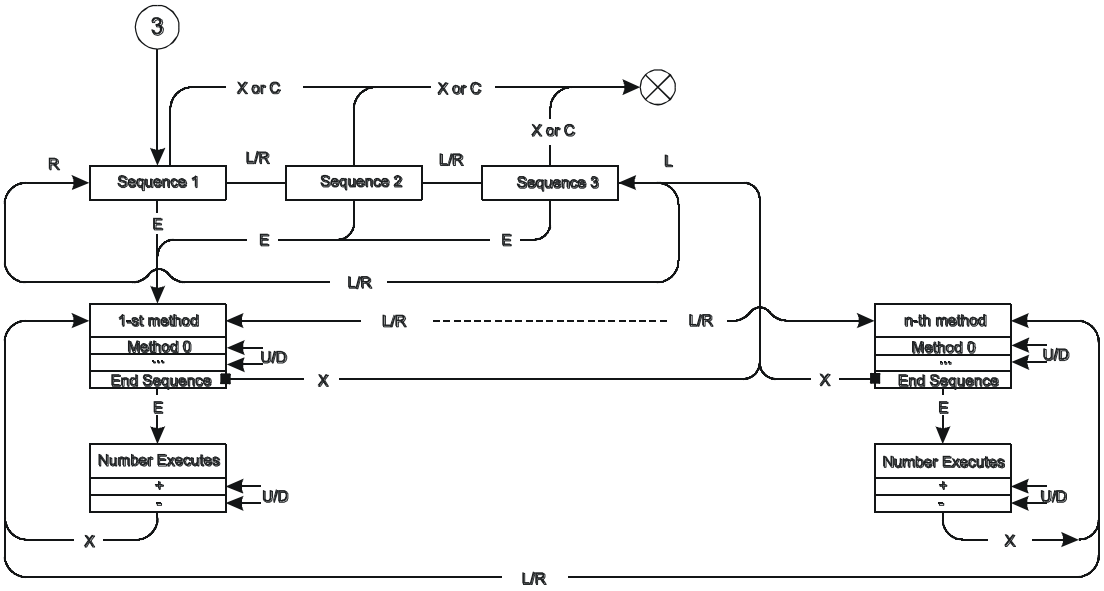
Details of the configuration options are included in the tables in Appendix D "OPTIC 3 Default Parameters", which also show the parameters which can be set both locally from the control unit keypad and the Evolution Workstation software and those which can only be set from the software. Parameters specifically related to the wide range of method types supported by OPTIC 3 are also listed in Appendix D.

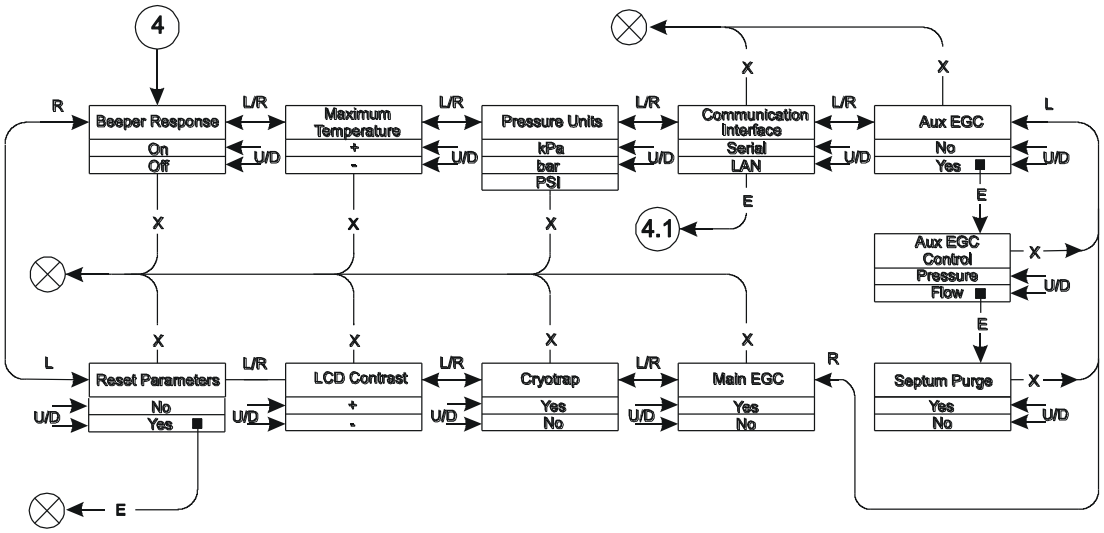
Settings that are related to the auxiliary gas channel and cryotrap are only available if these options are installed. The septum purge option is only available if the auxiliary gas channel is installed (OPTIC 3-D and OPTIC 3-DC), and requires the auxiliary channel to be flow-controlled.

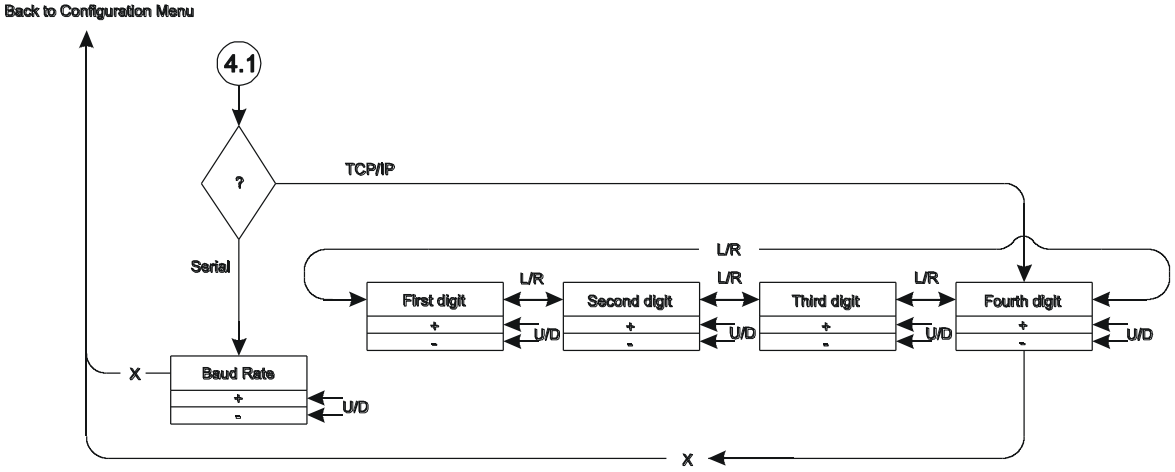
Note: *When using the software, please take into account that some options will only be available if the Evolution Workstation software is communicating with the OPTIC 3 control unit.*

