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Varian, Inc.  
679 Springvale Rd  
Mulgrave, Victoria, 3170  
Australia

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# ProStar 355 Refractive Index Detector

## Operation Manual

Overvoltage category II

Pollution degree 2

Safety class 1 (EN61010-1)



**Varian, Inc. offices**

Varian has offices in most countries. The major offices for chromatography are listed below:

Varian Australia Pty Ltd (Manufacturing site)

679 Springvale Road  
Mulgrave, Victoria 3170  
Australia

Phone: + 61 3 9560 7133  
Fax: + 61 3 9560 7950

Varian, Inc.  
Analytical Instruments  
2700 Mitchell Dr.  
Walnut Creek, CA 94598  
USA

Phone: 1.800.FOR.HPLC  
International Phone: +1.925.939.2400  
Fax: +1.925.945.2360

Varian, B.V.  
Herculesweg 8  
4338 PL, Middelburg  
The Netherlands  
Phone: +31. 0118.67.1000  
Fax: +31. 0118.62.3193

**Internet**

The Varian, Inc. Internet home page can be found at:  
[www.varianinc.com](http://www.varianinc.com)

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# ERC INC.

R006 Rev.0

## DECLARATION OF CONFORMITY

Supplier's Name : ERC, Inc.  
Supplier's Address : 5-8-6 Nishi-Aoki, Kawaguchi City,  
Saitama 332-0035 Japan

### Declares, that the product

Product Name : Refractive Index Detector  
Model Number(s) : ProStar 355

### Conforms to the following product specifications:

Safety: IEC 61010-1: 2001(Second edition); EN 61010-1: 2001(Second edition)  
Safety requirements for electrical equipment for measurement, control and laboratory  
use  
Part 1. General requirements.

EMC: EN 61326: 1997 + A1: 1998+A2: 2001  
EMC requirements for electrical equipment for measurement, control and laboratory use  
Part 1. General requirements.

1) EMI: CISPR 11 (1997), CISPR 14-1 (2000), CISPR 16-1 (1999), CISPR 16-2 (1996),  
CISPR 22 (1997)  
IEC 61000-3-2 (2000), IEC 61000-3-3 (1995)

2) EMS: IEC 61000-4-2 (1995), IEC 61000-4-3 (1995), IEC 61000-4-4 (1995),  
IEC 61000-4-5 (1995), IEC 61000-4-6 (1996), IEC 61000-4-8 (1993),  
IEC 61000-4-11 (1994)

Saitama Japan

January 25, 2005



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Location

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Date

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Y. Ishii, Quality Assurance Manager



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# Safety information

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## Operating Instructions

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

---

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

---











### **CAUTION**

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

### **WARNING**

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

Warnings are accompanied by a triangular symbol indicating the type of warning.

Warning Symbol	Warning Description
 <b>WARNING: SHOCK HAZARD</b>	Hazardous voltages are present inside instrument. Disconnect from main power before removing screw-attached panels.
 <b>WARNING: CHEMICAL HAZARD</b>	Hazardous chemicals may be present. Avoid contact, especially when replenishing reservoirs. Use proper eye and skin protection.
 <b>WARNING: BURN HAZARD</b>	Very hot or cryogenically cold surfaces may be exposed. Use proper skin protection.
 <b>WARNING: EYE HAZARD</b>	Eye damage could occur either from flying particles, chemicals, or UV radiation. Use proper eye and face protection.
 <b>WARNING: FIRE HAZARD</b>	The potential for fire may be present. Follow manual instructions for safe operation.
 <b>WARNING: EXPLOSION HAZARD</b>	The potential for explosion may exist because of type of gas or liquid used.
 <b>WARNING: RADIATION SOURCE</b>	Ionizing radiation source is present. Follow manual instructions for safe operation.
 <b>WARNING: MOVING PARTS</b>	Keep hands and fingers away.
 <b>WARNING: HEAVY WEIGHT</b>	A heavy object is present. Avoid back strain or injury by following all precautions for lifting heavy objects.
 <b>WARNING: BREATHING HAZARD</b>	Hazardous fumes may be present. Do not inhale fumes. Wear a face mask if required.






This symbol may be used on warning labels attached to the instrument. When you see this symbol you must refer to the relevant operation or service manual for the correct procedure referred to by that warning label.



## Information symbols

The following symbols appear on the ProStar 335 detector to provide you with additional information:

- I Mains power on
- Mains power off
-  Single phase alternating current
-  Fuse
-  Appears on the rear of the product to indicate that the product complies with the requirements of one or more EU Directives.

---

## General Safety Precautions

Follow these safety practices to ensure safe equipment operation.

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

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## Federal Communications Commission advisory

The following is a Federal Communications Commission advisory:

### CAUTION

This equipment has been tested and found to comply with the limits for a Class B digital device, pursuant to part 15 of the FCC Rules. These limits are designed to provide reasonable protection against harmful interference in a residential installation. This equipment generates, uses and can radiate radio frequency energy and, if not installed and used in accordance with the instructions, may cause harmful interference to radio communications. However, there is no guarantee that interference will not occur in a particular installation. If this equipment does cause harmful interference to radio or television reception, which can be determined by turning the equipment off and on, the user is encouraged to try to correct the interference by one or more of the following measures:

- Reorient or relocate the receiving antenna.
- Increase the separation between the equipment and receiver.
- Connect the equipment into an outlet on a circuit different from that to which the receiver is connected.
- Consult the dealer or an experienced radio/TV technician for help.



**WARNING:  
EXPLOSION HAZARD**

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



**WARNING:  
FIRE HAZARD**



**WARNING:  
EYE HAZARD**



**WARNING:  
CHEMICAL HAZARD**

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

## Electrical Hazards



### **WARNING: SHOCK HAZARD**

- Disconnect the instrument from all power sources before removing protective panels to avoid exposure to potentially dangerous voltages. Panels or covers which are retained by screws on the detector may be opened **ONLY** by Varian-trained, Varian-qualified, or Varian-approved Customer Service Representatives. Consult the manuals or product labels supplied with your PC to determine which parts are operator-accessible.
- When it is necessary to use a non-original power cord plug, make sure the replacement cord adheres to the color coding and polarity described in the manual and all local building safety codes.
- Good grounding/earthing is essential to avoid a potentially serious electric shock hazard. Ensure that there is an integral ground connection between the metal base of the detector and the 3 pin earth-grounded receptacle. Consult the manuals or product labels supplied with your PC for the relevant grounding requirements.
- Replace blown fuses with fuses of the size and rating shown on the fuse panel or in the manual.
- Replace faulty or frayed power cords immediately with the same type and rating.
- Make sure that voltage sources and line voltage match the value for which the instrument is wired.
- Avoid using power supplies from a source that may be subject to electrical or RF interference from other services (for example, large electrical motors, elevators and welders).

---

## Compressed Gas Cylinders

- Store and handle compressed gases carefully and in strict adherence to safety codes.
- Secure cylinders to an immovable structure or wall.
- Store and move cylinders in an upright, vertical position. Before transport, remove regulators and install cylinder cap.
- Store cylinders in a well-ventilated area away from heat, direct sunshine, freezing temperatures, and ignition sources.
- Mark cylinders clearly so there is no doubt as to their contents.
- Use only approved regulators and connections.
- Use only connector tubing that is chromatographically clean (Varian part number 0391832600) and has a pressure rating significantly greater than the highest outlet pressure from the regulator.

---

## GC Safety Practices

### *Exhaust System*

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



**WARNING:  
BREATHING HAZARD**

If you do install ducting:

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

### ***Radioactive Source Detectors***



#### **WARNING: RADIATION SOURCE**

- Read carefully and comply with all NOTES, CAUTIONS, and WARNINGS in the Ni63 ECD manual.
- Perform the tests for removable radioactive contamination described in the Ni63 ECD manual.
- Comply with leak test schedules and procedures.

### ***Burn Hazard***



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

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

---

## LC Safety Practices

### *High Pressure Hazard*



**WARNING:  
EYE HAZARD**



**WARNING:  
EXPLOSION HAZARD**

If a line ruptures, a relief device opens, or a valve opens accidentally under pressure, potentially hazardous high liquid pressures can be generated by the pump causing a high velocity stream of volatile and/or toxic liquids.

- Wear face protection when you inject samples or perform routine maintenance.
- Never open a solvent line or valve under pressure. Stop the pump first and let the pressure drop to zero.
- Use shatter-proof reservoirs capable of operating at 50/60 psi.
- Keep the reservoir enclosure closed when the reservoir is under pressure.
- Read and adhere to all NOTES, CAUTIONS, and WARNINGS in the manual.

### *Flash Chromatography*

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

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

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

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

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

### ***Ultraviolet Radiation***

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



**WARNING:  
EYE HAZARD**

For continued protection:

- Ensure that protective lamp covers of variable and fixed wavelength detectors are in place during operation.
- Do not look directly into detector fluid cells or at the UV light source. When inspecting the light source or fluid cell, always use protective eye covering such as borosilicate glass or polystyrene.
- Ozone can be generated by radiation from the source lamps. Exposure to ozone can result in severe irritation to the skin, eyes, and upper respiratory system. The maximum permissible exposure level is 0.1 ppm (0.2 mg/m<sup>3</sup>) for 8 hours per day. ALWAYS ventilate the area surrounding the detector such that the concentration of ozone does not exceed the maximum permissible level. All venting must be to outside air, never within the building.

---

## **CE-compliant products**

Your ProStar 335 Photodiode Array Detector has been designed to comply with the requirements of the Electro-magnetic Compatibility (EMC) Directive and the Low Voltage (electrical safety) Directive (commonly referred to as the LVD) of the European Union.

Varian has confirmed that the product complies with the relevant Directives by testing a prototype against the prescribed EN (European Norm), IEC or CISPR standards.

Proof that the product complies with the Directives is indicated by:

- The CE marking appearing on the rear of the product.
- The documentation package that accompanies the product containing a copy of the Declaration of Conformity. This Declaration is the legal declaration by Varian that the product complies with the Directives, and also shows the standards to which the product was tested to demonstrate compliance. It is also signed by Varian's Authorized Representative in the EU, and by the representative of the manufacturing plant.

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## **Spare Parts Availability**

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

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## **Service Availability**

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



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## Varian, Inc. Analytical Instruments Sales Offices

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

**Argentina**  
Buenos Aires  
Tel. +54.11.4.783.5306

**Australia**  
Mulgrave, Victoria  
Tel. +61.3.9566.1134

**Austria**  
Vösendorf bei Wien  
Tel. +43.1.699.9669

**Benelux**  
Bergen Op Zoom  
Tel. +31.164.282.800

**Brazil and  
Latin America (S)**  
São Paulo  
Tel. +55.11.820.0444

**Canada**  
Mississauga, Ontario  
Tel. 800.387.2216

**China**  
Beijing  
Tel. +86.106209.1727

**Europe**  
Middelburg, The Netherlands  
Tel. +31.118.671.000

**France**  
Les Ulis Cédex  
Tel. +33.1.6986.3838

**Germany**  
Darmstadt  
Tel. +49.6151.7030

**India**  
Mumbai  
Tel. +91.22.857.0787/88/89

**Italy**  
Torino  
Tel. +39.011.997.9111

**Japan**  
Tokyo  
Tel. +81.3.5232.1211

**Korea**  
Seoul  
Tel. +82.2.345.22452

**Mexico and  
Latin America (N)**  
Mexico City  
Tel. +52.5.523.9465

**Russian Federation**  
Moscow  
Tel. +7.095.937.4280

**Spain**  
Madrid  
Tel. +34.91.472.7612

**Sweden**  
Solna  
Tel. +46.8.445.1620

**Switzerland**  
Varian AG  
Tel. +41.848.803.800

**Taiwan**  
Taipei Hsien  
Tel. +886.2.698.9555

**United Kingdom and  
Ireland**  
Walton-on-Thames  
Tel. +44.1932.898000

**Venezuela**  
Valencia  
Tel. +58.41.257.608

**United States**  
Walnut Creek,  
California, USA  
Tel. 1.800.926.3000  
(GC and GC/MS)  
Tel. +1.800.367.4752  
(LC)



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



# Instrucciones de Seguridad

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## Instrucciones de Operación

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

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

---

### **PRECAUCION!**

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

### **ATENCION**

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

## Símbolo



**ATENCIÓN**  
**PELIGRO DE**  
**DESCARGA ELÉCTRICA**

## Descripción

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



**ATENCIÓN**  
**PELIGRO QUÍMICO**

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



**ATENCIÓN**  
**PELIGRO DE**  
**QUEMADURAS**

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



**ATENCIÓN**  
**PELIGRO PARA LOS OJOS**

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



**ATENCIÓN**  
**PELIGRO DE FUEGO**

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



**ATENCIÓN**  
**PELIGRO DE EXPLOSIÓN**

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



**ATENCIÓN**  
**PELIGRO DE RADIACIÓN**

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



**ATENCIÓN**  
**PARTES EN MOVIMIENTO**

Mantenga alejados los dedos y las manos.

---

## Precauciones Generales de Seguridad

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

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

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

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

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

---

## **Peligros Eléctricos**

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

---

## **Botellas de Gas Comprimido**

- Guarde y maneje las botellas de gas con cuidado y de acuerdo con las normas de seguridad.
- Asegure las botellas a una estructura inmóvil ó a la pared.
- Guarde y mueva las botellas en posición vertical. Retire los reguladores antes de transportarlas.
- Guarde las botellas en un área ventilada, lejos de fuentes de calor, de luz solar directa y de temperaturas extremadamente bajas.
- Identifique las botellas claramente para evitar cualquier duda sobre su contenido.
- Utilice sólo reguladores y conexiones aprobadas.
- Utilice sólo tubos de conexión cromatográficamente limpios (Varian p/n 0391832600) y que tengan una graduación de presión significativamente mayor que la mayor presión del regulador.

## **GC Prácticas de Seguridad**

### ***Sistema de Extracción***

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

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

### ***Detectores con fuentes radiactivas***

- Lea con cuidado y cumpla todas las NOTAS, PRECAUCION, y ATENCION del Manual del Detector Ni63 ECD.
- Realice los test de contaminación radioactiva descritos en el Manual del Detector Ni63 ECD.
- Cumpla con los plazos y procedimientos de test de fugas.

### ***Peligro de Quemaduras***

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

---

## **LC Prácticas de Seguridad**

### ***Peligro de Alta Presión***

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

- Lleve protección facial cuando inyecte muestras ó realice mantenimiento de rutina.
- Nunca abra una línea ó una válvula bajo presión. Apague la bomba antes y deje que la presión baje a cero.
- Utilice depósitos irrompibles que sean capaces de operar a 50/60 psi.
- Mantenga cerrada la junta del depósito cuando se haya bajo presión.
- Lea y cumpla todas las NOTA, PRECAUCION, y ATENCION del manual.

### ***Cromatografía Flash***

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

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

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

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

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



### ***Radiación Ultravioleta***

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

Para una correcta protección:

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

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### **Disponibilidad de Recambios**

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

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### **Disponibilidad de Servicio**

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

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## Oficinas de Instrumentación Analítica Varian

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

**Argentina**

Buenos Aires  
Tel. +54.11.4.783.5306

**Australia**

Mulgrave, Victoria  
Tel. +61.3.9566.1134

**Austria**

Vösendorf bei Wien  
Tel. +43.1.699.9669

**Benelux**

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**Brazil and  
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**Canada**

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**China**

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**Korea**

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**Mexico and  
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**Russian Federation**

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**United States**

Walnut Creek,  
California, USA  
Tel. 1.800.926.3000  
(GC and GC/MS)  
Tel. +1.800.367.4752  
(LC)

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

# Informations et mesures de sécurité

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## Instructions de fonctionnement

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

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

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### MISE EN GARDE

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

### AVERTISSEMENT

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

## Symboles d'avertissement



**ATTENTION  
RISQUE  
D'ELECTROCUTION**



**ATTENTION  
SUBSTANCES  
CHIMIQUES DANGER**



**ATTENTION  
RISQUE DE BRÛLURES**



**ATTENTION  
DANGER POUR  
LES YEUX**



**ATTENTION  
RISQUE D'INCENDIE**



**ATTENTION  
RISQUE D'EXPLOSION**



**ATTENTION  
SOURCE DE RADIATION**



**ATTENTION  
PIECES EN MOUVEMENT**

## Description

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

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

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

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

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

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

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

Garder les mains et les doigts hors de portée.

