OPERATOR'S MANUAL

FOR

PL-BV 400 HT VISCOMETER

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Agilent Technologies

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DECLARATION OF CONFORMITY

We, Varian, Inc.

Essex Road

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UK

declare that the product:

PL-BV 400 HT Viscometer Part # PL0810-3050

conforms with the requirements of EC Directives 89/392, 91/368 & 89/336 by complying with the following Harmonised European Standards:

Safety: EN61010 - 1 Class I

Installation category II Pollution degree 2

EMC: EN55022 Conducted emissions: Class B

Radiated emissions: Class B*

EN50082 ESD (C), Radiated immunity (A),

Fast Transient Burst (B), Surge

immunity (B), Conducted immunity (A).

SOm.

Dr. S.O'Donohue Head of Instrumentation, Church Stretton 3rd June 2010

PL-BV 400 HT VISCOMETER WARRANTY

(Extract from General Conditions of Sale)

Subject as hereinafter stated, if any goods supplied are proved to the reasonable satisfaction of the Seller to be defective in material or workmanship within a period of 12 months from the date of despatch and the Buyer notifies such defect to the Seller in writing within fourteen days of it becoming apparent the Seller shall repair or replace at its option the goods or any part thereof free of charge and any repaired (or replacement) goods will be guaranteed on these terms for the unexpired portion of the 12 month period PROVIDED THAT the Seller shall be under no liability in respect of any defect that has arisen because:-

of fair wear and tear; or

where the goods have not been used, maintained, stored or protected in the proper manner; or

the goods have been altered in any way whatsoever or have been subject to unauthorised repair; or

the goods have been improperly installed or connected (unless the Seller carried out such installation and connection); or

in the case of Instrument Consumables (including wetted parts, lamps etc) they prove defective as aforesaid more than 30 days after delivery notwithstanding the foregoing provisions of this condition; or

the Buyer is in breach of any other contract made with the Seller such as the Company's General Conditions of Sale.

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HEALTH AND SAFETY

PL-BV 400 HT Viscometer

This detector should be used only in accordance with the instructions stated within this manual. If the detector is used in a manner not specified by the manufacturer, the protection provided may be impaired. Users should observe the following general safety precautions:

Ensure that the instructions within this manual are understood and carried out in the operation of the detector. All persons utilizing the detector should have adequate training in its proper set-up, operation, and particularly its safety features.

No attempt should be made to service the detector without authorization from your Varian service department and contravention of this may result in personal hazard or damage to the detector and will invalidate the manufacturer's warranty.

We stress the importance of standard laboratory safe practice (e.g. COSHH regulations) for dealing with electronic laboratory equipment, solvents, etc., in preventing accidents, fires, or potentially hazardous conditions.

If in any doubt about the use of the detector contact your local Varian office or distributor.

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|---|---|--|
|---|---|--|

SAFETY

The following general safety precautions must be observed during all phases of operation, service, and repair of this detector. Failure to comply with these precautions or with specific warnings elsewhere in this manual violates safety standards of design, manufacture, and intended use of the detector. Varian, Inc. assumes no liability for the customer's failure to comply with these requirements.

Signs and Pictograms Used in this Manual



WARNING:

THE "WARNING SIGN" DENOTES A HAZARD. IT CALLS ATTENTION TO A PROCEDURE OR PRACTICE, WHICH, IF NOT CORRECTLY CARRIED OUT OR ADHERED TO, COULD RESULT IN SEVERE PERSONAL INJURY OR DAMAGE OR DESTRUCTION OF THE INSTRUMENT. PLEASE DO NOT PROCEED BEYOND A WARNING SIGN UNTIL THE INDICATED CONDITIONS ARE FULLY UNDERSTOOD AND MET.



ATTENTION:

The "ATTENTION sign" denotes relevant information. Read this information first before proceeding, it will be helpful or necessary to complete the task.



NOTE:

The "NOTE sign" denotes additional information. It provides the user with advice and suggestions to facilitate the operation of the instrument.

Safety Practices

The following general safety precautions must be observed during all phases of operation, service, and repair of this detector. Failure to comply with these precautions or with specific warnings elsewhere in this manual violates safety standards of design, manufacture, and intended use of the detector. Varian assumes no liability for the customer's failure to comply with these requirements.



Electrical Hazards

Disconnect the instrument from all power sources before removing protective panels.

Replace faulty or frayed power cords.

Check the PSU output voltage to confirm it is 5V DC before connecting it to the control unit.

Before the instrument is switched on ensure that the PSU is connected to protective earth via a grounded power socket. Any interruption of the protective earth grounding will cause a potential shock hazard that could result in serious personal injury. Whenever it is likely that the protection has been impaired, the instrument must be made inoperative and be secured against any unintended operation.