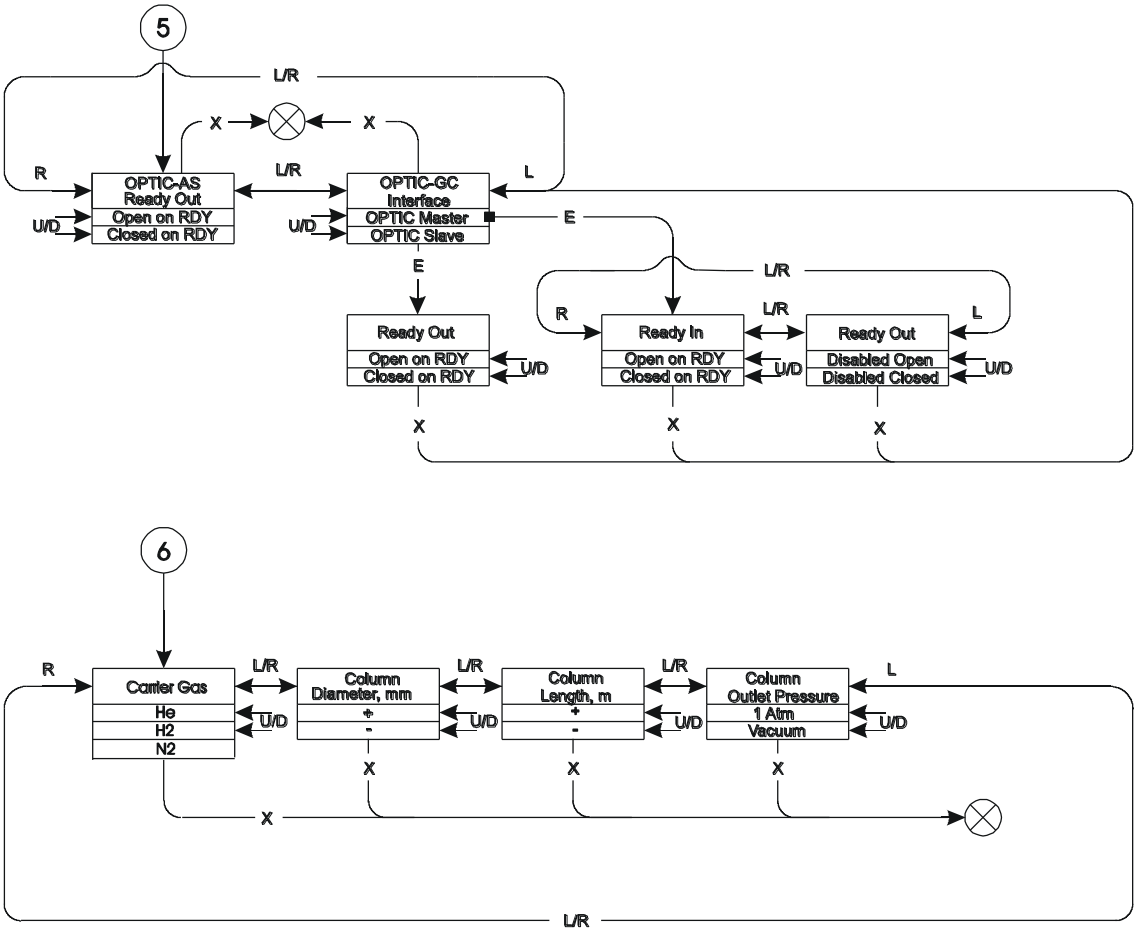
Appendix C: Local Control Menus




3
X or C
X or C
X or C
R
L/R
L/R
L
Sequence 1
Sequence 2
Sequence 3
E
E
E
L/R
1-st method
Method 0
"""
End Sequence
L/R
L/R
n-th method
Method 0
"""
End Sequence
U/D
U/D
X
X
E
E
Number Executes
+
-
U/D
Number Executes
+
-
U/D
X
X
L/R


4
X
X
R
L/R
L/R
L/R
L/R
L/R
L
Beeper Response
On
Off
Maximum Temperature
+
-
Pressure Units
kPa
bar
PSI
Communication Interface
Serial
LAN
Aux EGC
No
Yes
U/D
U/D
U/D
U/D
U/D
E
4.1
X
X
X
Aux EGC Control
Pressure
Flow
X
U/D
E
L
R
Reset Parameters
No
Yes
LCD Contrast
+
-
Cryotrap
Yes
No
Main EGC
Yes
No
Septum Purge
Yes
No
X
U/D
E


Back to Configuration Menu
4.1
?
TCP/IP
Serial
L/R
First digit
Second digit
Third digit
Fourth digit
+
-
U/D
Baud Rate
X
X


5
L/R
X
X
R
OPTIC-AS
Ready Out
Open on RDY
Closed on RDY
U/D
L/R
OPTIC-GC
Interface
OPTIC Master
OPTIC Slave
U/D
L
E
E
L/R
R
Ready Out
Open on RDY
Closed on RDY
U/D
L/R
Ready In
Open on RDY
Closed on RDY
U/D
L/R
L
Ready Out
Disabled Open
Disabled Closed
U/D
X
X
X
6
R
Carrier Gas
He
H2
N2
L/R
U/D
Column
Diameter, mm
+
-
L/R
U/D
Column
Length, m
+
-
L/R
U/D
Column
Outlet Pressure
1 Atm
Vacuum
L
U/D
X
X
X
X
L/R