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## Précautions générales en matière de sécurité

Les pratiques suivantes garantissent une utilisation sans risques du matériel:

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

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

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

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

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## Risques de chocs électriques

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

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## Bouteilles à gaz comprimé

- Ranger et manipuler les bouteilles à gaz comprimé avec précaution et conformément aux normes de sécurité.
- Fixer les bouteilles à gaz comprimé à un mur ou à une structure inamovible.
- Ranger et déplacer les bouteilles à gaz comprimé en position verticale. Avant de transporter les bouteilles à gaz comprimé, retirer leur régulateur.
- Ranger les bouteilles dans un endroit bien ventilé et à l'abri de la chaleur, des rayons directs du soleil, du gel ou des sources d'allumage.
- Marquer les bouteilles de manière à n'avoir aucun doute quant à leur contenu.
- N'utiliser que des connexions et régulateurs agréés.
- N'utiliser que des tuyaux de raccordement propres sur le plan chromatographique (Varian P/N 0391832600) et pouvant supporter des pressions sensiblement plus élevées que la plus haute pression de sortie du régulateur. Store and handle compressed gases carefully and in strict adherence to safety codes.

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## Mesures de sécurité en CPG

### ***Système d'échappement***

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

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

### ***Détecteurs à source radioactive***

- Se conformer au manuel d'utilisation de l'ECD Ni63, en particulier à ses NOTES, MISES EN GARDE ET AVERTISSEMENTS.
- Effectuer les tests de décontamination radioactive décrits dans le manuel d'utilisation de l'ECD Ni63.
- Se conformer aux procédures et au calendrier des essais d'étanchéité.

### ***Risque de brûlures***

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

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## Mesures de sécurité en CPL

### ***Risques liés aux hautes pressions***

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

- Mettre un masque de protection lors de l'injection des échantillons ou en effectuant les opérations de maintenance de routine.
- Ne jamais déconnecter un conduit de solvant ou une vanne sous pression. Arrêter préalablement la pompe et laisser la pression descendre à zéro.
- Utiliser des réservoirs incassables à 50/60 psi.
- Laisser l'enceinte du réservoir fermée lorsque le réservoir est sous pression.
- Se conformer aux NOTES, MISES EN GARDE ET AVERTISSEMENTS du manuel d'utilisation.

### ***Chromatographie Flash***

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

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

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

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

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



### ***Radiations ultraviolettes***

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

Pour s'assurer une protection permanente:

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

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### **Disponibilité des pièces de rechange**

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

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### **Service d'assistance à la clientèle**

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

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## Points de vente des instruments analytiques Varian

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

### **Argentina**

Buenos Aires  
Tel. +54.11.4.783.5306

### **Australia**

Mulgrave, Victoria  
Tel. +61.3.9566.1134

### **Austria**

Vösendorf bei Wien  
Tel. +43.1.699.9669

### **Benelux**

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Tel. +44.1932.898000

### **Venezuela**

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Tel. +58.41.257.608

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Tel. 1.800.926.3000  
(GC and GC/MS)  
Tel. +1.800.367.4752  
(LC)



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# Informazioni sulla Sicurezza

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## Instruzioni per l'Uso

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

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

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### **ATTENZIONE**

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

### **ATTENZIONE**

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

## Segnali di ATTENZIONE



**ATTENZIONE**  
Pericolo di folgorazioni



**ATTENZIONE**  
ESPOSIZIONE A  
SOSTANZA CHIMICHE



**ATTENZIONE**  
Pericolo di scottature



**ATTENZIONE**  
PERICOLO PER  
GLI OCCHI



**ATTENZIONE**  
Pericolo di incendio



**ATTENZIONE**  
Pericolo di esplosioni



**ATTENZIONE**  
Pericolo di radiazioni



**ATTENZIONE**  
Parti in movimento

## Descrizione del Pericolo

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

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

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

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

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

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

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

Non tenere le mani o le dita vicino.

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## Norme di Sicurezza

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

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

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

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

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

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## Pericoli Elettrici

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

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## Bombole dei Gas

- Occorre prestare molta attenzione quando si spostano bombole di gas compressi. Rispettare tutte le norme di sicurezza.
- Assicurare le bombole ad una parete o ad una struttura fissa.
- Spostare e conservare le bombole sempre in posizione verticale. Togliere i manometri prima di spostare le bombole.
- Conservare le bombole in un'area ben ventilata, non infiammabile, lontana da sorgenti di calore, non esposta a temperature troppo fredde o alla luce diretta del sole.
- Evidenziare in modo chiaro e che non lasci dubbi il contenuto di ogni bombola.
- Usare solo manometri e raccordi di qualità.
- Usare solo tubazioni cromatograficamente pulite (Numero di Parte Varian 0391832600) e calibrate per pressioni superiori a quella massima di uscita dal manometro.

## **Procedure di Sicurezza in GC**

### ***Scarico dei Gas***

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

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

### ***Rivelatori a Sorgente Radioattiva***

- Leggere e rispettare tutte gli avvisi di NOTA, CAUTELA e ATTENZIONE riportati nel manuale del rivelatore ECD al Ni63.
- Eseguire tutti i test di contaminazione radioattiva rimovibile descritti nel manuale dell'ECD al Ni63.
- Rispettare tutte le procedure e le scadenze di verifica per eventuali perdite.

### ***Pericolo di Scottature***

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

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## Procedure di Sicurezza in LC

### ***Pericolo di Alte Pressioni***

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

- E' opportuno adottare un sistema di protezione del viso quando si inietta il campione o si esegue una manutenzione routinaria del sistema.
- Non smontare mai una linea del solvente od una valvola quando il sistema è sotto pressione. Fermare prima la pompa ed aspettare che la pressione scenda a zero.
- Usare dei contenitori per solventi infrangibili ed in grado di lavorare a 50/60 psi.
- Quando i contenitori sono sotto pressione, usare una protezione esterna.
- Leggere e rispettare tutti gli avvisi di NOTA, CAUTELA e ATTENZIONE.

### ***Cromatografia Flash***

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

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

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

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

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



### **Radiazioni Ultraviolette**

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

Per una protezione sicura:

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

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### **Disponibilità delle Parti di Ricambio**

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

---

### **Servizi Tecnico**

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

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## Uffici Vendite della Divisione Strumenti Analitici della Varian

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

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Buenos Aires  
Tel. +54.11.4.783.5306

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Mulgrave, Victoria  
Tel. +61.3.9566.1134

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Tel. +43.1.699.9669

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Varian AG  
Tel. +41.848.803.800

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Tel. +886.2.698.9555

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Tel. +44.1932.898000

### Venezuela

Valencia  
Tel. +58.41.257.608

### United States

Walnut Creek,  
California, USA  
Tel. 1.800.926.3000  
(GC and GC/MS)  
Tel. +1.800.367.4752  
(LC)



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# Sicherheitsinformationen

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## Arbeitsanleitungen

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

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

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### **ACHTUNG**

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

### **WARNUNG**

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

## Warnungssymbol



**WARNUNG  
ELEKTRISCHER  
SCHLAG**



**WARNUNG  
CHEMISCHE GEFAHR**



**WARNUNG  
VERBRENNUNGSGEFAHR**



**WARNUNG  
AUGENVERLETZUNG**



**WARNUNG  
FEUERGEFAHR**



**WARNUNG  
EXPLOSIONSGEFAHR**



**WARNUNG  
STRAHLUNGSQUELLE**



**WARNUNG  
BEWEGTE TEILE**

## Warnungsbeschreibung

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

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

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

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

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

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

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

Bleiben Sie mit Ihren Händen und Fingern weg.

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## Allgemeine Sicherheitsmaßnahmen

Befolgen Sie diese Sicherheitspraktiken für eine gefahrlose Gerätebenutzung.

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

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

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

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

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## Elektrische Gefahren

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

---

## Gasdruckflaschen

- Lagern und handhaben Sie komprimierte Gase vorsichtig und in strikter Einhaltung der Sicherheitsvorschriften.
- Befestigen Sie die Gasflaschen an feststehenden Aufbauten oder an Wänden.
- Lagern und transportieren Sie Gasflaschen in aufrechter Stellung. Druckregler zuvor abnehmen.
- Lagern Sie Gasflaschen in gut durchlüfteten Räumen, weit genug weg von Heizungen, direktem Sonnenschein, Frosttemperaturen und Entzündungszonen.
- Kennzeichnen Sie die Flaschen so eindeutig, daß kein Zweifel über deren Inhalt bestehen kann.
- Benutzen Sie nur geprüfte Druckminderer und Verbindungsstücke.
- Benutzen Sie nur chromatographisch reines Verbindungsrohr (Varian part number 0391832600), das wesentlich höheren Druck als den höchsten Ausgangsdruck des Druckminderers aushält.

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## GC Sicherheitspraktiken

### **Abgassystem**

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

- Benutzen Sie nur feuerfeste Führungen.
- Installieren Sie ein Gebläse am Ausgang.
- Ordnen Sie die Ansaugöffnung so an, daß ihre Erschütterungen oder Luftströmungen nicht die Detektorfunktion beeinträchtigen.
- Prüfen Sie regelmäßig die einwandfreie Arbeitsweise der Abgasführung.
- Sorgen Sie für gute Entlüftung im Laborbereich.

### **Radioaktive Detektoren**

- Lesen Sie sorgfältig und befolgen Sie alle HINWEISE, ACHTUNGEN und WARNUNGEN im Ni63 ECD Handbuch.
- Führen Sie die Tests für zu beseitigende radioaktive Kontamination durch, die im Ni63 ECD Handbuch beschrieben sind.
- Erfüllen Sie die Zeitpläne und Verfahren zur Dichtigkeitsprüfung.

### **Verbrennungsgefahr**

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

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## LC Sicherheitspraktiken

### **Gefahr durch hohen Druck**

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

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

### **Blitzlicht-Chromatographie**

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

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

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

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

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



### **Ultraviolette Strahlung**

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

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

---

### **Verfügbarkeit von Ersatzteilen**

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

---

### **Serviceverfügbarkeit**

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

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## Varian, Inc. Analytical Instruments Verkaufsbüros

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

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# Introduction

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## General Description

The ProStar 355 differential refractometer is a high-performance universal detector designed for analyses requiring the continuous monitoring of the refractive index of a flowing liquid with respect to a reference. Its small cell volumes, high sensitivity, excellent temperature control and flexible control options make it well-suited for use as a detector in automated and manual high performance liquid chromatography.

The ProStar 355 is a deflection or Snell type refractive index detector. Snell's law states that a parallel light beam, when passing through a dielectric interface separating two media of different refractive index at an angle of incidence greater than zero, will be refracted as a function of the magnitude of difference of the refractive indices of the two media. See figure below.

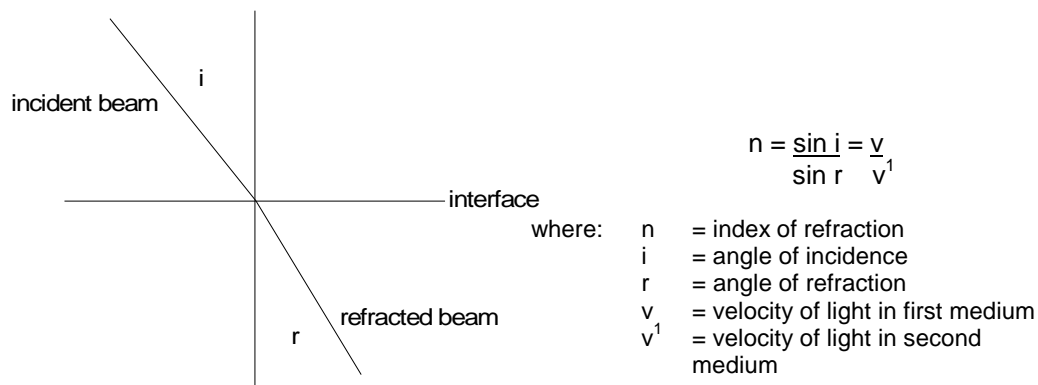


Figure 1 Snell's Law

Light from a low-power, long-lifetime tungsten lamp is collimated by a lens and slit and passed through reference and sample cells, reflected off a mirror, passed back through the optical cells, and focused by lenses onto a pair of photosensing diodes.

During operation, the ProStar 355 reference and sample cells are filled with mobile phase. The reference cell is then isolated from the flow path and mobile phase flows through the sample cell only. As long as no difference exists between the refractive indices of the media of the two cells, there is no refraction of the light passing through them. The light shines on a pair of photodiodes, each of which gives an electrical signal; these signals are amplified and the difference in the two signals is measured. Zero refraction should give a zero-volt difference in these signals. An electrically controlled mechanical linkage allows the user to optimize the photodiodes' outputs for zero deflection via a refractive lens in the optical path. Additional circuitry enables the user to easily correct the signal output to electronic zero.

When a change occurs in the refractive index of the mobile phase, the light passing through the interface between the sample and reference cells is refracted, causing the light intensity on one photodiode to increase and on the other to decrease. This difference gives a signal having both amplitude and polarity; the signal is amplified to drive a chart recorder or digital integrator.

One difficulty with refractive index detection in HPLC is that the refractive index of a liquid is a function of temperature.

Temperature variation of the mobile phase at the point of measurement produces signal drift and noise. The ProStar 355 minimizes this problem by active thermostating of its optics module combined with efficient heat-exchange technology and temperature-monitoring feedback. As a result, the flowing mobile phase is always well-equilibrated at the operating temperature.

The ProStar 355 has external connections on its rear panel for contact closure operation by a data system, instrument controller, autosampler, or other external device. Any device or instrument that can supply a contact closure can purge the ProStar 355 reference cell, Auto Zero its outputs, and generate an event marker signal. Reference cell purging permits the Varian ProStar 355 Refractive Index Detector to run for extended periods of time without exhibiting signal drift due to concentration changes in the reference cell medium. The ProStar 355 is both reliable and sensitive in performance, and straight-forward and flexible in operation.

# Installation

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## Unpacking and Inspection

### CAUTION

**All phases of the installation site preparation must conform to local safety, electrical, and building codes. These codes take precedence over any recommendations in these instructions, and compliance to them is the responsibility of the customer.**

Receiving inspection instructions are detailed in the Pre-installation manual that you will have received before delivery of the detector.

In summary, before accepting delivery, you must inspect the package externally for signs of obvious damage; for example, crushed corners, forklift punctures, tears or cuts and water stains. A further inspection must be made for concealed damage, within the time limit stated in the terms and conditions of carriage. Any shipping damage must be reported to the carrier, and to your Varian Sales Office.

The ProStar 355 detector is packed with a number of accessory parts. Do not discard the packing material until all parts are accounted for. Remove the detector from the box, holding the bottom of the cabinet. Do not lift by knobs or terminals.



**WARNING**

**The Varian ProStar 355 weighs 13 kg (27 lb). Use proper lifting techniques to avoid potential injuries.**

---

## Do's and Don'ts of RI Detectors

1. **NEVER** subject the reference and sample cells to back pressures greater than 7 atm (100 psi). High pressures can break the cells, which are difficult to replace.

**NEVER** place a backpressure regulator (such as Varian part number 0391939300) on an RI detector. Maximum flow cell backpressure is 7 atm (100 psi).

2. When placing more than one HPLC detector in series, **always** place the RI detector **last**.
3. Use tubing of 0.040" ID or greater on the waste outlet of the ProStar 355 for less back pressure.
4. Locate the ProStar 355 in an area that does not incur large temperature changes. The ProStar 355 has a sophisticated temperature control system but care in choosing its operating environment will ensure better operation, see *Installation Site Requirements*, page 48.
5. Always pre-mix and degas the mobile phase. Do not allow pumps to mix solvents for the mobile phase. Use only isocratic conditions.
6. Keep the RI cells clean. See page 93 for the *Cell Cleaning Procedure*.
7. When filling the reference cell, ALWAYS use mobile phase that has eluted through the analytical column. Allow mobile phase to flow through the column for 10 minutes prior to filling the reference cell.

---

## Installation Site Requirements

Select the installation site in accordance with the *Do's and Don'ts* in the previous section. Temperature fluctuation and airflow will affect the performance, causing drift, noise, unstable baseline, etc. The following locations should be **avoided**:

- Near an air conditioner
- Near heating equipment; especially cycling ovens
- Areas exposed to direct sunlight
- Near a ventilation fan
- Near vibration sources
- Near openable windows and doors

- Near sources of electrical noise
- Areas where corrosive gases exist
- Dusty areas

Allow sufficient bench space for peripheral instrumentation and to satisfy the following additional installation site requirements.