Ensure the PSU power switch and appliance coupler remain easily accessible at all times.



Cleaning

The individual or group responsible for the use and maintenance of this equipment must ensure that appropriate decontamination is carried out if hazardous material is spilt on or inside the instrument.

Before using any cleaning or decontamination method except those recommended by Varian, users should check with Varian that the proposed method would not damage the equipment.

See Appendix for detailed recommended procedures.



General Precautions

Perf orm peri odic _leak

checks on liquid supply lines and connections.

Do not allow flammable and/or toxic solvents to accumulate.

Follow recommended procedures and protocols for evacuation and disposal of flammable and/or toxic solvents. Never dispose of such products through municipal waste systems

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1 General Information

1.1 Introduction

The PL-BV 400 HT viscometer is based on the proven four-capillary design and is intended for online measurement of viscosity when integrated into the PL-GPC 120/220 or PL-XT 220 instruments. The combination of refractive index (RI) and viscosity detection provides accurate molecular weight determination for all polymer types based on the universal principle, as well as valuable branching information not otherwise obtainable from a concentration detector alone.

Due to the four-capillary technology, the differential pressure (DP) signal is very stable against flow rate fluctuations. A continuous monitor of the pressure through the bridge network also provides a consistent reference of the flow system, the inlet pressure (IP). The instrument can be used in both a batch mode, which yields an average intrinsic viscosity, and a true gel permeation chromatography (GPC) mode, which yields a slice-by-slice intrinsic viscosity distribution. From the intrinsic viscosity and known retention volumes or known Mark-Houwink-Sakurada coefficients, the molecular weight as well as distribution of a polymer may be determined.

The PL-BV 400 HT viscometer consists of four components.

- 1. Precision-engineered 4-capillary bridge
- 2. High sensitivity pressure transducers
- 3. Electronic control module
- 4. Discrete 5V power supply module (PSU)

The viscometer bridge is located in the PL-GPC 120/220 column oven and the column oven in the PL-GPC module of the PL-XT 220 instrument. The pressure transducer assembly is housed in the temperature controlled solvent module of the PL-GPC 220 or a dedicated compartment to the right hand side of the either the main compartment of the PL-GPC 120 or the PL-XT GPC module of the PL-XT 220 instrument. The electronic control module, where the differential pressure (DP) and inlet pressure (IP) signals are obtained, is stand-alone and located alongside the GPC system.

1.2 **Specifications**

| PL-BV 400 HT | Linearity | 0.5% FS |
|--------------|--|-----------------------------------|
| Viscometer | Shear rate (typical) | 3000 s ⁻¹ |
| | Sensitivity □ _{sp} | 1x10 ⁻⁵ Pa.s |
| | Baseline noise (for DP output) | <0.25mV |
| | Baseline drift (for DP output) | <5mV |
| | Capillary dimensions | 0.01 in. id x 24 in. I |
| | Recorder output differential pressure | 1mV/Pa |
| | Recorder output inlet pressure | 10 mV/Pa |
| | Cell volume | 120 μL |
| | Electrical Power – Control unit | 4.75-5.25V DC, 1.5A |
| | Electrical Power - PSU | 100-240V AC, 50/60Hz, 0.8A max |
| | Environmental – Control unit & transducers | 10 -40 °C, 10%-80%RH |
| | Environmental – Capillary bridge | 0 - 250 °C |
| | Dimensions (wxhxl) | 44 cm x 23.5 cm x 45 cm |
| _ | Weight (packed) | Approximately 50 lbs. (23 kg) |

1.3 Installation Procedures

1.3.1 Connecting the PL-BV 400 HT Electronic Control Module

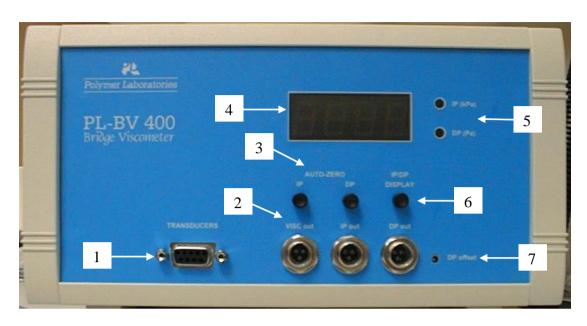


Figure 1.3.1.1 Front View of the Electronic Control Module for the PL-BV 400 HT Viscometer

- 1. Transducers cable connection port Connects the DP and IP pressure transducers to the control module.
- 2. Viscometer analog outputs; 1V FSD The viscometer analog outputs, DP, IP and Specific Viscosity (Visc).



THE SPECIFIC VISCOSITY OUTPUT CANNOT BE USED WITH CIRRUS ™ MULTI.