Appendix D: OPTIC 3 Default Parameters

D.1 Standby and System Configuration Parameters

D.1.1 Standby Parameters

Parameter Name	Default Value	Allowed Range
Timeout	30:00 min:sec	0 - 120 min
Injector Temperature Control	Yes	Yes/No
Injector Temperature	35 °C	-180 ÷ 600 °C
Standby Column Flow	1.0 ml/min	0 - 20.0 ml/min
Standby Split Valve State	Splitless	Split/Splitless
Standby Split Flow	0.0 ml/min	0.0 ÷ 300 ml/min
Injector Cooling	OFF	ON/OFF
Standby Septum Purge Flow (OPTIC 3–D and OPTIC 3-DC)	0.0	0.0 ÷ 10.0 ml/min
Standby Auxiliary Flow (OPTIC 3–D and OPTIC 3–DC)	0.0	0.0 ÷ 100 ml/min
Standby Auxiliary Pressure (OPTIC 3–D and OPTIC 3-DC)	0.0	0 - 700 kPa
Cryotrap Temperature Control	Yes	Yes/No
Cryotrap Temperature	35 °C	-150 ÷ 350 °C
∗Auxiliary Output 1	Open	Open/Closed
∗Auxiliary Output 2	Open	Open/Closed
∗Auxiliary Output 3	Open	Open/Closed
∗Auxiliary Output 4	Open	Open/Closed
∗Auxiliary Output 5	Open	Open/Closed
∗Auxiliary Output 6	Open	Open/Closed
∗Auxiliary Output 7	Open	Open/Closed

* Accessible through the Evolution Workstation control software only (other settings also accessible from the OPTIC 3 control unit keypad).

D.1.2 System Configuration Parameters

Parameter Name	Default Value	Allowed Range
∗OPTIC 3 Access Control	No	Yes/No
∗Password		String maximum 11 characters
Maximum Temperature	200 °C	100 ÷ 600 °C
Communication Interface	LAN	Serial/LAN
Main EGC	Yes	Yes/No
Pressure Units	kPa	kPa, bar, PSI
Auxiliary EGC	No	Yes/No
Auxiliary EGC Control	Flow	Flow/Pressure
Septum Purge	No	Yes/No
Cryotrap	No	Yes/No
Keyboard Beeper	Enabled	Enabled/Disabled
∗∗LCD Contrast	100	0 ÷ 100
Reset Parameters	No	Yes/No

* Accessible through the Evolution Workstation control software only (other settings also accessible from the OPTIC 3 control unit keypad).

** Accessible from the OPTIC 3 control unit keypad only.

D.1.3 GC and Autosampler Input/Output Configuration

Parameter Name		Default Value	Allowed Range
OPTIC – Autosampler Ready Out		Closed On Ready	Closed On Ready/Open On Ready
OPTIC – GC Interface		OPTIC Slave	Master/Slave
Master	Ready In	Closed On Ready	Closed On Ready/Open On Ready
	Ready Out	Disabled Closed	Disabled Open/Disabled Closed
Slave	Ready In	Disabled	Disabled
	Ready Out	Closed On Ready	Closed On Ready/Open On Ready

D.1.4 GC Column Parameters

Parameter Name	Default Value	Allowed Range
Internal Column Diameter	0.32 mm	0 - 1 mm
Column Length	25 m	0 - 100 m
Column Outlet Pressure	1 Atm	1 Atm/Vacuum
Carrier Gas	He	He/N_2/H_2

Appendix E: Rear Panel Connections

Note: *Any pins that are not listed in the following tables are not connected.*

E.1 Serial Port

A 9-way, female, D-type connector. The function and pin connections are compatible with an IBM PC serial port. Use a crossed (null modem) cable for connection to the PC.

Pin	Function
2	TxD
3	RxD
5	GND

E.2 Gas Chromatograph Interface

These inputs and outputs are provided on a sub-D 15 pin male connector.

Pin	Function	Remarks
1	Ready In	
2	Run In	External contact closure. When the contacts are closed, OPTIC 3 enters the Run state
6,13	Ready Out	Relay output (max. 30 V/500 mA).
7,14	Run Out	Relay output (max. 30 V/500 mA). The contact closes for 1 sec when the Run Out signal is generated.
8,15	Integrator Run Out	Relay output (max. 30 V/500 mA). The contact closes for 1 sec when the Run Out signal is generated.
9,10,11,12	GND	

E.3 Autosampler interface and auxiliary inputs

These inputs and outputs are provided on a sub-D 15 pin female connector.

Pin	Function	Remarks
1,2	Ready Out	Relay output (max. 30V/500mA)
6	Run In	External contact closure. When the contacts are closed OPTIC 3 enters the Run state.
3	Auxiliary Input 1	
7	Auxiliary Input 2	
11	Auxiliary Input 3	
13	Auxiliary Input 4	
4,5,8,9,10,12,14,15	GND	

E.4 Auxiliary outputs

These outputs are provided on a sub-D 25 pin female connector.

Pin	Signal	Remarks
1	Power output	24 V ± 20 %. Max. current 250 mA,
13	Power output	5 V ± 20 %. Max. current 250 mA,
4(COM), 5(NO), 6(NC)	Auxiliary Out 1	Relay output (max. 30 V/500 mA)
7(COM), 8(NO), 9(NC)	Auxiliary Out 2	Relay output (max. 30 V/500 mA)
10(COM), 11(NO), 12(NC)	Auxiliary Out 3	Relay output (max. 30 V/500 mA)
14(COM), 15(NO), 16(NC)	Auxiliary Out 4	Relay output (max. 30 V/500 mA)
17(COM), 18(NO), 19(NC)	Auxiliary Out 5	Relay output (max. 30 V/500 mA)
20(COM), 21(NO), 22(NC)	Auxiliary Out 6	Relay output (max. 30 V/500 mA)
23(COM), 24(NO), 25(NC)	Auxiliary Out 7	Relay output (max. 30 V/500 mA)
2,3	GND	

Appendix F: Accessories, Consumables, and Spares

1. Injector liners

	Part number	Pack Size
Fritted liner, frit 15 mm	A100001	5
Liner for split injections (single necked)	A100092	5
Empty liner for splitless injections	A100049	5
Baffled liner for splitless injections	A100132	5
On-column liner for 0.32 mm columns	A100128	5
On-column liner for 0.53 mm columns	A100129	5
DMI liner with taper	L100011	5
Fritted liner (20 mm) with single taper	L100009	5
"A" type packed liner for large volume injections	A100060	5
"8270" packed liner for large volume injections	A100095	5
Multi-capillary liner for large volume injections	A100056	5
At-column liner for large volume injections	A100106	5