1. System pressure fluctuations will be measured as density variations by the ProStar 355 detector. Use a high-performance pumping system with no flow pulses to minimize the baseline noise level. For Varian Star 9000 series pumps and Varian 5000/5500 pumps or ProStar 210/215 pumps, make sure the pump is operating at 100 atm of backpressure and that the pulse damper is functioning properly. The backpressure can be achieved either by the column itself or a coil of 0.005" ID tubing placed between the pump and the column. If pulse damping is needed, use the Varian Pulse Reduction Accessory (part number 0391970101) or the Ultra High Pulse Reduction Accessory (part number 0391970102). For the PrepStar SD1 pumps, no special operating pressure is required.
2. The entire flow system, consisting of pump, damper, injector, column, sample and reference cells, etc., must be primed with the mobile phase. Flush the entire system with 5–10 times the system volume until a steady baseline is obtained.
3. The mobile phase should be pre-mixed and degassed before use. Alternately, the mobile phase can be degassed online by a solvent degassing system.

---

**NOTE:** NEVER try to mix immiscible liquids in your HPLC system. If you wish to replace one solvent with an immiscible one, flush out the existing mobile phase with an intermediary solvent intermiscible with your initial and final solvents. For example, you wish to replace water in your HPLC with chloroform; water and chloroform are immiscible. Replace the water in your system with 2-propanol, which is freely miscible with water and chloroform. When you are certain all water is removed, replace the 2-propanol with chloroform. See the Miscibility Chart on page 110.

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4. If you are using more than one detector in series with the ProStar 355, the RI detector must always be the last component.
5. The cell pressure upper limit is 7 atm (100 psi). Use tubing of 0.040" ID or greater on the waste outlet to protect the cells from elevated pressure which could break them. Should the cells be damaged, the eluate will flow from the DRAIN on the side panel.

The cell assembly should only be replaced by a Varian Customer Support Representative.

## CAUTION

**Do not use a backpressure regulator on the waste outlet side of the ProStar 355.**

6. Leave the system powered continuously for daily operation. Before using the high sensitivity range, it is best to leave the system temperature control on overnight.

---

## Power Requirements

The ProStar 355 detector requires a clean 50 or 60 Hz single-phase power source capable of providing up to 0.8 amps at 100/120 Vac, or 0.4 amps at 200/240 Vac.

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**NOTE:** Running the ProStar 355 detector on a voltage other than the correct single-phase supply will void the warranty.

---

A separate circuit free of equipment with intermittent start and stop cycles and/or heavy starting current demands is recommended for each instrument.

A measured GROUND to NEUTRAL potential of greater than 3 volts AC or DC indicates grounding problems that may need correction before connecting an instrument to the power source. Any power source suspected of having noise problems should be evaluated with a recording power line monitor prior to being used for operating instruments.

---

**NOTE:** Varian instruments are designed for use on single-phase (phase-neutral) power ONLY. If your facility provides only phase-phase (i.e., three-phase) power, consult the nearest Varian facility for information about isolation transformers BEFORE applying power to your instruments. For phone numbers of Sales and Service Assistance, refer to the Safety section.

---

The power cord for use in the US and other 120 Vac, 60 Hz applications is terminated in a 3-prong parallel blade plug, requiring a matching receptacle wired as shown in Figure 2. Outside of the 120V, 60 Hz area, the instrument is supplied with a power cord terminated in a "Schuko" CEE 7/7 2-prong plug. Replacement of the instrument power plug



requires strict compliance with power cord color coding as listed in Figure 2.

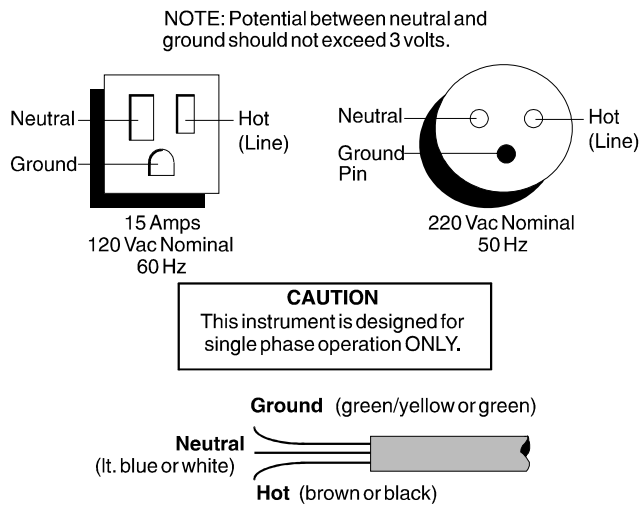


Figure 2 Power Cord Wiring

### Power Button, Fuse & Grounding

The ProStar 355 Power button is located on the bottom left of the front panel. The power should OFF before connecting the power cord to the detector. The receptacle for the power cable is located on the rear of the detector.

A 3.15 A fuse is provided. The fuse will be installed as described on page 52, in *Installing the fuse and power cable*.

Ground the detector using the grounding cable supplied in the Accessory Kit. Connect the circular end to one of the grounds on the back panel (see Figure 5) and the alligator clip end to a known ground.



**WARNING:  
SHOCK HAZARD**

Electrically conducting spills can occur when conductive HPLC solvents are spilled on or in the instrument. Ground the instrument properly to protect the operator from electrical shock. Proper grounding also protects the instrument from power line noise.

Verify that the instrument is properly grounded through the power line ground terminal. Do not remove or otherwise disable the power cord's ground prong.

---

## Preparing the ProStar 355 for Operation

The ProStar 355 as delivered is not ready for operation. The following three steps must first be performed.

- Install the fuse and power cable
- Loosen the shipping bolts
- Install the drain tube

### *Installing the fuse and power cable*

The instrument is shipped without a power cable or fuse installed. Install as follows:

1. Check that the power is OFF.



**WARNING:  
FIRE HAZARD**

Replace only with same type and rating of fuse.

2. Confirm that the voltage of the power socket into which the ProStar 355 will be plugged is the same as the voltage indicated on the rear panel of the detector.
3. Install the supplied fuse.
4. Ensure that the power is OFF and connect the appropriate power cord.



**WARNING:  
SHOCK HAZARD**

Prior to connection, ensure that the voltage of the power socket into which the detector power cable is being plugged is the same as the power supply voltage indicated on the detector.

The power socket should be of a three-pin type with a grounding terminal. Do not use other types of power socket.

Use only the power cable provided.



**WARNING:  
FIRE HAZARD**

Do not use the detector in places where combustible gas or any source of fire or sparks might exist.

### ***Loosening the Shipping Bolts***

The Varian ProStar 355 is shipped with the optical module securely tightened down by two Allen bolts. These bolts must be loosened so the optical system can rest on rubber shocks that will isolate the optics from vibration.

1. Refer to Figure 3. Gently place the right side of the detector on the end of a bench, while supporting it to prevent it from falling.

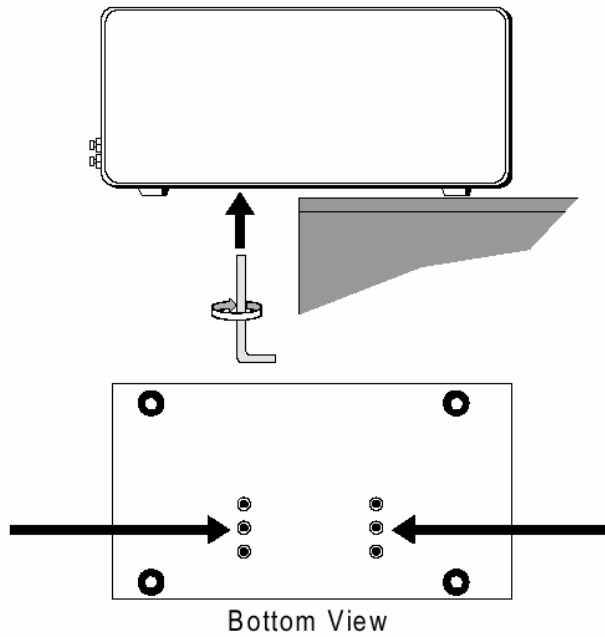


Figure 3 Loosening the Shipping Bolts

2. **Locate** the 6 bolts in the ProStar 355 floor panel. **Loosen the two center bolts** one full turn counterclockwise using the 5 mm Allen key in the Accessory Kit.

---

NOTE: Do not loosen all 6 bolts. Do not remove the bolts. Do not run them out so far that the detector's weight rests on the bolt heads instead of its feet.

---

3. Move the detector securely back onto the bench.

---

NOTE: Detector will not stabilize if shipping bolts are not loosened.

---

### **Optics Module Drain Tube**

The drain tube attachment for the optic module allows liquid to drain from the module if a sample or reference cell should break. The tube exits the side of the instrument. Connect PTFE tubing (6 mm OD, 4 mm ID) to this port. Lead the tubing to a safe waste disposal container.



Electrically conducting spills can occur when conductive HPLC solvents are spilled on or in the instrument. Ground the instrument properly to protect the operator from electrical shock. Proper grounding also protects the instrument from power line noise.

Verify that the instrument is properly grounded through the power line ground terminal. Do not remove or otherwise disable the power cord ground prong.

## Tubing Connections to the ProStar 355

To maximize performance of the ProStar 355 and minimize backpressure to the refractive index cells, review *Do's and Don'ts of RI Detectors*.

### Connection to the Inlet

**NOTE:** The ProStar 355 must be the last component in any LC system. No detector or backpressure regulator can follow it. Do not use narrow-bore tubing after the ProStar 355. Refer to Figure 4.

1. Install the narrow-bore stainless steel tubing from the Accessory Kit (0.25 mm ID, 1 m long) and Parker-Hannifin nut and ferrule into the "IN" fitting of the ProStar 355. Swage the ferrule securely onto the tubing with the nut.
2. Connect the opposite end of the tubing in the same manner to the column exit end. See Figure 4.

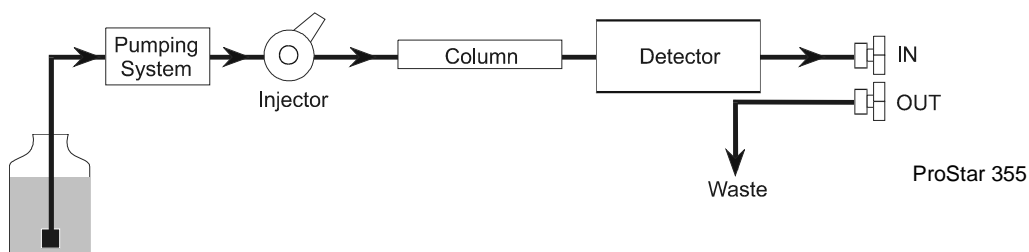


Figure 4 ProStar 355 connected to the HPLC System

### **Connection on the Outlet Side**

1. Install the 5 cm long ID stainless tubing from the Accessory Kit in the "OUT" port of the ProStar 355 and swage the ferrule securely onto the tubing with the nut.
2. Slip the 1.5 mm ID PTFE tubing over the outside of the outlet tube. Insert the opposite end of tubing into a waste bottle.

#### **CAUTION**

Use only the tubing provided for connection to the outlet.

---

## **Cable Connections to the ProStar 355**

### **Recorder Connection**

1. Using the output signal cable supplied in the Accessory Kit, connect one end to the chart recorder, black lead to recorder negative (-) terminal and red lead to recorder positive (+). Connect the opposite end of the positive (+) wire to the ProStar 355 REC OUT + terminal. Connect the negative (-) terminal to ProStar 355 REC OUT - terminal.
2. Connect the clear lead (shield) to the FG (cable shield ground) terminal.

---

NOTE: The ground posts below the COM port on the rear panel (see Figure 5) are not the same as the cable shield ground (FG) terminal. Do not interchange these connections.

---

### **Integrator Connection**

An output signal cable is supplied in the Accessory Kit for connecting the ProStar 355 to an integrator. Connect the red lead (+) to the INTEG OUT + terminal on the rear of the ProStar 355. Connect the white lead (-) to the INTEG OUT - terminal. Connect the clear lead (shield) to the FG (cable shield ground) terminal. Connect the opposite end of the signal cable to the correct integrator terminals.

INTEG OUT sensitivity: 2 mV/ $\mu$ RIU or 8 mV/ $\mu$ RIU  
(integrator range 500  $\mu$ RIU/V or 125  $\mu$ RIU/V)

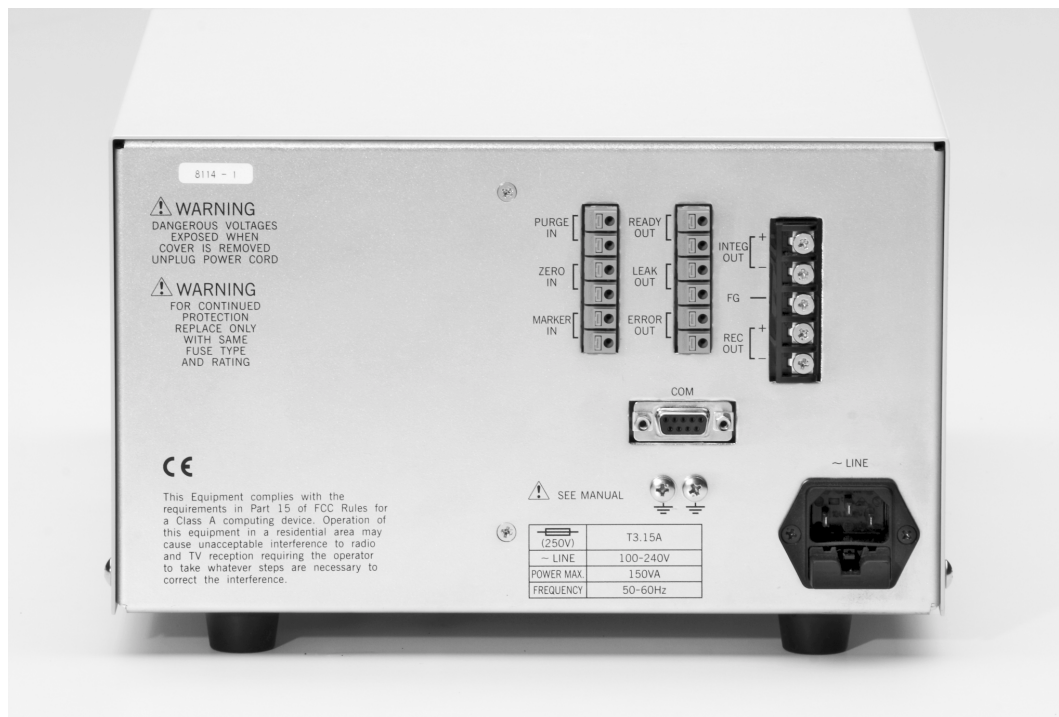


Figure 5 Rear Panel Controls

Purge terminals (PURGE IN)	When these two terminals are short-circuited, the flow line of the eluent is changed, and both sample and reference cells are filled with the same eluent, as occurs when the Purge key is pressed.
Zero in terminals (ZERO IN)	When these two terminals are short-circuited, an Auto Zero is activated, as occurs when the Zero key is pressed.
Marker in terminals (MARKER IN)	When these two terminals are short-circuited, a momentary voltage “spike” is put on top of the recorder output, as occurs when the Marker key is pressed.
Ready out terminals (READY OUT)	A contact pulse is sent out through these terminals when the sequence is completed.
Leak out terminals (LEAK OUT)	A contact pulse is sent out through these terminals if there is an eluent leak.
Error out terminals (ERROR OUT)	A contact pulse is sent out through these terminals when an error occurs, such as overheating, null glass home position, optical balance error, lost parameters and low light intensity.
Integrator terminals (INTEG OUT)	Signals to the data processing unit are sent out through these terminals. The sensitivity of the output signals is 2 mV/μRIU or

	8 mV/ $\mu$ RIU (integrator range: 500 $\mu$ RIU/V or 125 $\mu$ RIU/V).
Ground terminal for cable shield (FG)	The shield of the provided signal cable should be connected to this terminal.
Recorder terminals (REC OUT)	Signals to the recorder are sent out through these terminals. The sensitivity of the output signals is 10 mV/FS.
Communication port (COM)	RS232C Communication port
Ground terminals	Terminals to ground the main body of the detector.
Power connector	The power cable provided should be plugged into this connector.

### ***Connection to a Varian Star 800 Module Interface Box***

The Star 800 Module Interface Box (MIB) provides analog-to-digital signal conversion (ADC). Connections are made by connecting a round miniDIN connector to one of the analog signal input ports on the middle right side of the Star 800 MIB. For more information about connecting your ProStar 355 to a Star 800 MIB, refer to your Star 800 MIB documentation.



### Connection to a Varian Star Chromatography Workstation

Figure 6 illustrates the ProStar 355 connection to the Varian Star Chromatography Workstation.