- 3. DP and IP autozero buttons Autozeros the DP and IP outputs. For the IP autozero button, the button needs to be pressed and held for a few seconds before the IP output is autozeroed.
- 4. DP/IP output display Displays the DP/IP output.
- 5. DP/IP output display indicators A red LED indicates which output is being displayed.
- 6. Toggle display button Toggles the output display between the DP and IP outputs.
- 7. DP offset adjustment screw Adjusts the DP zero offset by ±20Pa. To adjust the zero offset turn the screw either clockwise or anticlockwise to increase or decrease the zero value.

The electronic control module for the PL-BV 400 HT Viscometer should be connected as follows.

- Using the supplied transducer cable connect the 6 pin MIL-C connector end to the port on the right hand side panel of the PL-GPC 120/220 instrument or the PL-XT GPC module of the PL-XT 220 instrument and the 9 pin D connector end to the port labelled "Transducers" on the control module.
- 2. Using the Detector output cables supplied, attach one end of the cables to the Ports labelled "DP out and IP out" on the front of the control module and the other ends to the Detector Input Channels (e.g. Channel 2 and 3) on the front of the PL DataStream.



The PL-BV 400 HT Viscometer also has a Specific Viscosity output, which is obtained from the "Visc out" port.



THE SPECIFIC VISCOSITY OUTPUT CANNOT BE USED WITH CIRRUS ™ MULTI.

3. Connect the supplied 5V DC power supply unit to the rear of the control module and connect the power supply unit to an <u>earthed</u> mains supply using the power cable provided.



Powering the control module off and on will reset the output offsets.

1.3.2 Connecting the PL-BV 400 HT Viscometer Bridge

The PL-BV 400 HT viscometer bridge is connected in parallel to the RI detector. The plumbing connections are all situated within the column oven compartment. The viscometer bridge is located at the rear of the oven compartment on the right hand side and the RI detector is on the left hand side. The viscometer bridge and RI detector should be connected as follows.

1.3.2.1 Connecting into a PL-GPC 120/220 Instrument

- 1. Connect the supplied 30 cm 0.010 in. ID stainless steel tube from the outlet of the column to the supplied Valco T-piece.
- Connect the coiled 0.010 in. ID stainless steel tube inlets of the viscometer and RI detector to the Valco T-piece. Ensure the RI inlet is fitted with a Valco nut and ferrule.



The lengths of the inlet tubes are such as to obtain a flow split between the viscometer and RI of between 50-70%. See below for flow split ratio calculations. 3. The viscometer bridge outlet tube for the PL-GPC 220 is located in the solvent module similar to the RI detector outlet tube. The viscometer bridge outlet tube for the PL-GPC 120 is connected to the RI detector outlet tube via a Valco T-piece, which in turn is connected internally to the "Waste Out" port located on the front of the instrument.



The "Purge Out" port on the PL-GPC 120 is connected internally to the DP transducer purge lines.

CHECK ALL FLUID LINES FOR LEAKS AFTER INSTALLATION.

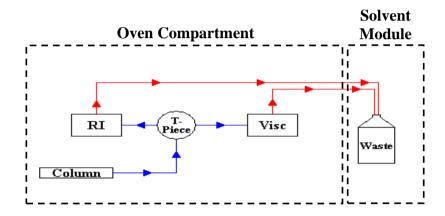


Figure 1.3.1 Plumbing Schematic of the PL-BV 400 HT Viscometer and RI Detector in the PL-GPC 220 Instrument

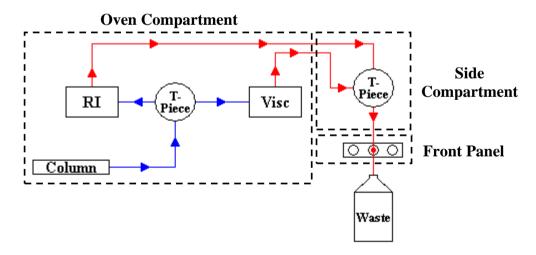


Figure 1.3.2 Plumbing Schematic of the PL-BV 400 HT Viscometer and RI Detector in the PL-GPC 120 Instrument

1.3.2.2 Connecting into a PL-XT 220 Instrument

- 1. Connect the supplied 30 cm 0.010 in. ID stainless steel tube from port 2 of the detector selector valve to the supplied Valco T-piece.
- 2. Connect the coiled 0.010 in. ID stainless steel tube inlets of the viscometer and RI detector to the Valco T-piece.



The lengths of the inlet tubes are such as to obtain a flow split between the viscometer and RI of between 50-70%. See below for flow split ratio calculations.