2. Consumables

Low Bleed White Septum, 11 mm	H100001	25
High Resistance Silicon O-Ring	3007-11605	20
Graphite Ferrule, 0.53 mm ID Column	H100003	10
Graphite Ferrule, 0.1-0.32 mm ID Column	H100004	10

3. Spares

Injector Bottom Nut, Stainless Steel	H200001	5
Standard Injector Septum Nut	H200002	1
Top Boss PTFE Insulator	A100067	10
1/8 - 1/16 PTFE Reducing Ferrule	A100030	10
OPTIC 3 Injector Body High Power	H400054	1
Injector Top Boss Assembly	H400019	1
Solvent Monitor-Filter Assy	H400057	1
GC Oven Thermocouple	H200005	1
OPTIC 3 Main EGC Channel	H400004	1

4. Interface cables

OPTIC 3-FOCUS	H400001	1
OPTIC 3-HP5890	H400002	1
OPTIC 3-Agilent 6890	H400003	1
OPTIC 3-Varian 3800	H400009	1
OPTIC 3-Trace GC	H400011	1
OPTIC 3-Shimadzu GC-2010 Interface Cable	H400034	1

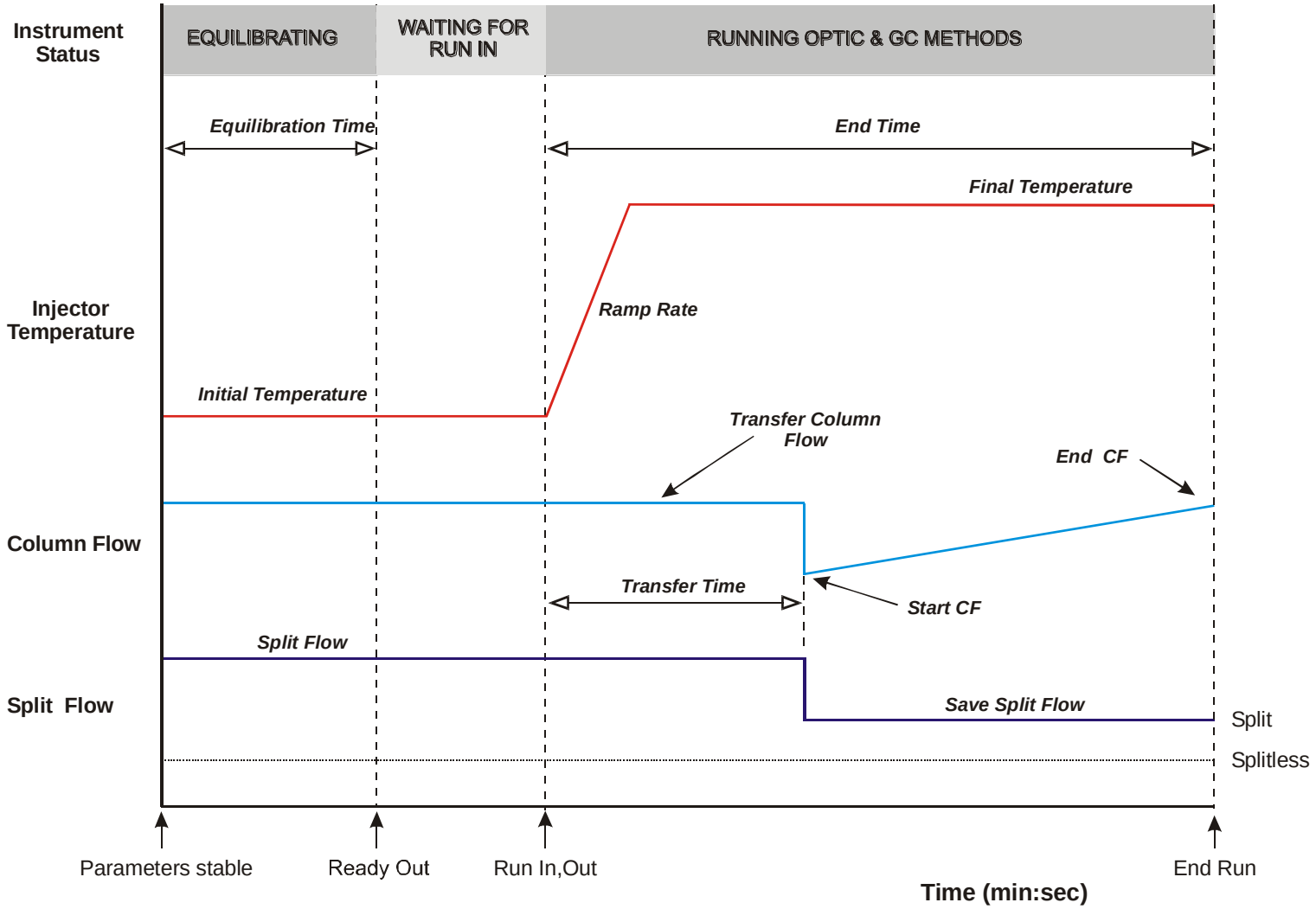
5. Accessories/upgrades

OPTIC 3 Auxiliary EGC Unit	H400005	1
OPTIC 3 Cryotrap Option, Heat Exch. Cooling	H500007	1
Improved Liquid CO2 Injector Cooling Option	H500009	1