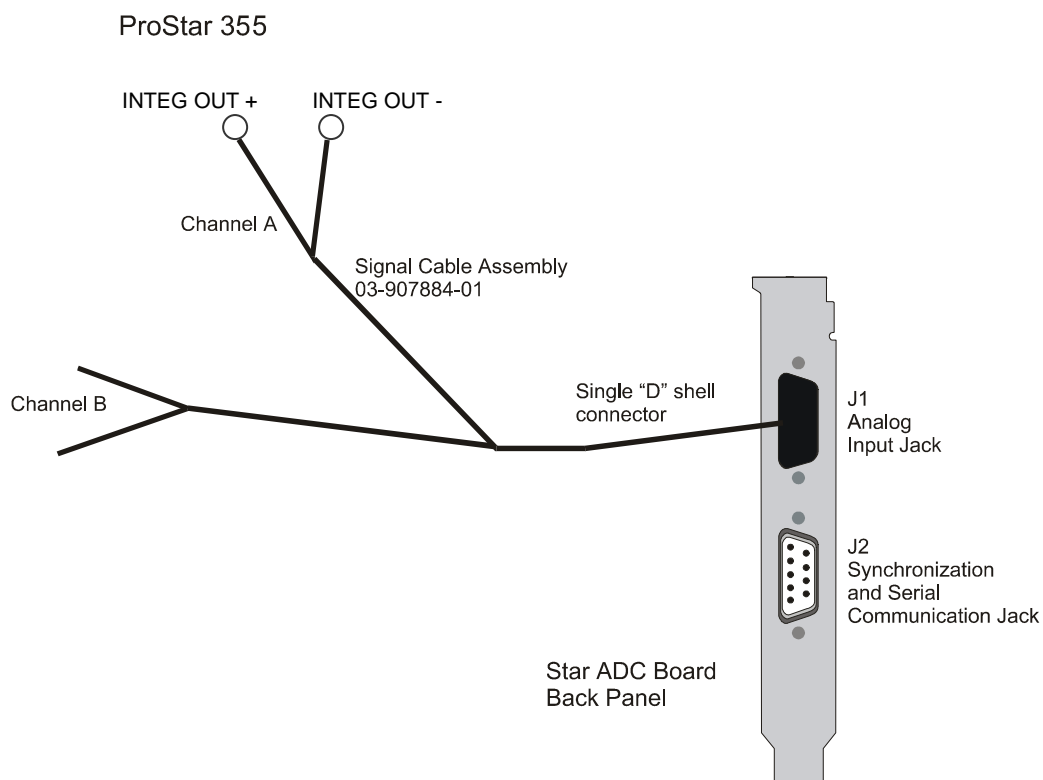


Figure 6 ProStar 355 to Varian Star Chromatography Workstation

### Connection to a Varian 4400 Integrator

Figure 7 illustrates connection to the Varian 4400 Integrator. Its input voltage is 0–1 V. To monitor via recorder and integrator simultaneously, you will need to order Varian part number 0200088200, Recorder Cable (spade lug-spade lug). Attach black leads to negative (–) terminals and clear/white leads to positive (+) terminals.

## ProStar 355

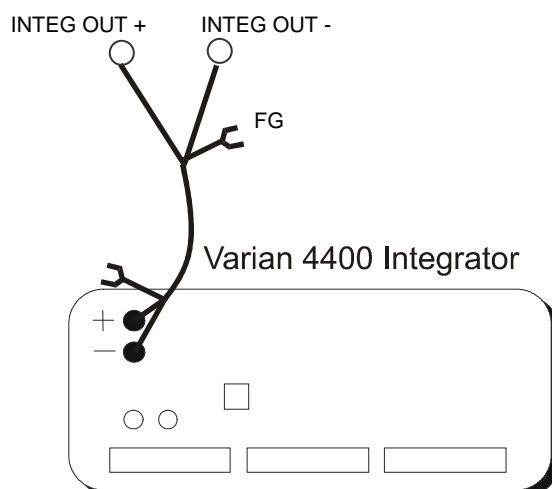


Figure 7 ProStar 355 to Varian 4400 Integrator

### **External Control of the ProStar 355**

The ProStar 355 has the capability of having the following functions controlled by external devices: Auto Zero, purge, and event marker. Two items are necessary for external control of the ProStar 355:

1. A separate instrument or device that is capable of generating a contact closure via a powered relay. The following instruments from the Varian product line can provide this contact closure:
  - Varian SD 200/300 Pumps and ProStar 210/215
  - Varian SD-1 Pump
  - Varian 9050, ProStar 310 UV Detector
  - Varian 9002/9012 HPLC Pumps/ProStar 220/230/240
  - Varian 4400 Integrator with external events option and BASIC option
  - Varian Star 800 MIB relay control

2. A cable for the contact closure signal such as:
  - Varian part number 0200088200 (spade lugs both ends)
  - Varian part number 0200195400 (3-pin Molex receptacle-spade lug)
  - Varian part number 0391972101 (ProStar 355 to 9002/9012/9050 sync cable) External Input/Output Connections for the ProStar 355

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**NOTE:** Relays that output a “TTL active low pulse” will appear to the ProStar 355 as a contact closure because the ProStar 355’s events are triggered when the voltage of the positive (+) terminal of any input is zeroed (“pulled low”). If a relay does not control events in the ProStar 355, try switching cable leads at one end.

---

### **CAUTION**

**Voltages greater than TTL (0 to 5 V) are not recommended as input to the ProStar 355. The closure to the ProStar 355 must be momentary, i.e., more than 0.5 s but less than approximately 10 s. A constant short across some terminals may temporarily disable some ProStar 355 features, see *External Control of the ProStar 35*, page 60.**



# Operation

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## How to Use This Section

This section provides guidelines for the operation of the ProStar 355. The following topics are covered:

- Solvent limitations, recommendations and preparation
- Sample preparation
- ProStar 355 controls
- Pre-operation check list
- Instrument operation
- Initial test procedures
- Examples of use
- Shutdown

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**NOTE:** To document the performance of your new ProStar 355 detector for future reference, perform the following tests and archive the results:

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1. Noise and Drift Test, see page 81.
2. Chromatogram of RI Test Sample (see page 88 and Figure 15).

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## Solvent Recommendations

### *Solvent Limitations*

Some solvents may corrode the wetted surfaces of the ProStar 355 detector, if they are left in the detector after operation. The quartz cell window is easily etched by strong bases. It is recommended that some solvents be rinsed from the ProStar 355 detector for overnight and

weekend storage. Refer to Table 1, *Solvent Limitation Recommendations*.

The solvents used in the ProStar 355 detector are limited by the materials used for the hydraulic parts they come in contact with (e.g., quartz glass, PTFE, and 316 stainless steel). These limitations should be considered in selecting mobile phase solvents.

NOTE: Table 1 is intended to serve as a guide and does not cover all possible conditions. Since actual effects depend upon a number of conditions, tests should be made using pieces of 316 stainless steel (column, tube, ferrule, etc.) under these conditions: 1) when the solvent to be used is close to the boundary indicated in Table 1, 2) when a mixture of solvents is to be used; and 3) when the solvent contains some active solutes other than those listed.

*Table 1 Solvent Limitation Recommendations*

<i>Unusable</i>	<i>Usable Below 10%</i>	<i>Usable Below 30%</i>	<i>Usable Below 50%</i>
Hydrohalogenic Acids: HCl, HF, etc. Metal Halides >2M KCl	Sulfuric Acid Boric Acid Acetic Anhydride KOH	Citric Acid NaOH Ammonium salts: -citrate -oxalate -sulfate -H <sub>2</sub> PO <sub>4</sub>	Acetic Acid Lactic Acid Hydrazine Ammonium nitrate K <sub>2</sub> CO <sub>3</sub> Sodium formate
Ammonium Halides Ammonium Formate All hypochlorites Tetrachloromethane	Ammonium salts: -formate -perchlorate -HPO <sub>4</sub> K, Na salts of: -bicarbonate -chlorate -nitrite		

If a limited condition solvent listed above is in use, flush out all hydraulic lines sufficiently with an inert solvent that is compatible with your chromatographic system and column. Buffers, acids, and other highly ionic aqueous solutions should be flushed out with large amounts of water (5–10 times the volume of liquid from pump head to detector outlet). Under some circumstances you may want to follow this deionized water flush with a flush of 2-propanol, if it is compatible with the column packing. If you neglect this flushing, the pump, injector and column may become corroded and badly damaged.

When an organic solvent that contains halogens, such as chloroform and methylene chloride, has been used, flush out all lines with a solvent compatible with your chromatographic conditions and column. For example, hexane or another hydrocarbon. For isocratic work with a

normal phase column, the alcohols methanol or 2-propanol, a non-carcinogenic aromatic such as the xylenes, acetone, or an ether that is non-volatile and not a ready producer of peroxides can be used. DO NOT use ethyl ether.

---

NOTE: Ethanol stabilized chloroform does not form chloride ions readily; therefore, it is recommended in place of pure chloroform.

---

Fluorocarbon solvents will alter PTFE over long exposure. Flush with pentane or another light hydrocarbon.

Limitations in addition to those listed in Table 1 may exist for a specific column. Always refer to the column instruction manual for recommendations on solvent limitations.

If the detector is to be exposed to sub-freezing temperatures, an antifreeze flush, such as methanol must be used.

## ***General Recommendations for Solvent Preparation***

### ***Grade of Solvents***

HPLC grade solvents are recommended for better instrument performance and chromatographic data.

Solvents that are easily oxidized, e.g., Tetrahydrofuran (THF), require the addition of an anti-oxidant. Anti-oxidants, however, will not affect refractive index data.

THF when used as an isocratic mobile phase in GPC (Gel Permeation Chromatography) has a tendency to cause baseline drift due to oxidation during analysis and, therefore, requires the addition of an anti-oxidant. However, the addition of an anti-oxidant may cause the eluent to be unusable with a UV detector due to absorption by the anti-oxidant.

### ***Solvent Filtering***

All solvents, including deionized water, should be filtered with a 0.45  $\mu\text{m}$  or 0.2  $\mu\text{m}$  filter. Filtering protects the pump, injector and column from potential damage and early failures due to large particles adhering to surfaces. Vacuum filtration functions to filter and degas solvents.

## **Solvent Degassing**

All solvents must be properly degassed prior to use on the ProStar 355 detector. Solvents that are not properly degassed may cause bubble formation in pump heads and in the detector cells potentially causing pressure flow problems and a noisy chromatographic baseline. Degassing is especially important when the column temperature is elevated above room temperature, or with solvents that have a high gas solubility, e.g., methanol. Analysis times that run greater than 5 to 6 hours will also require degassing of solvents.

Solvents can be degassed by vacuum degassing, filtration, stirring, ultrasonic agitation, sparging with helium, or an in-line degasser may be used. Vacuum filtration of solvents through a 0.2  $\mu\text{m}$  or 0.45  $\mu\text{m}$  membrane filter is a convenient method to both degas and filter solvents. Varian Aqueous and Organic Solvent Clarification Kit (part number 0099750700) is available for this purpose.

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## **Sample Preparation**

Generally, it is desirable to dissolve samples in the mobile phase solvent. When a different solvent is used, a large solvent peak may appear on the chromatogram.

All particulates larger than 0.5  $\mu$  must be removed. Sample filtration kits are available for this purpose and can be found in the Varian, Inc. Consumables and Supplies catalog. If filtration is inconvenient, a pre-column filter or guard column should be installed.



## ProStar 355 Controls

### Block Diagram and Basic Circuitry of the ProStar 355

Figure 8 is a block diagram of the ProStar 355 Refractive Index Detector. Figure 9 shows the basic circuitry of the ProStar 355 input and output signals.

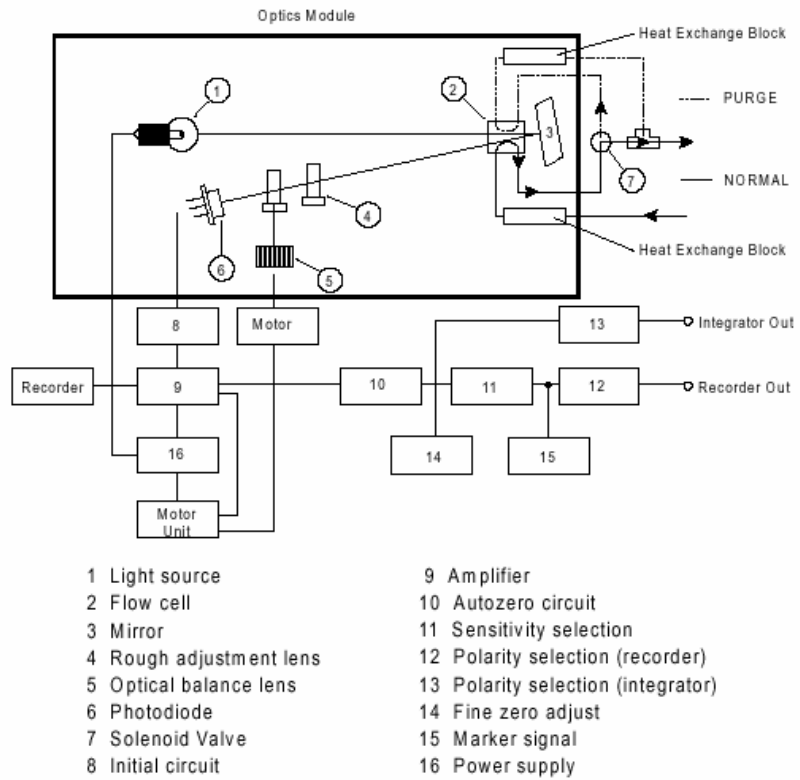


Figure 8: ProStar 355 Refractive Index Detector Block Diagram

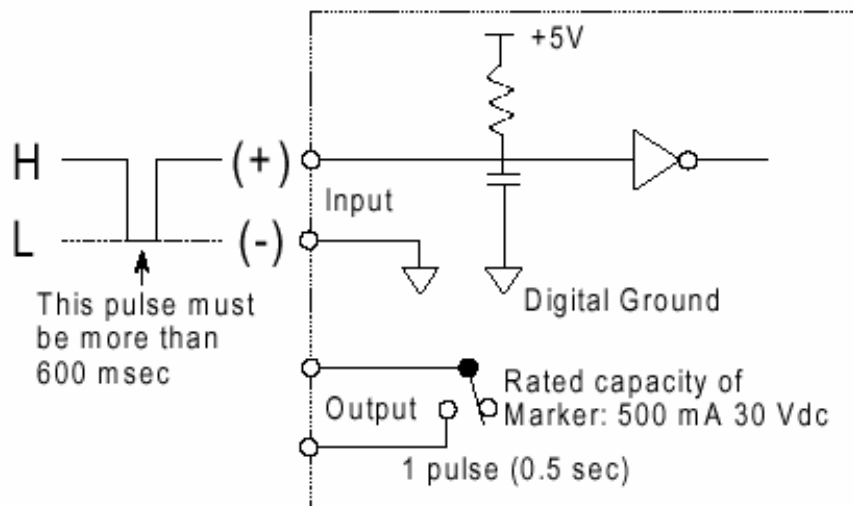


Figure 9: Input/Output Terminal Circuit Diagram

## Front panel controls

Figure 10 shows the front panel controls on the ProStar 355.



Figure 10: Front Panel Controls

Power key (Power)	Turns the unit ON or OFF.
LCD	Liquid Crystal Display
Leak LED (Leak)	This light is illuminated when a solvent leak is detected.
Start-Up Sequence key (S. Seq)	Initiates a Start-Up Sequence.
Purge key (Purge)	Turns the purge valve on or off in order to change the flow path. When the valve is on, the PURGE sign is highlighted with a yellow background, and solvent flows through the reference side of the flow cell instead of the sample side. Press the Purge key again to turn off the valve. The Purge key is not functional during the Start-Up Sequence.
Auto Zero key (Zero)	Activates an Auto Zero. The Zero key does not function during a sequence.
Marker key (Marker)	Generates an event marker signal, i.e., a momentary voltage “spike” is put on top of the recorder output (10% of Full Scale).

Arrow keys	These keys move the cursor in the LCD and enable you to edit values. By default, the cursor will scroll in a circular motion.
Escape key (Escape)	Returns the LCD display to the Monitor Screen.
Cancel key (Cancel)	Cancels an edited value in the LCD (the cursor will return to the tab), or cancels a command.
Enter key (Enter)	Saves an edited value in the LCD (the cursor will return to the tab), or confirms a command.

### **Using the front panel controls**

Use the right and left arrow keys to view four main screens in the LCD display. The four screens are: Monitor, Parameters Setting, Validation Display and Span Check.