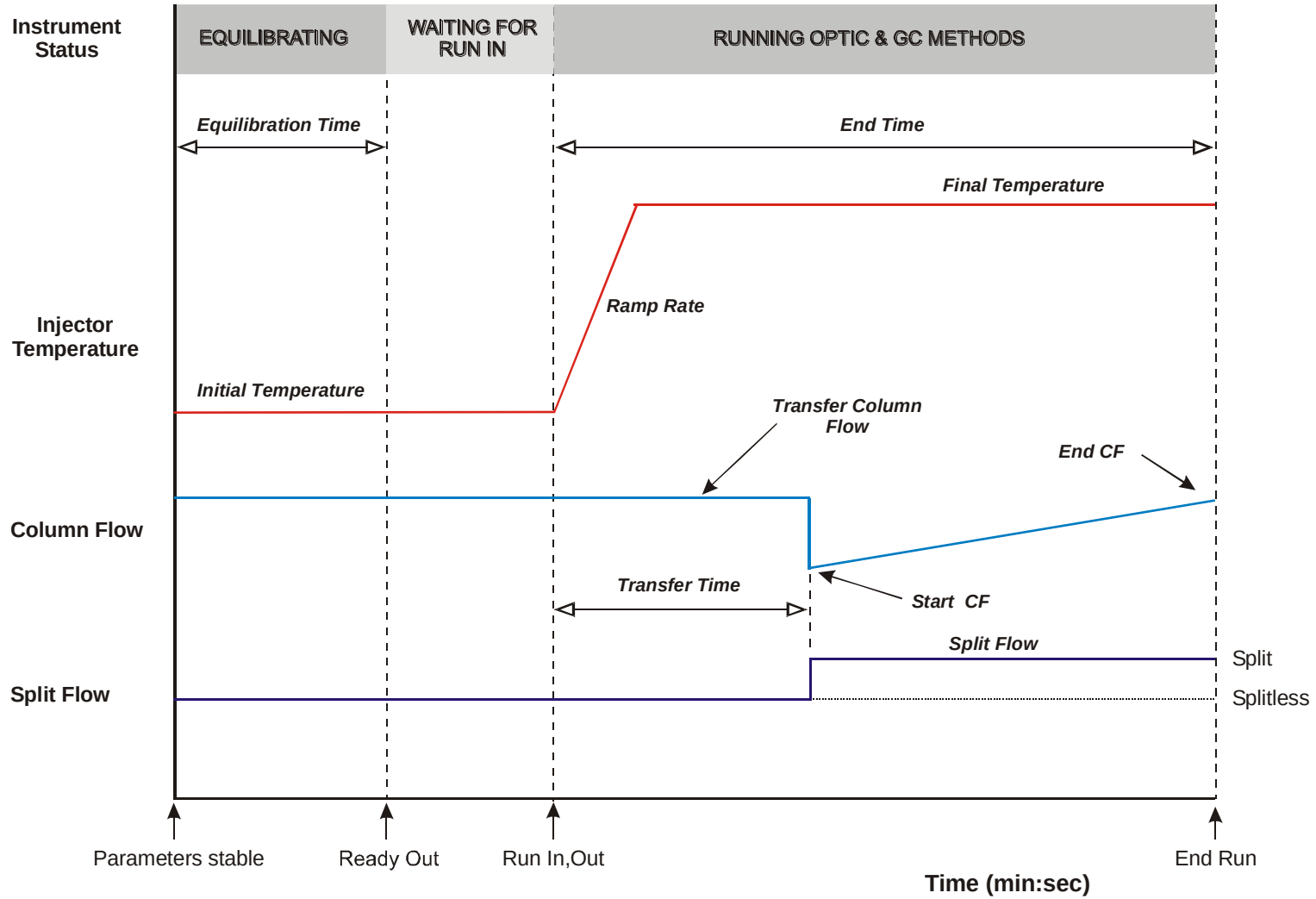
6. **Installation Kits** (including interface cables)

Agilent 6890/7890	H500002	1
HP 5890	H500010	1
Trace GC	H500011	1
Varian 3800	H500012	1
Shimadzu GC-2010	H500019	1

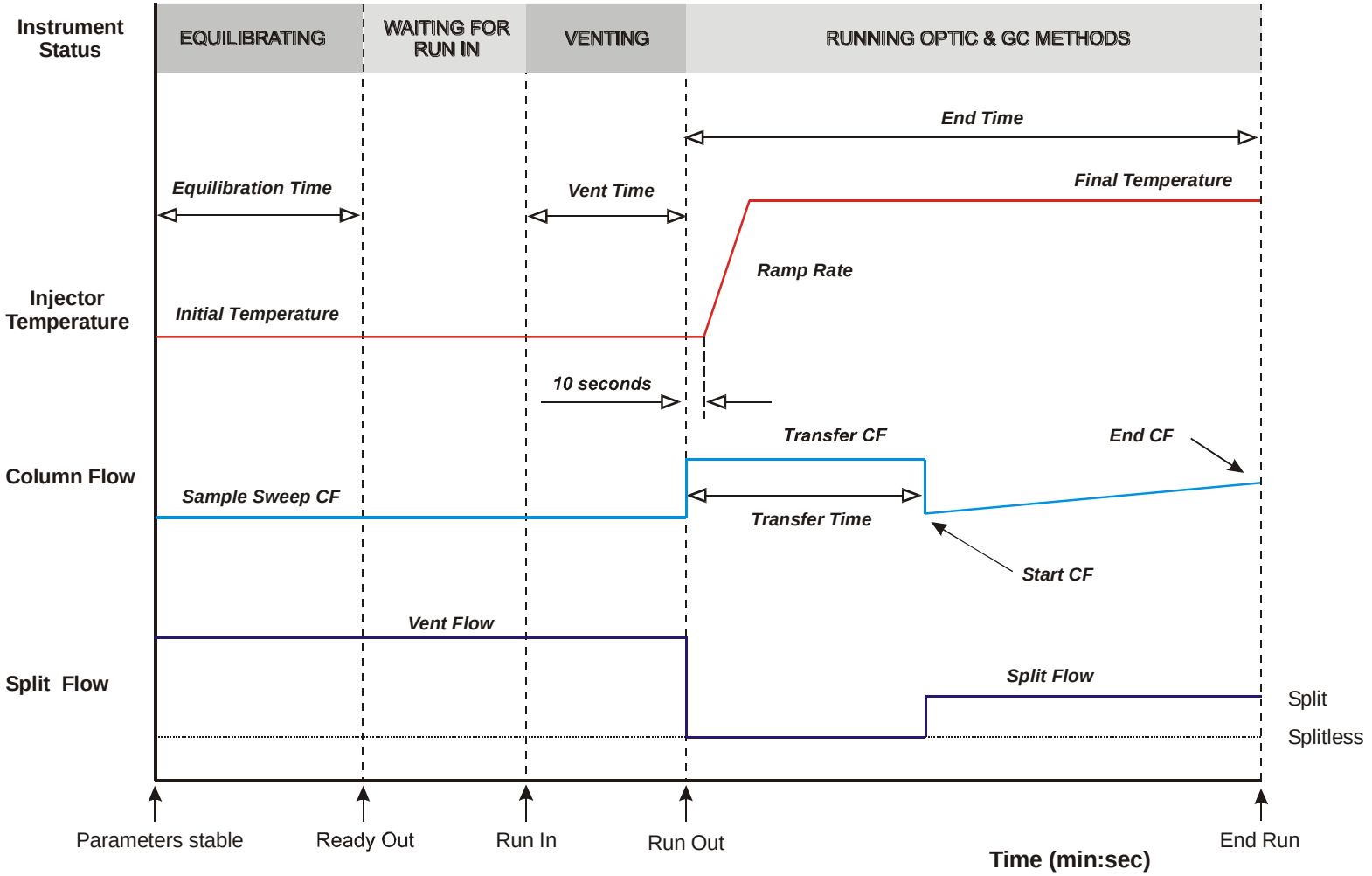
Appendix G: Examples of OPTIC 3 Method Profiles