### **Monitor Screen**

The Monitor Screen displays refractive index, temperature and status on the upper line, and errors, alarms and responses on the lower line. The following table outlines the meaning of various codes displayed in the Monitor Screen:

<b>Code</b>	<b>Meaning</b>
LK	The keys are locked
ER	An error has occurred.
RD	Ready (Start-Up Sequence)
ROM	ROM error
RAM	RAM error
PARAMETER	Parameter memory error
HOME POSITION	Null glass motion error
LEAKAGE	Solvent leak within flow path
OPT. BALANCE	Exceeded null glass motion range. If this error is displayed, replace the solvent in the flow path. If this does not fix the problem, contact your local Varian representative.
TEMP. UNSTABILIZE/UNTEMP	The temperature hasn't stabilized to a constant temperature (set temperature $\pm 1$ °C) over a period of 10 minutes
INTENSITY	The voltage of the source lamp has exceeded the limit.
PURGE	The purge is on.
AUTO ZERO/ZERO	An Auto Zero is running.
S.SEQ	A Start-Up Sequence is running.
F	Start-Up Sequence mode is Fine

S	Start-Up Sequence mode is Standard
C	Start-Up Sequence mode is Coarse
INTENS	Intensity
xxx min	Maximum time remaining for the Start-Up Sequence.

## Parameter Settings Screen

To set the operation parameters:

1. From the Monitor Screen, press the right arrow key to display the Parameters Screen.
2. Use the Up and Down arrow keys to scroll through the parameters. To change a parameter, scroll to the appropriate parameter, press the Enter key, then use the Up and Down arrows to find the desired new setting.
3. Once the appropriate new setting is displayed, press the Enter key to save the setting. You can press the Cancel key to cancel a new setting, or the Escape key to return to the Monitor Screen.

The following table outlines possible operation parameters:

Parameter	Selectable values	Unit	Default
REC. RANGE	0.25, 0.5, 1, 2, 4, 8, 16, 32, 64, 128, 256, 512 (12 steps)	$\mu$ RIU/10 mV	512
INTEG. RANGE	125, 500	$\mu$ RIU/1 V	500
TEMPERATURE	0, 30–50 (1 step) 0: Temp. control OFF	$^{\circ}$ C	35
TIME CONSTANT	0.1, 0.25, 0.5, 1, 1.5, 2, 3, 6 (8 steps)	s	3
POLARITY	+, -	N/A	+
BASELINE SHIFT	0–50 (1 step)	10 mV	0
S. SEQ MODE	Fine, Standard, Coarse	Refer to the next table	Standard
LEAK SENSOR	ON, OFF	N/A	ON
KEY LOCK	YES, NO	N/A	NO
DEFAULT DATA	YES, NO	N/A	NO

---

## Pre-Operation Check List

Before operation, check the following detector and system connections and switches.

- Shipping bolts loosened per Loosening the Shipping Bolts on page 53.
- Desired analytical column installed.
- Mobile phase flushed through all of the HPLC system; all incompatible or immiscible solvents have been flushed out, see page 48.
- Connections made to chart recorder, integrator, data system, or other external equipment, see page 59 and page 60.
- All tubing connections are made and checked for leaks, see page 55.
- Power socket voltage matches voltage indicated on rear panel, see page 52.
- Drain tube is installed, see page 54.
- Detector, pump, and peripherals are plugged into appropriate power receptacles.
- Power switches on the pump, peripherals, and other detectors are ON

---

## Instrument Operation

### *Starting up using Start-Up Sequence*

To perform an unattended start-up:

1. Set the operating parameters using the Parameter Settings Screen.
2. Start pumping mobile phase solvent at a flow rate of 1 mL/min.
3. Press the S. Seq key to initiate the Start-Up Sequence.

The following table shows fixed values for the Start-Up Sequence:

Parameter	Fine	Standard (Default)	Coarse
Purge Cycle	30 s		
Number of Cycles	3		
Time to Auto Zero	240 s		
Equilibration Time	80 min	60 min	40 min
Measuring Time	80 min	60 min	40 min
Drift	100 nRIU/h	500 nRIU/h	2500 nRIU/h
Noise	50 nRIU		

4. If drift or noise did not meet target values within a defined period, FAIL will be displayed on the Start-Up Sequence status line. Press the S. Seq key to re-do the sequence. You can press Enter or Cancel to abort the sequence.
5. When drift and noise meet the target values within the time limit, the sequence is finished and READY will be displayed on the Start-Up Sequence status line.

The ProStar 355 is now ready for injection.

### **Starting up manually**

#### **Recorder Output**

1. The ProStar 355 and all peripheral instruments should be plugged in.
2. Press the ProStar 355 Power button and set the temperature to the desired temperature using the Parameters Setting Screen. The detector will be operational in one to three hours for most applications. However, at the highest sensitivities up to 24 hours may be needed for stabilization.

---

**NOTES:** If the LCD displays TEMP. UNSTABILIZE, the ProStar 355 cell is not at temperature. This message will disappear temperature stabilization is complete.

Before activating the purge valve (LED light OFF), pump about 10 mL of liquid through the cell. This will flush possible dust or particulate matter and reduce the possibility of damaging the valve seals.

---

3. Press the Purge key and pump mobile phase through the sample and reference cells at 1 mL/min, for approximately 20 minutes.
4. The digital output display may or may not be at zero (000 mV) at this time. Press the Zero key. Watch the display. When the output appears constant, proceed to the next step. If the mV output drifts, the chromatographic system and/or the temperature control have not stabilized. Repeat this step until the output remains close to zero and stabilizes.
5. Go to the Parameters Setting screen and select the REC. RANGE parameter. Use the UP or DOWN arrows until the desired sensitivity is reached. Turn the recorder on and start the paper.
6. Press Zero. When the recorder trace goes off scale, re-zero with the Zero key.
7. Monitor the ProStar 355 signal drift (i.e., the rate of change of the signal in RIU/hr) to see if the instrument and chromatographic system are sufficiently stabilized for the selected sensitivity.

---

NOTE: At a range setting of  $1 \times 10^{-6}$  RIU/FS with water flowing at 1 mL/min, drift is typically 3% or less of full scale within one hour. Higher sensitivities may require more time to achieve this drift.

---

8. Once the desired level of stability is reached, press the Purge key to isolate the reference cell from the flow path. A single deflection of the pen is expected. Because the chromatographic and temperature systems have stabilized, baseline restabilization usually occurs rapidly.

---

NOTE: Tetrahydrofuran (THF), if not stabilized with antioxidant, is an exception. The THF in the reference cell will oxidize until a plateau is reached before restabilization occurs.

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9. At time of sample injection press Marker to mark the moment of injection.



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### **Integrator Output**

1. Perform steps 1 through 4 of Recorder Output on page 72.
2. Set the integrator or data system to the desired attenuation. Press Zero on the ProStar 355 to begin monitoring the signal.
3. Monitor the ProStar 355 signal drift (i.e., the rate of change of the signal in RIU/hr) until the instrument and chromatographic system are sufficiently stabilized to use at your desired attenuation.

---

NOTE: Many integrators and data systems have mechanisms to correct for signal offsets at time zero. For this reason it is advisable to Auto Zero the ProStar 355 prior to monitoring the signal on the integrator or data system. Otherwise signal zero may be off scale on the integrator.

---

4. Once the desired level of stability is reached, press the Purge key to isolate the reference cell from the flow path. A signal deflection of the pen, due to a pressure differential between reference and sample cells, is to be expected. Press Zero. Because the chromatographic and temperature systems have stabilized once already, restabilization usually occurs rapidly.

---

NOTE: Tetrahydrofuran (THF), if not stabilized with an antioxidant, is an exception. THF in the reference cell will oxidize until a plateau is reached before restabilization occurs.

---

The ProStar 355 is now ready for injection.

### **Routine Operation**

The ProStar 355 Refractive Index Detector should indicate to the user that it is ready for operation. First, the digital signal (recorder or integrator) output display should be near zero. Second, all lighted indicators should be green. A yellow LED indicates "Caution" — the ProStar 355 is not ready for injection.

During analysis, the digital signal display may move up scale (or sometimes down scale) indicating the passage of compounds through the sample cell. This is a normal operating characteristic. During a long run at high sensitivity, the baseline may drift slightly. This may be due to a temperature change, or change in the composition of one or both liquid cells (oxidation, dissolved gas, solvent impurities, column

“bleeding,” etc.). If such drift is tolerably slow, continuous compensation can be made by repeating the zero adjustment procedures, but only during periods when no sample peaks are passing through the detector.

### ***Familiarization Exercise***

The following procedure demonstrates the operating characteristics of the ProStar 355 controls, and shows the effects of the controls on the recorder and integrator output signals.

1. The ProStar 355 should be filled with water, thermostatted, equilibrated, and zeroed. Refer to steps 1–7 in Recorder Output on page 72.
2. Set the REC. RANGE to  $1 \times 10^{-6}$  RIU if a chart recorder is in use.
3. If an integrator/data system is in use, find an attenuation setting that gives about the same signal amplitude for your recorder and integrator. For Varian data systems, start with attenuation 64.
4. Press the Zero key to re-zero the electronics and optics of the ProStar 355. You will observe that both recorder and integrator signals are affected by these controls. Auto Zero is slightly displaced from your recorder zero; however, this displacement is fairly constant from re-zeroing to re-zeroing.
5. Press Purge to place both the reference and sample cells in line with the mobile phase. The resulting change may or may not be significant enough to observe as an output signal. All analyses should be performed with the purge OFF.
6. Press the Marker key. You will notice that only the recorder output is affected. A momentary voltage “spike” is inserted on top of the recorder output.

---

## Calibrating and Testing the ProStar 355

### *Validation Display Screen*

The Validation Display Screen allows you to verify that your ProStar 335 hardware is functioning properly. You can view:

- Temperature: If the temperature is at the specified temperature  $\pm 1$  °C, GOOD will be displayed. If the temperature is outside this range, NG will be displayed.
- Source lamp voltage: If the source lamp voltage is under 4.5 V, GOOD will be displayed. Otherwise, NG will be displayed.
- Drift and Noise: If drift and noise values are under the fixed values in the Start-up Sequence, GOOD will be displayed. Otherwise, NG will be displayed.
- Span: If the span is  $512 \pm 25$   $\mu$ RIU, GOOD will be displayed. If the span is outside this range, NG will be displayed.

### *Recorder and Integrator Span Test and Adjustment*

Span adjustment for chart recorder and integrator may shift after any of the following procedures have been performed:

1. Source lamp replacement
2. Photodiode replacement
3. Cell replacement

Use the following procedure for recorder and integrator span testing and adjustment.

These are the items you need to perform this test:

- ProStar 355 with signal cable from REC OUT to a recorder set at appropriate voltage (1 mV or 10 mV)
- 5 mL glass syringe with Luer<sup>®</sup> tip (from Accessory Kit)
- #16 Luer needle with nut and ferrule (from Accessory Kit)
- Small flat-bladed screwdriver (from Accessory Kit)
- Small Phillips screwdriver (from Accessory Kit)
- Approximately 600 mL H<sub>2</sub>O; deionized, degassed
- Sucrose
- Analytical balance accurate to 1 mg
- 500 mL volumetric flask
- An accurate volt-ohmmeter for integrator span adjustment

1. Remove the cover of the ProStar 355 with the Phillips screwdriver. Locate the VR2201 Blue trimming potentiometer with the Red mark on the Amplifier PC Board (located in Daughter PC Board area); see figure below. Turn ProStar 355 power on. Set the temperature to 35 °C.

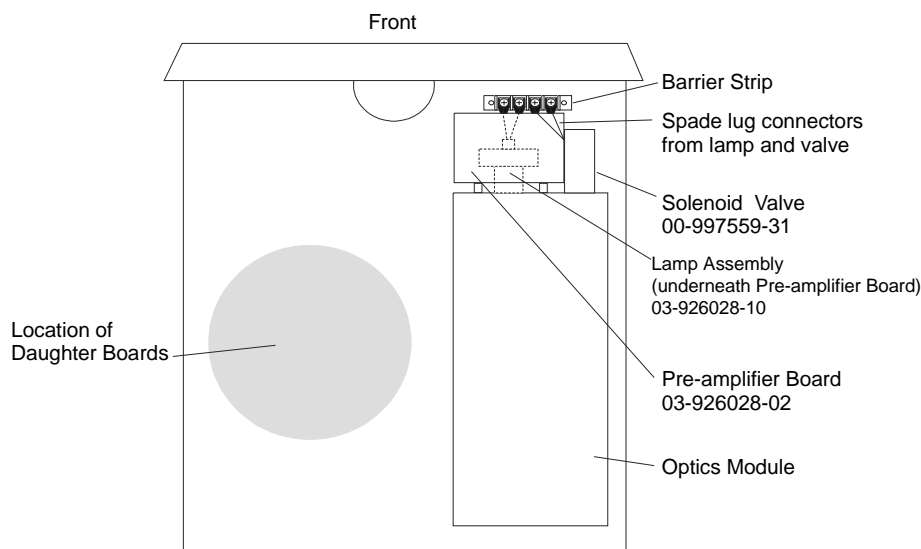


Figure 11 Interior Components of the ProStar 355

2. Preparation of standard sucrose solution: Weigh out 853 mg sucrose and transfer quantitatively to a 500 mL volumetric flask. Dissolve in the deionized, filtered, degassed water and dilute to flask mark. The refractive index of this standard solution is  $256 \times 10^{-6}$  RIU, referenced to deionized water at room temperature.

---

**NOTE:** Use the sucrose solution within one week. Keep refrigerated.

---

3. Prepare the syringe and needle by placing the nut and ferrule onto the end of the needle, inserting the needle into the IN port of the ProStar 355 until it bottoms out. Hold the needle with one hand and swage the ferrule onto the needle with the 5/16" wrench. Remove the needle and assemble it to the syringe.
4. Equilibrate the ProStar 355 by pumping deionized water through both the reference and sample cells or, with the

syringe inject deionized water into the two cells. Use the same deionized water as that used to prepare the sucrose standard solution.

*To equilibrate with a pump:*

- Set pump flow rate to 1 mL/min.
- Let flow for 5 minutes.
- Press Purge; flow path will include both sample and reference cells.
- Let flow for 5 minutes.
- Press Purge; flow path excludes reference cell.

*To equilibrate with a syringe:*

- Fill the 5 mL syringe with deionized water.
- Connect the syringe needle to the IN port of the ProStar 355.
- Press Purge; flow path will include both sample and reference cells.
- Inject 4–5 mL of water into the IN port.

---

NOTE: It is normal to feel considerable backpressure on the syringe. A 1–3 mL syringe (e.g. Varian part number 0099672118, 2.5 mL) can be used in place of the 5 mL syringe for less pressure.

---

5. Select a range of  $512 \times 10^{-6}$  RIU.
6. After the detector signal output stabilizes, Auto Zero to adjust optics and signal and signal zero to recorder zero. For further details, see paragraph 3.6.1. Monitor signal to verify that everything is zeroed and stable.
7. With Purge off, using the 5 mL syringe, inject 2–3 mL of the standard sucrose solution prepared in step 1 into the IN port and allow to equilibrate several minutes. This fills the sample cell with the standard solution.  $256 \times 10^{-6}$  RIU is now obtained, referenced to the deionized water in the reference cell.
8. The recorder pen should have deflected by  $50\% \pm 10\%$  of full scale. If it does not (and you are certain the recorder is correctly set and calibrated), adjust the span by turning the VR2201 potentiometer on the Amplifier Board with a small

screwdriver until the recorder registers 50% of full scale. Refer to Figure 3-6 for an example of span test recorder output.

- Integrator span adjustment tracks with the recorder span. Therefore, to set integrator span, repeat steps 4 through 7 (ignore step 5 as range setting is not relevant to an integrator). With a volt-ohmmeter, measure the voltage at the integrator output positive (H) and negative (O) terminals. The volt-ohmmeter should show a response to the sucrose solution of 820 mV ( $\pm 50$  mV).

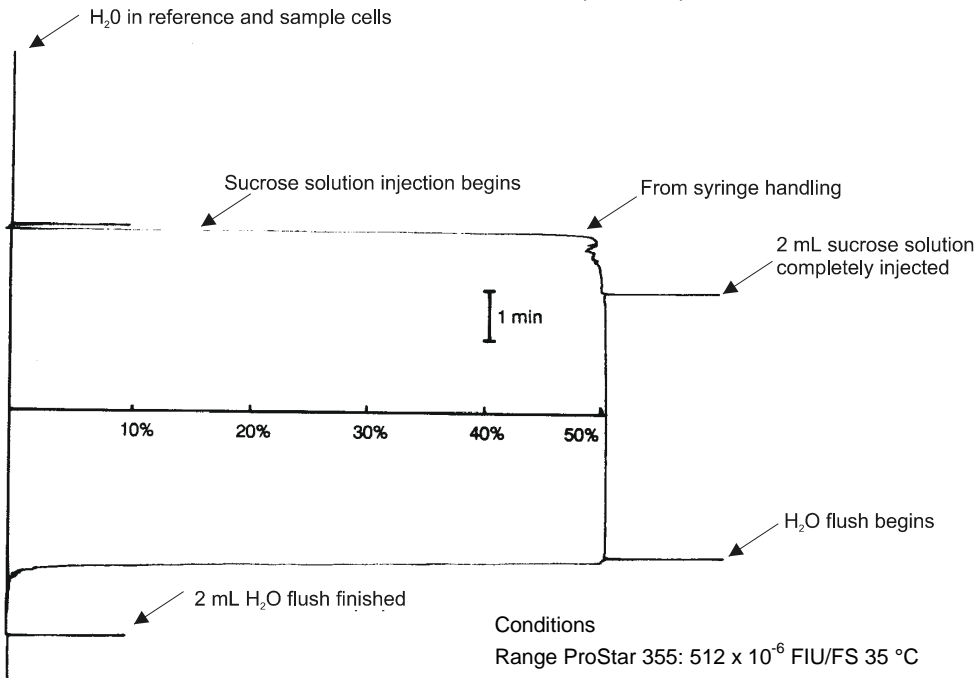


Figure 12 Recorder Span Adjustment

- Carefully flush the sample cell with water. The pen should return to its original, zero-point setting.
- Repeat steps 5 through 8 to confirm reproducibility.
- Reassemble the ProStar 355.

## Noise and Drift Test

Noise and drift are measured according to ASTM Method No. E 1303-95 for Refractive Index Detectors used in Liquid Chromatography. For comprehensive information, refer to that ASTM method. The ASTM method also covers Flow Sensitivity, Minimum Detectability, Linear Range, Dynamic Range and Calibration. Specifications for the Varian ProStar 355 follow.

**Noise:**  $2.5 \times 10^{-9}$  RIU, static H<sub>2</sub>O in cell, temperature at 35°C for 2 hours

**Drift:**  $2.5 \times 10^{-7}$  RIU/hr under same conditions.

Measure drift before noise because noise requires the ProStar 355 to be operating at maximum sensitivity.

To perform both tests you need the following equipment:

- ProStar 355 with signal cable from REC OUT to a recorder set at appropriate voltage (1 mV or 10 mV) or from INTEG OUT to an integrator.
  - 5 mL glass syringe with Luer tip, and #16 needle with ferrule and nut (from Accessory Kit)
  - Degassed, filtered, deionized water, about 50 mL.
1. Turn ProStar 355 power and temperature control ON. Equilibrate unit at 35 °C. Room temperature must be greater than 18 °C for the ProStar 355 to maintain 35 °C internal temperature.
  2. Prepare the syringe and needle by placing the nut and ferrule onto the end of the needle, inserting the needle into the IN port of the ProStar 355 until it bottoms out. Hold the needle with one hand and swage the ferrule onto the needle with the 5/16" wrench. Remove the needle and assemble it to the syringe.
  3. Equilibrate the ProStar 355 by pumping deionized water through both reference and sample cells or with the syringe inject deionized water into the two cells.

*To equilibrate with a pump:*

- Set pump flow rate to 1 mL/min.
- Let flow for 5 minutes.
- Press Purge; flow path will include both sample and reference cells.
- Let flow for 5 minutes.
- Press Purge; flow path excludes reference cell.

*To equilibrate with a syringe:*

- Fill the 5 mL syringe with deionized water.
- Connect the syringe needle to the IN port of the ProStar 355.
- Press Purge; flow path will include both sample and reference cells.
- Inject 4–5 mL of water into the IN port.

---

NOTES: It is normal to feel considerable backpressure on the syringe. A 1–3 mL syringe (Varian part number 0099672118) can be used in place of the 5 mL syringe for less pressure.

Complete the noise and drift measurements with the pump off.

Elevating the waste container above this detector will help eliminate drift by preventing siphoning of mobile phase from the flowcell.

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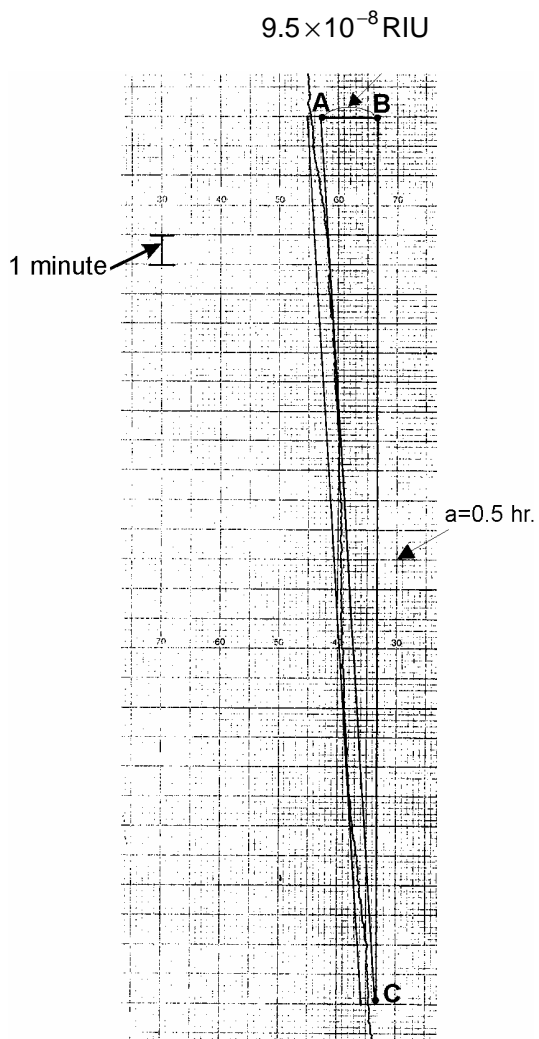
4. When the detector has stabilized, turn the Purge off to isolate the reference cell from the flow path. Press Zero to adjust signal zero to recorder zero. Monitor the signal to verify that output is approximately zero and remains stable.

#### **DRIFT**

5. Set the REC. RANGE to  $1 \times 10^{-6}$  RIU.
6. Turn pump flow off and disconnect the inlet line from the detector, if you equilibrated the detector with an HPLC pump. If you injected the deionized water into the cells with the needle, the needle can remain in the inlet.
7. Record thirty minutes of baseline. Draw two parallel lines in such a way that the entire thirty minutes of baseline is encompassed by the two parallel lines while keeping the vertical distance between these two parallel lines at a minimum. Refer to Figure 13.



8. Calculate the slope of either line in refractive index units per hour (RIU/hr). This is the static drift. See Figure 13 for an example.
9. If drift is outside of specifications, check to see if there are noticeable temperature fluctuations in the lab environment. If so, repeat the drift test with the ProStar 355 in an enclosure, such as a cabinet or box.



1. Collect at least 30 minutes of baseline at range of  $1 \times 10^{-6}$  RIU/FS.
2. Draw two parallel lines such that all data are enclosed, yet the distance between the lines is minimized.
3. Find the slope of either line in RIU/Hr so:

$$a=BC=0.5\text{hr.}$$

$$b=AB=9 \times 10^{-8}\text{RIU}$$

$$\text{SLOPE} = \frac{9 \times 10^{-8}\text{RIU}}{0.5\text{hr.}}$$

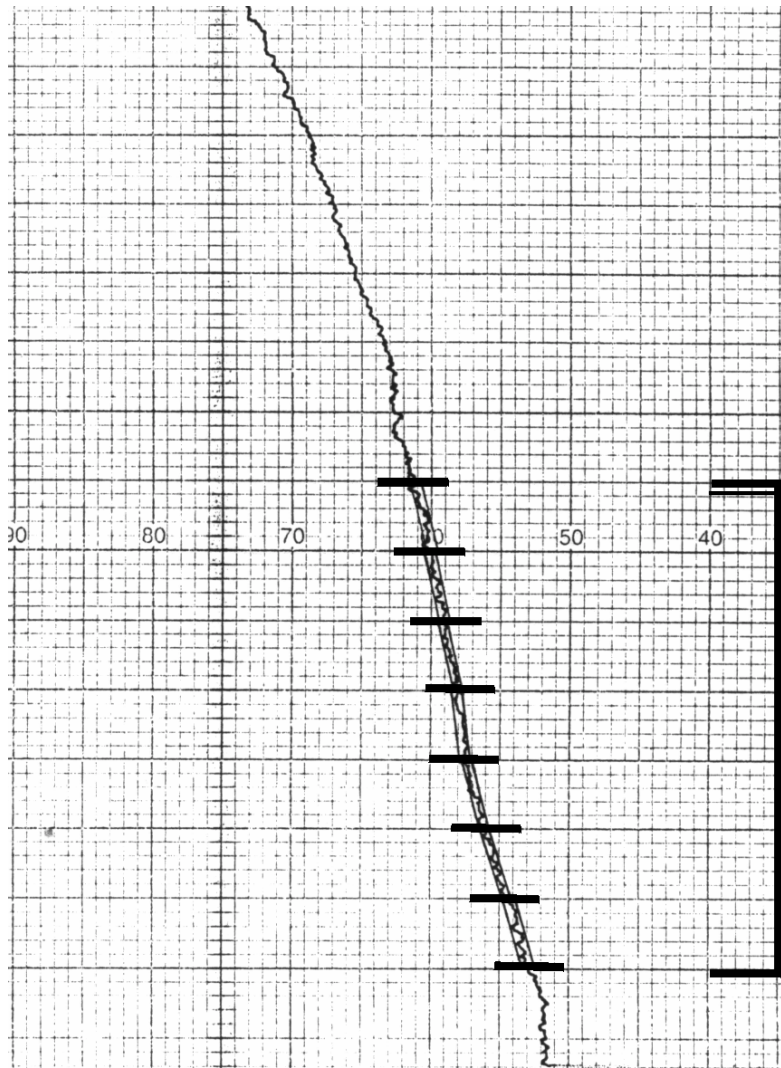
$$= 18 \times 10^{-8}\text{RIU/hr.}$$

$$\text{Noise} = 1.8 \times 10^{-7}\text{RIU/hr.}$$

Figure 13 Example: Drift Test Data of the ProStar 355 Detector

**NOISE**

10. Set attenuation REC. RANGE  $\frac{1}{4}$ . If instrument is not fully equilibrated, the signal will be very erratic. You may choose to monitor on REC. RANGE 1 until the signal is stable, then monitor at REC. RANGE  $\frac{1}{2}$  until signal is stable at that setting, then finally monitor at REC. RANGE  $\frac{1}{4}$ .
11. Collect 20 minutes of signal. The pen cannot go off scale during any portion of this 20 minute period. Divide the 20 minutes of signal trace into 1 minute segments with vertical lines.
12. For each minute segment: draw two parallel lines so that the entire minute of signal trace is encompassed by the two parallel lines while keeping the vertical distance between these two parallel lines at a minimum.
13. Measure the vertical distance between the two parallel lines in each minute segment. Convert these values to refractive index units (RIU). Look at the 20 minutes of noise data and find 15 consecutive minutes of noise when summed together, give the **smallest** total. Average these values to find detector noise. See below for an example of the ProStar 355 noise measurement.



1. Collect at least 15 minutes of baseline at range  $1/4 \times 10^{-6}$  RIU/FS.
2. Draw parallel lines one minute in length, such that all random variations in that minute are enclosed, yet the distance between the lines is minimized. See A.
3. Convert the distance between the parallel lines into refractive index units.
4. Find the mean value of this one minute noise over 15 minutes. Noise is  $\sim 1.8 \times 10^{-9}$  RIU in this example.
5. Effect of environment on baseline. B was taken in a thermally stable room. C was measured with the same detector less than one hour later beneath air conditioning vent.

Figure 14: (A) Example: Noise Test Data of the ProStar 355 Detector

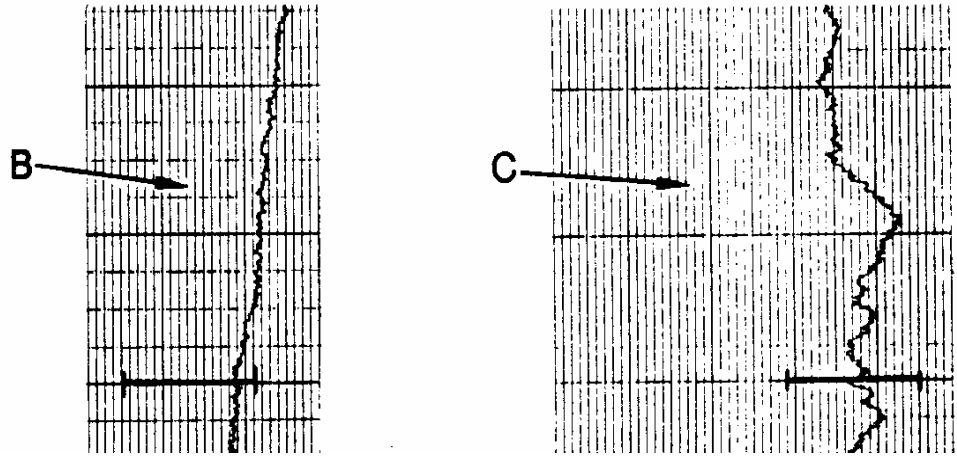


Figure 19 (B and C): Example: Noise Test Data of the ProStar 355 Detector

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## Examples Using the ProStar 355

### *Manual Mode*

1. Prepare the mobile phase from 900 mL of HPLC-grade methanol and 100 mL of distilled, deionized, filtered water.

The water and methanol should be measured separately, combined and thoroughly mixed with a magnetic stirring bar, and then degassed to avoid bubble formation in the RI flow cell. Magnetic stirring must be continued during chromatography to avoid gravity-induced phase changes.

---

**NOTE:** Do not construct a solvent mixture by pump proportioning a combination of solvents from 2 or 3 reservoirs. Refractive index detectors are too highly sensitive to small, short-term compositional changes in the mobile phase. Never attempt to use an RI detector with gradient programming.

---

2. Use a Microsorb MV C18, 4–6 mm x 15 cm column (part number R008620005) or equivalent reverse-phase column. Equilibrate column and system in the mobile phase.
3. The RI Test Sample can be purchased from Varian as a set of six sealed ampoules, part number 8200504806.
4. Use these chromatographic conditions. Refer to the chromatogram in Figure 15.

Sample: RI Test Sample  
Mobile Phase: 90% MeOH/10%/H<sub>2</sub>O at 1.0 mL/min  
Column: Microsorb MV C18, 4-6 mm x 15 cm  
Detector: Varian ProStar 355  
Attenuation:  $8 \times 10^{-6}$  RIU/FS  
Polarity: Normal position (Refer to operating parameters)  
Chart Speed: 1 cm/min  
Injection Volume: 20  $\mu$ L  
ProStar 355 Temperature: 35°C

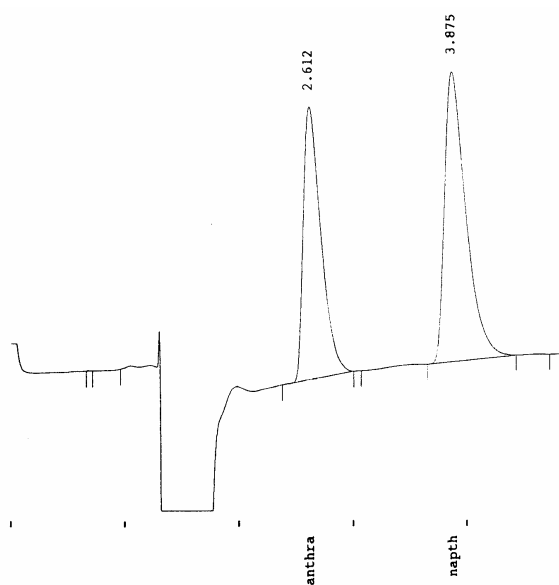


Figure 15: Chromatogram of RI Test Mixture

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## Shutdown Procedures

### **Corrosive Solvents**

Some solvents may corrode the detector if they are left in the detector, and should be thoroughly flushed from the entire system, including the reference and sample flow cell. See *Table 1 Solvent Limitation Recommendations*. The quartz cell window, in particular, is easily etched by strong bases. Do not turn power to the ProStar 355 off without rinsing these solvents from the detector.

Some solvents can be left in the cells at the end of an operation. For example, water, acetonitrile, 2-propanol, the xylenes, and paraffinic hydrocarbons are quite innocuous. They may be left in the ProStar 355 overnight or over a weekend.