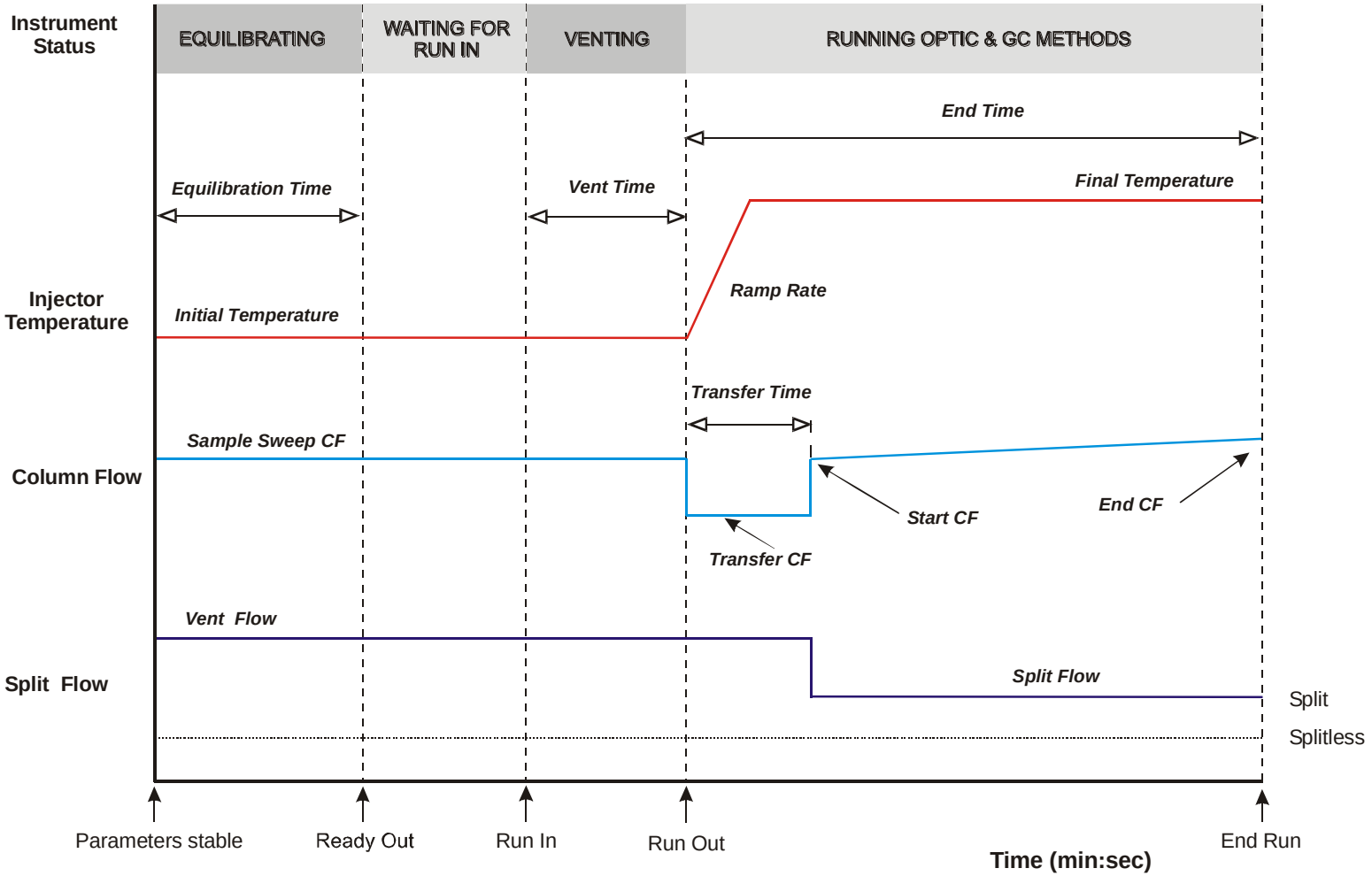
G.1 Split method



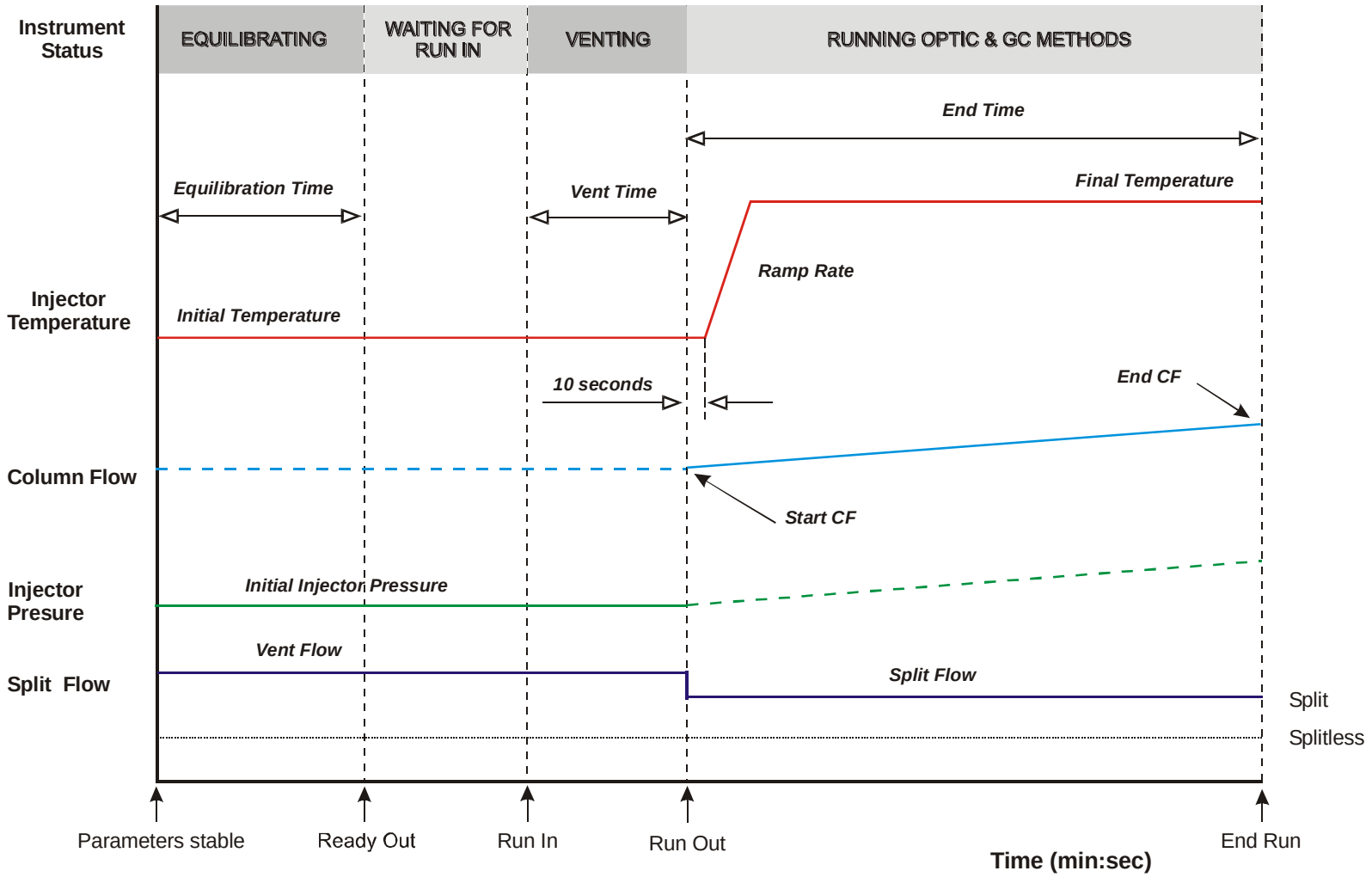
G.2 Splitless method



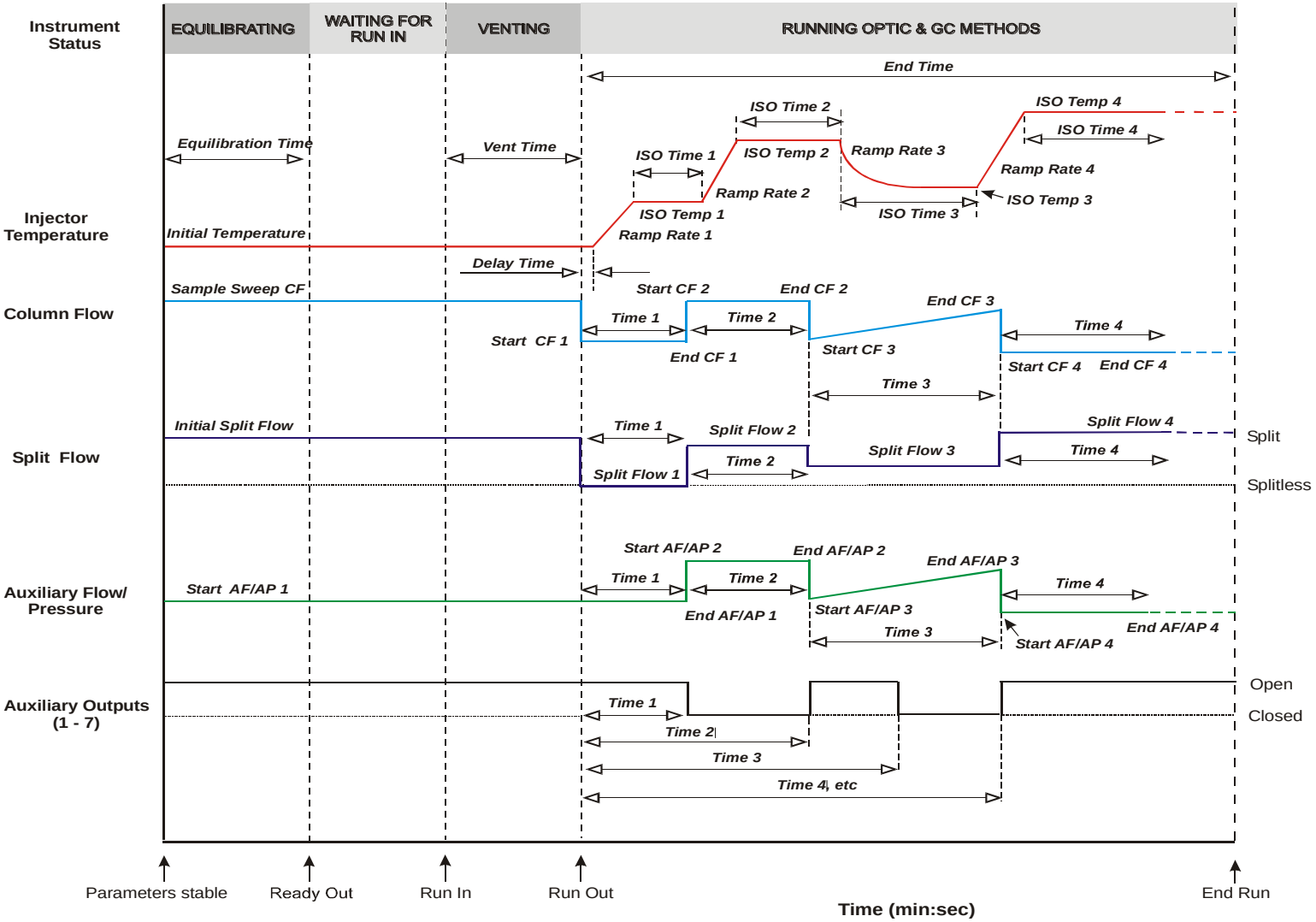
G.3 Large Volume method



G.4 LINEX-TD/LINEX-DMI methods



G.5 At-Column method



G.6 Expert method

Appendix H: Liner Selection Guide

Notes:

1. *All liners are compatible with OPTIC 2 as well as with OPTIC 3 standard injectors.*
2. *Silanisation of liners: Each liner can be deactivated using a high pressures silanisation process. To order silinazed liners, add an "S" to the part number of the liner. For example, A100001-S.*
3. *For information on liners for ATAS GL DTD unit, please send a request to info@atasgl.com.*

P/No A100001, Fritted Liner, frit at 15 mm from the liner bottom

This liner contains a medium porosity frit made from sintered, deactivated glass. This liner can be used for hot and cold split and splitless injections. In addition, if used with a suitable packing material the liner can be used for large volume injection, thermal desorption, solid sampling and pyrolysis applications. If packed, a bed depth of up to 25 mm can be employed and retained in place with a plug of deactivated glass or quartz wool. Pack size: 5

P/No A100092, Single-Necked Liner for Split Injections

This liner is specially made for hot or cold split injections. The liner's large internal diameter of 3.4 mm facilitates mixing of the sample with the carrier gas. The liner may be packed with a glass wool if desired. The bottom of the liner has a small slot to ensure free passage of gas to the split exit. Pack size: 5

P/No A100049, Liner for Splitless Injections

This liner is for hot and cold splitless injection of sample volumes below 1 µl. The narrow internal diameter of 1 mm ensures fast transfer of the sample to the column under splitless conditions. We advise this liner for SPME techniques. Pack size: 5

P/No A100132, Baffled Splitless Liner

This liner is for hot and cold splitless injection of sample volumes below 1 µl. The narrow internal diameter of 1 mm ensures fast transfer of the sample to the column under splitless conditions. With this liner the sample is not injected directly to the bottom of the injector. This liner will give better results for the high boiling components. Pack size: 5

P/No A100128, On-Column Liner for 0.32mm columns

This liner has a small ID with a tapered restriction 10 mm from the top. With a column installed into the restriction, true on-column injection can be performed. On-column provides the best possible reference when developing large volume injection methods. This liner can not be used with ATAS GL LINEX option. Pack size: 5

P/No A100129, On-Column Liner for 0.53mm columns

This liner is identical to A100128 but is for use with 0.53 mm ID columns. Liner can not be used with the LINEX option. Pack size: 5

P/No L100009, Fritted Liner with single taper and frit on 20 mm

This liner contains a medium porosity frit made from sintered, deactivated glass. It can be used for hot and cold split and splitless injections. In addition, if used with a suitable packing material the liner can be used for large volume injection, thermal desorption, solid sampling and pyrolysis applications. If packed, a bed depth of up to 25 mm can be employed and retained in place with a plug of deactivated glass or quartz wool. The taper will improve the sensitivity for the active samples. Pack size: 5

Liners for Large Volume Injections

These liners have properties that are very well understood by ATAS GL. They provide a reliable solution to the majority of LVI applications. Some liners are packed with proprietary packing materials, which have been especially developed for large volume injections.

P/No A100060, A-type Packed Liner for Large Volume Injections

The liner contains a highly deactivated packing material that combines a maximum operating temperature (350°C) with a high sample capacity (in excess of 100 µl in most cases). This liner has been found to be suitable for the following classes of compounds:

- Pesticides
- Triazine herbicides
- PAH's
- Mineral oils
- Derivatized acid herbicides
- Drugs of abuse

Pack size: 5

P/No A100095, "8270" Packed Liner for Large Volume Injections

This liner contains a highly deactivated packing that combines a maximum operating temperature of 350 °C with a high sample capacity (in excess of 100µl in most cases). This liner has been developed for the EPA 8270 method. Pack size: 5

P/No A100056, Multi-Capillary Liner for Large Volume Injection

This liner is very inert and capable of operation up to 600°C. As such, it is suitable for the large volume injection of high molecular weight compounds. The capacity of the liner is lower than that of a packed liner (25-50 µl, depending upon solvent used). To maximize its capacity, the liner should be used in combination with a syringe fitted with a dome tipped, side-hole needle. This liner is suitable for the following applications:

- High MW hydrocarbons
- Parathyroid's
- OCP pesticides

Pack size: 5

P/No A100106, At-Column Liner

This liner is used for the At-Column technique. The At-Column technique is a patented rapid large volume injection technique with the advantages of the on-column technique. Liner can be used with the LINEX option. Pack size: 5

P/No A100133, Sintered Glass Liner with taper

This liner is both very inert and capable of operation up to 600°C. As such it is suitable for the large volume injection of high molecular weight compounds. The capacity of the liner is lower than that of a packed liner (about 50 µl, depending upon solvent used). To maximize its capacity, the liner should be used in combination with a syringe fitted with a dome tipped, side-hole needle. Pack size: 5

LINEX-DMI Liners

P/No L100011, LINEX DMI Liner

This liner has 3 small indentations inside made to hold a micro-vial. The liner is used for Difficult Matrix Introduction (DMI) in combination with L100010 and D100005. DMI technique extends the OPTIC capability to the analysis of dirty liquid samples containing non-volatiles and/or solid-like suspended matrix. Row sample or roughly filtered extract placed into a sample container (micro-vial, p/n D100005), which is inserted into the liner. DMI Needle Guide (p/n L100010) is used to ensure an accurate sample injection. Pack size: 5

P/No L1000012, LINEX DMI Needle Guide Straight

Syringe needle guide used in combination with L100011. Pack size: 5

P/No D100005, DMI Liner Insert (micro-vial)

This micro-vial is used as a sample container in the DMI technique. It is used in combination with L100009. Pack size: 100

LINEX-TD Liners

P/No L100009, Fritted Liner with single taper and frit at 20 mm

This liner contains a medium porosity frit made from sintered, deactivated glass. It can be used for the LINEX direct (in-injector) thermal desorption of all kind of solid samples. If used with a suitable packing material the liner can be used for thermal desorption of gaseous samples. If packed, a bed depth of up to 25 mm can be employed and retained in place with a plug of deactivated glass or quartz wool. Pack size: 5