### **No Flow Versus Reduced Flow**

A continuous slow flow through the ProStar 355 is the preferable shutdown procedure, especially if buffers, tetrahydrofuran and organohalocarbons are in use.

Reduced flow may be 0.5 mL/min to 0.01 mL/min but the HPLC pump must be able to stay primed at reduced flow. If the ProStar 355 is under external control, you may alternate between Purge on and Purge off while you flush at reduced flow.

**Buffers:** Even if the buffer is non-corrosive (see *Solvent Recommendations*, page 63 and *Corrosive Solvents*, page 90), it is better to keep the solvent flowing at a reduced rate to eliminate the possibility of salt precipitation in the ProStar 355 cells and tubing.

**Tetrahydrofuran:** Because THF does oxidize, you may find that, if you keep solvent flowing at a reduced rate, the chromatographic system takes less time to re-stabilize upon start-up. Generally a reduced-flow shutdown procedure will minimize re-stabilization time; the time saved is noticeable with THF as the solvent.

**Organohalocarbons, such as Methylene chloride and Chloroform:** Keep a small amount of flow to keep down the amount of corrosive chloride impurities in the cell.



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NOTE: Ethanol stabilized chloroform does not form chloride ions readily; therefore, it is recommended as a mobile phase if conditions permit.

---

### ***Continuous Power On Versus Power Off***

Leave the ProStar 355 power and heating on overnight and on weekends. This will keep the ProStar 355 close to operating conditions and minimize stabilization time. The heating temperature becomes more critical the more removed from room temperature you wish to operate.

### ***Long-Term Storage***

If the ProStar 355 will not be used for a week or more, the following storage procedure should be used.

1. Flush out **all** ProStar 355 lines, reference and sample cells (i.e., flush with Purge on then with Purge off) with at least 10 mL of solvent. This solvent should have the following characteristics.
  - No dissolved salts, acids, bases, or halides
  - Low vapor pressure at storage temperatures
  - Miscible and compatible with next probable chromatographic use of instrument
  - Does not support bacterial growth
  - Does not oxidize or form peroxides

Examples include: acetonitrile, acetonitrile in water in concentrations greater than 10% V/V, 2-propanol, xylenes, and heptane.

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NOTE: Buffers must be rinsed out with water before the introduction of acetonitrile to avoid precipitation of salts.

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2. Turn the power off.
3. Remove tubing lines to ProStar 355 and cap off. Label detector with tag describing storage solvent.



# Maintenance and Troubleshooting

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## Cell Cleaning Procedure

Many cases of performance degradation in sensitive instruments with flow-through cells are traceable to cell contamination. The use of filtered solvents with solvent reservoir filters (Varian part number 2718038700), an in-line filter between the pump and detector, and a pre-column will protect the cell from contamination and decrease the amount of cleaning required. However, contamination from trapped particulates or bubbles, from precipitates, or from thin films of residues can still occur.

### *Preparation for Cell Cleaning*

To introduce cleaning solution into the ProStar 355 by pump, run a tubing line directly from the pump outlet to the ProStar 355 IN port bypassing the column. Some materials should be injected directly into the ProStar 355 cells by syringe due to their high corrosivity or safety risk.

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**NOTE:** Remember that refractive index cells are stable to only 700 kPa (7 atm). Gently flush the cells under all conditions. If you encounter a large backpressure in the ProStar 355, proceed cautiously. You will be risking cell rupture, and cell assemblies should only be replaced by Varian Customer Support Representatives.

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### *Cell Cleaning Hints*

The following recommendations may be applicable to a variety of cleaning conditions:

1. Clean all internal lines of the ProStar 355 by injecting cleaning solution with Purge off, and inject cleaning solution again with Purge on.

2. Particulate matter can be removed by forcing liquid through the cell using the syringe and needle with tubing from your Accessory Kit. Sometimes it helps to reverse flow and inject in the OUT port. If liquid purging does not work, try gas purging. Replace cell liquid with a volatile (e.g., acetone) solvent. Purge and dry the cells with clean gas at about 50 psi. Gas flowing through the cell will sometimes dislodge particles. Also try reversing the flow direction.

For difficult entrapments, follow the above gas purge procedure with a liquid purge. When liquid hits a particle in a dry cell, it is generally more effective than a continuous liquid purge.

3. If you suspect that you have a problem with entrapped bubbles, purge with 2-propanol.
4. Heating the flow cells may remove marginally soluble material.

### **Acid Cleaning**



**WARNING:  
CHEMICAL HAZARD**

**Corrosive acids are used. Use extreme caution to avoid spillage on skin, clothing, or the instrument. Protective gloves are advised.**

#### **CAUTION**

**Never put hydrochloric acid in the cell. This acid in any concentration will corrode the cell.**

1. Diluted (10–20%) or concentrated nitric acid is a good cleaning solution.
2. The sample and reference cells should be filled with water or air (blown dry) before proceeding.



**WARNING:  
EXPLOSION HAZARD**

**Do not allow nitric acid to contact methanol. An explosion could result. Completely rinse the flow cell with water following cleaning with nitric acid.**

3. Connect the syringe to the IN or OUT port of the ProStar 355. To the opposite port, attach plastic tubing from the cleaning solution. For safety, always draw the cleaning solution through the cells with the syringe. Allow 3 to 10 minutes cleaning time; longer cleaning periods (>1 hour) may harm the cells.
4. Flush acid cleaning solutions from the cells with large amounts of water, such as 1 mL/min. Flush for 15 to 30 minutes.

### ***Other Cell Cleaning Procedures***

If buffers or solutions of high salt content have been in use, the cells may be contaminated by precipitated salt. Large amounts of distilled, deionized water, such as 1 mL/min, for up to several hours, is the simplest clean-up procedure. An elevated cell temperature will speed dissolution. The water wash can be acidified, if the precipitated salt is more soluble in acidic solutions. However, do not use strongly basic (pH 10 or higher) solutions as these will etch the refractive index cells.

If contamination is suspected when a non-aqueous solvent is in use, flush the cells with a solvent that is (1) miscible with your mobile phase, (2) a good solvent for the predicted contaminant, and (3) generally of greater polarity than your mobile phase. A number of HPLC references exist that classify the solvent properties of common HPLC eluents, and these may be used as a guide [L.R. Snyder, *Journal of Chromatography*, 92, 223 (1974); *Journal of Chromatography*, 16, 223 (1978)].

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## **Lamp Replacement and Adjustment**

### ***Lamp Removal and Replacement***

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NOTE: Following a lamp replacement, you may want to document the performance of the ProStar 355 detector for future reference. The following tests are recommended:

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- Span Adjustment (page 77)
- Noise and Drift Test (page 81)
- Chromatogram of RI Test Sample (page 88 and Figure 15)

The light source of the ProStar 355 is a low-power tungsten filament bulb. As such, it should have a long lifetime (10,000 hours is common). Do not replace a lamp until testing indicates the lamp has failed.



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

Remove the ProStar 355 cover. Locate the optics module, Figure 16, which is a black box on the left side of the ProStar 355. The lamp is located on the front of the optics module, behind the front panel underneath the Pre-amplifier PC Board, see Figure 17

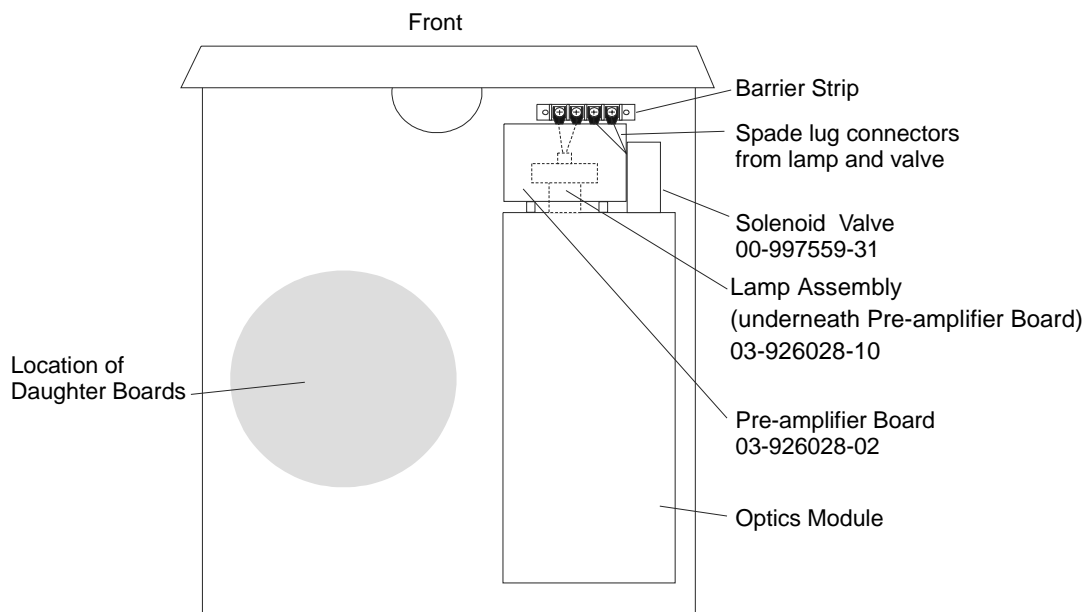


Figure 16: Interior Components of the ProStar 355

- 1 Outer module cover
- 2 Urethane foam insulant
- 3 Inner module cover
- 4 Flow cell assembly
- 5 Photodiode assembly
- 6 Lamp assembly
- 7 Mirror assembly
- 8 Mirror assembly screws (2)
- 9 Flow cells, with retainer
- 10 Flow cell retaining screws (2)
- 11 Optical unit
- 12 Mounting Plate for Pre-amplifier PC Board

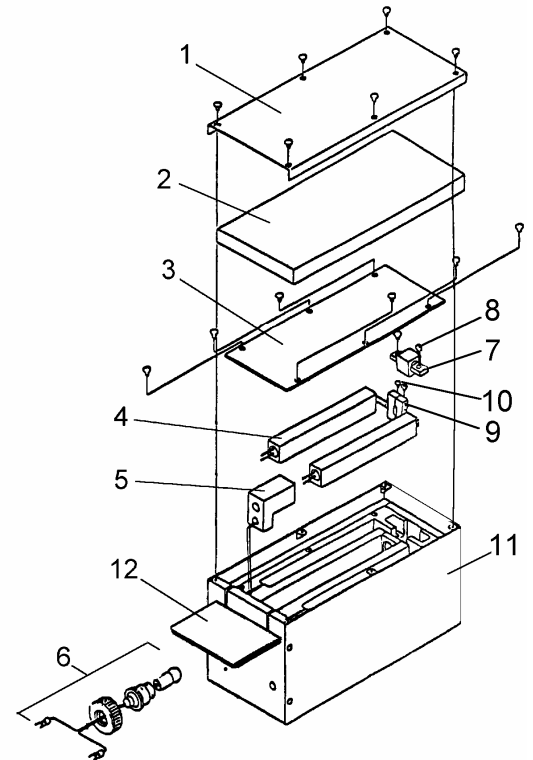
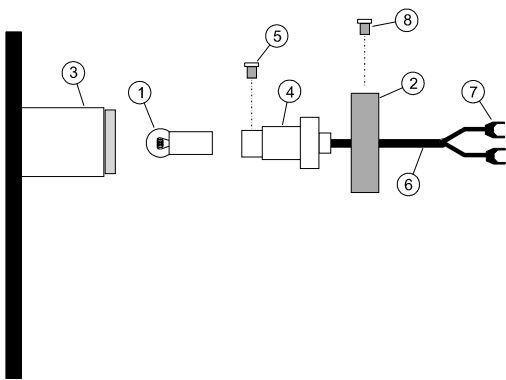


Figure 17: Optics Module

2. Test the lamp: disconnect the lamp power cable from its terminals. Using an ohmmeter, measure the resistance across the filament. An extremely high or infinite resistance indicates a burned-out filament. Next, reconnect the lamp to its terminals and turn ProStar 355 power on. The lamp should have a nominal voltage of 3 V. A voltage reading across the lamp filament of 5 V or more indicates a fatigued filament which should be replaced. However, if neither a burned-out nor fatigued filament is found, double check for other possible problems in your ProStar 355, (see *Troubleshooting*, page 102). Any adjustment of the lamp will necessitate lamp alignment and span adjustment.

3. To replace the lamp assembly remove the Pre-amplifier PC Board and remove the lamp power cable from its connection on the barrier strip on the inside of the front panel. Loosen the locking ring setscrew (8) in figure below with a 1.5 mm Allen wrench. Turn the lamp socket locking ring counterclockwise and remove the lamp assembly.
4. Hold the new lamp/socket assembly as indicated in. Figure 18. Notice that the lamp filament (1) is aligned with the vertical axis, as is one of the three setscrews (8), as shown in Figure 19. It is important that the filament (not the lamp's bulb) be centered within the concentric circles of the lamp socket. Leave the locking ring setscrew and lamp socket locking ring slightly loose for the light pass adjustment (page 99). Connect the spade lugs of the lamp power cable to its terminals.
5. Proceed immediately to page 99, *Adjustments*.



1. Lamp
2. Socket Locking Ring
3. Socket Holder
4. Socket
5. Setscrew (1 of 3)
6. Lamp Power Cable
7. Spade Lugs (2)
8. Locking Ring Setscrew (1)

*Items 1, 4, and 6 constitute the Lamp Assembly, 0392602810 on the ProStar 355.*

*Figure 18 Lamp Assembly Exploded View*



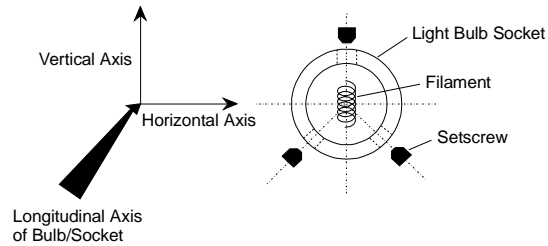


Figure 19 Orientation of Filament in Lamp Socket

## Adjustments

Two adjustments are necessary with lamp replacement: optical alignment of the lamp and span adjustment. Span adjustment is covered on page 77.

1. *Optical adjustment of lamp.* Remove the optics module cover (Figure 17). Remove the polyurethane heat insulator and remove the inside cover beneath it. If power is not yet on, turn it on at this time.
2. The figure below shows how the light from the source lamp should engulf and be centered around the slit entrance. Adjust the light pattern using the three lamp setscrews. Keep the filament aligned with the vertical axis. You may need to press the lamp assembly slightly in and out, if the light pattern is slightly off-centered. Sometimes, it is more appropriate to press the lamp assembly in the direction opposite the offset. When the light is centered, tighten the locking ring while holding the lamp assembly firmly in position. When optimal adjustment is attained, tighten the setscrews.

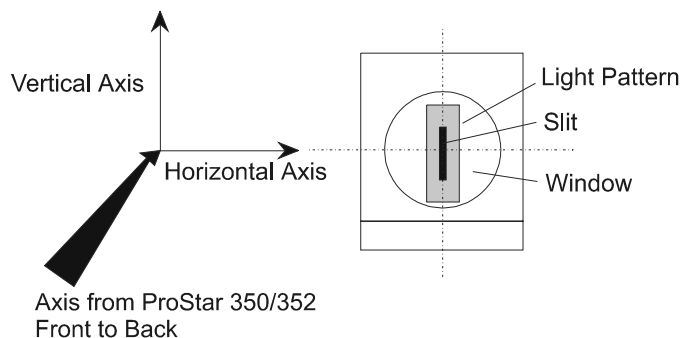


Figure 20 Optimal Light Pattern on Flow Cell Slit

3. Turn power off. Remove the lamp and socket from the optics module and clean it with methanol and a lint-free wipe. Reassemble lamp and socket in the optics module. Tighten the locking ring by hand, then tighten the locking ring setscrew with a 1.5 mm Allen wrench.
4. Turn power on. Check alignment of the filament and the light pattern on the slit. Repeat steps 2 and 3 if necessary.
5. Replace inside cover, polyurethane foam insulate, outside cover, and case cover.
6. Check span adjustment per page 77.

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## Testing of Flow Cell Integrity

Replacement of the ProStar 355 flow cells is a non-routine, exacting procedure. Only Varian Customer Support Representatives should replace flow cells. Additionally, the cells are well-hidden from inspection. Therefore, the following procedure is offered for determining if the cells have ruptured without disassembly of the ProStar 355.

The flow cell can be purchased only as part of the flow cell assembly.

### Checking the Drain Tube

The simplest way to check for cell rupture is to observe liquid coming out of the drain tube on the rear panel when liquid is flowing through the detector. However, the optics module would have quite a bit of liquid in it before liquid appears in the drain tube.

***Using the Span Adjustment Procedure***

1. Follow *Recorder and Integrator Span Test and Adjustment*, on page 77.
2. After flushing the sample cell well with water, re-zero the ProStar 355 detector. Verify that the detector is stable before continuing.
3. Perform steps 6 and 7 of the recorder procedure beginning on page 77. Note that you have been instructed to flush the reference cell with sucrose solution.
4. If the cells are intact, the recorder pen will deflect 50%  $\pm 5\%$ . Integrator (H) output will measure approximately 820 mV  $\pm 50$  mV.
5. However, if the cells are broken, the measured deflection will be noticeably diminished. This is because the water of the reference cell and the sucrose of the sample cell will dilute each other, making the span apparently out of adjustment at each successive attempt.
6. Do not conclude that the flow cells are ruptured prematurely. Retest the flow cells if a rupture is indicated.

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## Replacement of Flow Cell Assembly

To replace a flow cell assembly, contact your Varian Customer Support Representative. This is not a recommended procedure for the user.

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## Troubleshooting

Malfunctions within the ProStar 355 can arise from three general sources:

- The ProStar 355 itself can be dirty or operating non-optimally.
- The HPLC system can have a broken, dirty, or non-optimally operating component, but the problem is manifesting itself in the ProStar 355 detector.
- A mobile phase and/or column problem, which by its very nature is spread throughout the HPLC system but appears as a malfunction of the ProStar 355 detector.

To troubleshoot the ProStar 355, you must be able to separate the performance of the ProStar 355 within the HPLC system from its performance outside the HPLC system. Therefore, this section begins with guidelines for testing the ProStar 355 as a stand-alone.

Following is the *Troubleshooting Table*, page 104, which lists the observed Problem with the Possible Cause and the Suggested Solution.

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## Testing the ProStar 355 in Stand-Alone Mode

You must know how well the detector performs by itself before you can troubleshoot it in an HPLC system.

To perform the tests, disconnect all cables from the detector except one signal cable from REC OUT to a calibrated, functioning recorder. The recorder should match input to the output of the Varian ProStar 355 detector. Refer to page 56 for correct cabling.

Before proceeding, verify also that the ProStar 355 shipping bolts on the optics module have been loosened (see page 53); the power socket voltage matches the voltage indicated on the rear panel and the fuse is installed, (see page 52); and that degassed water is in the reference cell, the sample cell, and all hydraulic lines of the ProStar 355. Other solvents should be replaced according to the instructions in *Solvent Limitations*, page 63.

Test the performance of the ProStar 355 in stand-alone mode, using the Start-Up Sequence function.

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## Troubleshooting a HPLC System

As you go through the Troubleshooting Table, you will have occasional instructions for HPLC systems. Generally, however, the rule of thumb is to add one component at a time back into the HPLC system so that, should the condition arise again, the component causing the problem is indicated. You will begin by adding the pump to the ProStar 355 first and you will add the column last. If another type of detector is available, it is advantageous to use it before the ProStar 355 to aid in troubleshooting.

Example: you have a UV detector before the ProStar 355 and only the ProStar 355 has a noisy baseline. One possible implication is that the noise arises from pressure fluctuation, to which the refractive index detector is more sensitive. On the other hand, if both detectors are showing noise, a power line current may be indicated. If both detectors show anomalous baseline performance, such as huge peaks that continue indefinitely, a bleed-off problem (material from the column or immiscible solvents trapped in the system) is more likely.

## Troubleshooting Table

For further assistance phone 1-800-FOR-HPLC or contact your local Varian office.

Problem	Cause	Solution
Noise	Bubble in the pump.	Purge pump heads. Use only premixed/degassed mobile phase.
	Bubble in the detector.	Elevate waste reservoir above the level of the detector cells to create slight backpressure. Note: Do not add restrictor to waste line. Premix and degas mobile phase.
	Dirty flow cell(s).	Clean flow cells. Refer to Cell Cleaning procedure on page 93.
	Weak lamp.	Test lamp and replace or adjust as necessary. Refer to Lamp Test procedure on page 95.
	Environmental temperature fluctuations.	Move detector to a more stable environment. Place cover over the detector.
	Vapor pressure of the mobile phase is too high for the detector.  Example of difficult solvents above 30 °C: Acetone, cyclopentane, methyl t-butyl ether, trichlorotrifluoroethane, pentane, dichloromethane, ethyl ether, and many fluorocarbons.	Reduce or turn temperature off. Cool mobile phase, column, and detector.  Modify method to exclude or decrease concentration of troublesome solvent.
	Recorder or signal cable from the detector to the recorder is faulty or shorting.	Repair or replace defective cable. Examine all connections.
	Electrical transients from power line or radio frequency source.	Isolate the detector power source from other heavy equipment, motors, etc.  Use an EMI/RF power line filter to remove voltage spikes.  Ground the detector to earth.

Problem	Cause	Solution
<b>Noise appearing after 8–10 hours of operation</b>	Formation of gases in the mobile phase reservoir.	Continuously sparge the mobile phase with helium.
<b>Noise: cyclical</b>	Environmental temperature fluctuation.	Move the detector to a more stable environment. Place a cover over the detector
	Bubbles in reference cell.	Flush detector by turning the Purge on and off.
<b>Noise: cyclical matching pump stroke frequency</b>	Waste line too small.	Verify that correct exit line is installed. Inspect exit line for crimps
	Damper has inadequate damping capacity for system.	Add a second damper.
	Damper is operating at a pressure at which its performance is poor.	Add a restrictor coil between pump and injector valve.
<b>Drift</b>	Detector not yet thermally stabilized.	Monitor output signal 1–2 hours at 30 °C or overnight until stability is reached.
	Flow cell(s) dirty.	Clean references and sample cells. Refer to Cell Cleaning procedure on page 93.
	Flow cell(s) damaged.	Check for liquid in rear drain tube indicating a broken cell.  Test cell integrity. Refer to Cell Integrity Test procedure on page 100.
	Contamination from HPLC System.	Flush HPLC system with a solvent stronger than the mobile phase (less polar for reverse phase, more polar for normal phase, etc.) until contaminant disappears.
	Contaminated or non-HPLC grade solvents.	Prepare fresh mobile phase (premixed/degassed)

<b>Problem</b>	<b>Cause</b>	<b>Solution</b>
	<p>Vapor pressure of mobile phase is too high for operating temperature causing bubble formation in reference cell.</p> <p>Example of difficult solvents above 30 °C: Acetone, cyclopentane, methyl t-butyl ether, trichlorotrifluoroethane, pentane, dichloromethane, ethyl ether, and many fluorocarbons.</p>	<p>Reduce or turn temperature the temperature off.</p> <p>Cool mobile phase, column, and detector.</p> <p>Modify method to exclude or decrease concentration of troublesome solvent.</p>
	<p>Tetrahydrofuran (THF) in the mobile phase will oxidize in the reference cell.</p>	<p>Add an antioxidant to stabilize the THF, if compatible with other chromatographic requirements.</p> <p>Allow &gt;2 hours stabilization time for oxidation in reference cell to reach a steady state condition.</p>
<b>Baseline drift starts after several hours of analysis.</b>	<p>Reference cell solvent has aged and deteriorated.</p>	<p>Flush reference cell with mobile phase.</p>
<b>Baseline will not zero</b>	<p>Sample and reference cells do not contain identical solutions.</p>	<p>Flush sample and reference cells with mobile phase.</p>
	<p>Reference cell contains air bubbles.</p>	<p>Flush sample and reference cells with mobile phase.</p>
	<p>Flow cell(s) dirty.</p>	<p>Clean flow cell. Refer to Cell Cleaning procedure on page 93.</p>
	<p>Deteriorating lamp or lamp out of adjustment.</p>	<p>Test lamp and replace or adjust as necessary. Refer to the Lamp Test procedure on page 95.</p>
<b>Intensity Alarm Lights</b>	<p>Lamp is burned out.</p>	<p>Test lamp and replace or adjust as necessary.</p>
	<p>Flow cell(s) dirty.</p>	<p>Clean flow cell. Refer to Cell Cleaning procedure on page 93.</p>



# Appendix

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## Specifications

### **Performance Specifications**

<b>Detector type:</b>	Deflection
<b>Refractive index range:</b>	1.00–1.75 RIU
<b>Range:</b>	$\frac{1}{4}$ to $512 \times 10^{-6}$ RIU/FS
<b>Noise:</b>	$2.5 \times 10^{-9}$ RIU (Response: 1.5 s)
<b>Linearity:</b>	600 $\mu$ RIU
<b>Response time:</b>	0.1, 0.25, 0.5, 1.0, 1.5, 2, 3, 6 s
<b>Temperature control:</b>	OFF, 30–50 °C (1 °C increment). 77 °C fuse
<b>Flow cell volume:</b>	8 $\mu$ L
<b>Maximum flow rate:</b>	10 mL/min (mobile phase: pure water)
<b>Pressure rating:</b>	50 kPa (0.5 kgf/cm <sup>2</sup> )
<b>Internal volume:</b>	Inlet port/Flow cell: approx. 60 $\mu$ L Flow cell/Outlet port: approx. 520 $\mu$ L Total: approx. 590 $\mu$ L

### **General Specifications**

<b>Auto zero:</b>	Electrical and optical zeroing
<b>Auto zero range:</b>	All refractive index ranges
<b>Auto zero resolution:</b>	$\leq 1$ (@ 2 mV/ $\mu$ RIU) / 4 (@ 8 mV/ $\mu$ RIU) nRIU
<b>Offset range:</b>	0–500 mV (Integrator output) 0–50% (Recorder output)

<b>Offset resolution:</b>	10 mV (same with Integrator output sensitivity)
<b>Event marker:</b>	Marker out: 5% of FS
<b>Reference cell purge:</b>	Flow of mobile phase through a reference cell is controlled by the front panel PURGE key. When activated, a solenoid switch places both the reference and sample cell in line with the mobile phase.
<b>Polarity:</b>	Each momentary contact closure switches the event to its opposite state (ON to OFF, positive to negative).
<b>External signal input (contact closure):</b>	Auto Zero, Marker, Purge On/Off
<b>Signal output (contact closure):</b>	1) Ready 2) Solvent leak 3) Error; one of the following errors has occurred: Overheating, Low light intensity, Null glass home position error, Lost parameters, Optical balance Capacity: $\leq$ DC 24 V 0.1 A
<b>External communication:</b>	RS-232C
<b>Wetted material:</b>	SST316, PTFE, Quartz Glass
<b>Operator support:</b>	Span/Validation guide

### **Physical Specifications**

<b>Recorder output:</b>	0–10 mV/FS
<b>Integrator output:</b>	0–1 V/FS (Sensitivity: 2 mV/ $\mu$ RIU, 8 mV/ $\mu$ RIU)
<b>Power requirement:</b>	AC 100–240 V $\pm$ 10%; 50/60 Hz
<b>Power consumption:</b>	150 VA maximum
<b>Dimensions:</b>	260 mm (W) x 150 mm (H) x 430 mm (D) (a" W x b" H x c" D)
<b>Weight:</b>	13 kg (27 lb)
<b>Environment:</b>	Your instrument is designed for indoor use only. It is suitable for the categories stated on the front of this manual. Operation within specifications from 10 to 35°C, without failure from 0 to 50°C, and non-operational storage from -20 to 65°C. Operates in relative humidity from 5% to 95%. For optimum analytical performance, the ambient temperature of the laboratory should be between 20 and 25 °C and be held constant to within $\pm$ 2 °C throughout the entire working day.

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## Physical Constants of Select Solvents

From "High Purity Solvent Guide," Copyright 1982, Burdick and Jackson Laboratories, Inc., 1953 South Harvey Street, Muskegon, Michigan USA 49442. Reprinted with permission of publisher. All measurements were made on Burdick & Jackson solvents at 20°C unless otherwise indicated. Listed in order of increasing refractive index.

Methanol	1.3284	2-Methoxyethyl Acetate	1.4022
Water	1.3330	Cyclopentane	1.4064
Acetonitrile	1.3441	Methyl Isoamyl Ketone	1.4070
Ethyl Ether	1.3524	Tetrahydrofuran	1.4072
Trichlorotrifluoro-ethane (25°C)	1.3557	2-Ethoxyethanol	1.4077
Pentane	1.3575	Propylene Carbonate	1.4210
Acetone	1.3587	Dioxane	1.4224
Petroleum Ether	1.3650	Methylene Chloride	1.4241
Methylt-Butyl Ether	1.3689	Cyclohexane	1.4262
Ethyl Acetate	1.3724	Dimethyl Formamide	1.4305
Iso-hexanes	1.3740	Isopropyl Myristate	1.4332
Hexane	1.3749	Hexadecane	1.4340
2-Propanol	1.3772	Dimethyl Acetamide	1.4384
Methyl Ethyl Ketone	1.3788	Ethylene Dichloride	1.4448
Glyme	1.3796	Chloroform	1.4458
Diethyl Carbonate	1.3846	Carbon Tetrachloride	1.4601
1-Propanol	1.3856	N-Methylpyrrolidone	1.4700
Heptane	1.3876	Decahydronaphthalene	1.4751
Methyl n-Propyl Ketone	1.3901	Trichloroethylene	1.4767
Trimethylpentane	1.3914	Dimethyl Sulfoxide	1.4783
n-Butyl Acetate	1.3942	Toluene	1.4969
Methyl Isobutyl Ketone	1.3957	Benzene	1.5011
Isobutyl Alcohol	1.3959	o-Xylene	1.5054
2-Butanol	1.3972	Pyridine	1.5102
1-Butanol	1.3993	Chlorobenzene	1.5248
n-Butyl Chloride	1.4021	β-Phenethylamine	1.5332
2-Methoxyethanol	1.4021	o-Dichlorobenzene	1.5514

## Miscibility Chart

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Acetic Acid																							
Acetonitrile																							
Chloroform																							
Cyclohexane		X																					
Methylene Chloride																							
Dimethyl Formamide				X																			
Dioxane																							
Ethyl Ether																							
Hexane		X				X																	
Methanol				X										X									
Methyl t-Butyl Ether																							
Trimethylpentane		X				X								X									
Pentane		X				X								X									
Propanol-2																							
Tetrahydrofuran																							
Water			X	X	X			X	X		X	X	X										
	Acetic Acid	Acetonitrile	Chloroform	Cyclohexane	Methylene Chloride	Dimethyl Formamide	Dioxane	Ethyl Ether	Hexane	Methanol	Methyl t-Butyl Ether	Trimethylpentane	Pentane	Propanol-2	Tetrahydrofuran	Water							

X = Immiscible

## Polarity Index (P')

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Pentane	~0.0	Isobutyl Alcohol	~4.0
Trichlorotrifluoro-ethane	~0.0	2-Methoxyethyl Acetate	~4.0
Cyclopentane	~0.1	Methyl Isoamyl Ketone	~4.0
Heptane	~0.1	1-Propanol	4.0
Hexane	0.1	Tetrahydrofuran	4.0
Iso-hexanes	~0.1	Chloroform	4.1
Petroleum Ether	~0.1	Methyl Isobutyl Ketone	~4.2
Trimethylpentane	0.1	Ethyl Acetate	4.4
Cyclohexane	0.2	Methyl n-Propyl Ketone	~4.5
Hexadecane	~0.5	Methyl Ethyl Ketone	4.7
n-Butyl Chloride	~1.0	Dioxane	4.8
Trichloroethylene	~1.0	2-Ethoxyethanol	~5.0
Carbon Tetrachloride	1.6	o-Phenethylamine	~5.0
Toluene	2.4	Acetone	5.1
Methyl t-Butyl Ether	~2.5	Methanol	5.1
o-Xylene	~2.5	Pyridine	5.3
Benzene	2.7	Diethyl Carbonate	~5.5
Chlorobenzene	2.7	2-Methoxyethanol	5.5
o-Dichlorobenzene	~2.7	Acetonitrile	5.8
Ethyl Ether	2.8	Propylene Carbonate	6.1
Methylene Chloride	3.1	Dimethyl Formamide	6.4
Ethylene Dichloride	3.5	Dimethyl Acetamide	6.5
1-Butanol	3.9	N-Methylpyrrolidone	6.7
2-Propanol	3.9	Dimethyl Sulfoxide	7.2
2-Butanol	~4.0	Water	10.2
n-Butyl Acetate	~4.0		

Classification of the Solvent Properties of Common Liquids, L. R. Snyder, *Journal of Chromatography*, 92, 223 (1974); *Journal of Chromatography Science*, 16, 223 (1978).