- 3. If a PL-ELS 1000 detector is fitted then connect the 0.040 in. ID stainless steel tube outlets of the viscometer and RI detector to the Valco T-piece within the PL-ELS 1000 module. Then connect a 0.040 in. ID stainless steel tube from the Valco T-piece to the T-connection. Connect the T-connection to the internal connection of the waste port on the rear panel of the PL-ELS 1000 module.
- 4. If a PL-ELS 1000 detector is not fitted then connect the 0.040 in. ID stainless steel tube outlets of the viscometer and RI detector to the Valco T-connection. Then connect the ½ in. OD PTFE tube to the T-connection and the internal connection of the waste port on the rear panel of the PL-XT GPC module.
- 5. Connect an ½ in. OD PTFE tube from the waste port on the rear of the PL -XT GPC module to the waste container.



The "Purge Out" port on the rear of the PL-XT GPC module is connected internally to the DP transducer purge lines.



CHECK ALL FLUID LINES FOR LEAKS AFTER INSTALLATION.

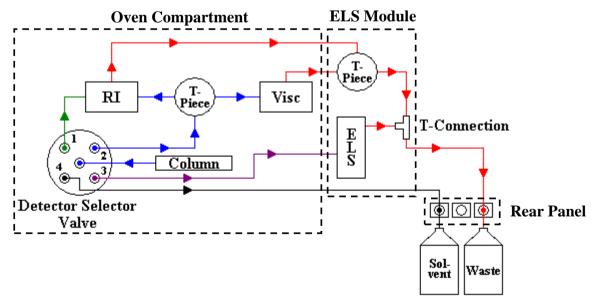


Figure 1.3.3 Plumbing Schematic of the PL-BV 400 HT Viscometer and RI Detector in the PL-XT 220 with a PL-ELS 1000 Detector Fitted

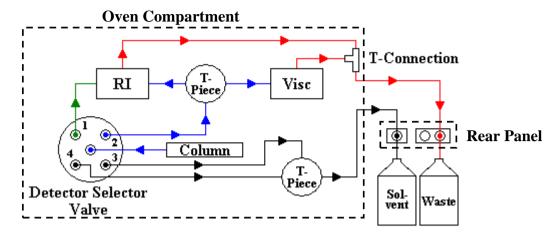


Figure 1.3.4 Plumbing Schematic of the PL-BV 400 HT Viscometer and RI Detector in the PL-XT 220 without a PL-ELS 1000 Detector Fitted

Flow Split Ratio Calculations

The flow split ratio is the ratio between the recorded inlet pressures (IP) from the viscometer with and without the RI detector connected in the system.

$$F \qquad \text{SI} \quad \text{oRp} \quad \text{w=1} \\ \begin{matrix} I & P_{RV} & I \\ i & \text{the inset} \\ I & P_{VO} & i \text{ nset} \end{matrix} \end{matrix} \quad \stackrel{c}{\text{o}}$$

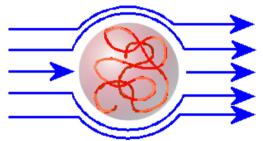
Therefore if the flow split ratio were 60% then 60% of the flow would be going through the viscometer, i.e. if running the system at 1.0 mL/min then the flow through the viscometer would be 0.6 mL/min.

To record the IP for the viscometer only disconnect the stainless steel tube connecting the RI inlet to the T-piece and connect a column end plug to the T-piece. Autozero the IP output and set the same flow rate as used when both detectors are connected. Purge both the IP and DP transducers and allow the system to stabilize. Once the system has stabilized record the IP.

2 Overview of Viscometry Theory

2.1 Theory of Online Viscosity Measurements

When a polymer dissolves in a liquid, the interaction of the two components stimulates an increase in polymer dimensions over that of the unsolvated state. Due to the vast difference in size between solvent and solute, the frictional properties of the solvent in the mixture are drastically altered, and an increase in viscosity occurs which should reflect the size and shape of the dissolved solute, even in dilute solutions.



The dissolved polymer coil disturbs the linear flow of solvent, resulting in a resistance to flow, which is observed as an increase in viscosity.

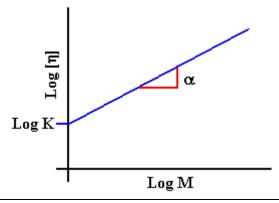
This was first recognized in 1930 by Staudinger, who found an empirical relation between relative magnitude of increase in viscosity and the molar mass of the polymer.

The Intrinsic viscosity $[\eta]$ is a measure of the viscosity of a polymer solution, as the concentration tends to zero, i.e. at very low concentrations. It is this parameter, therefore that is a characteristic of isolated polymer chains in solution and can be considered to be proportional to the density of the polymer coil.

For a given polymer and solvent system at a specified temperature, can be related to molecular weight, M, through the Mark-Houwink-Sakurada equation.

$$[\eta] = KM^{\alpha}$$

Where K and α are coefficients specific to the polymer, solvent system and temperature. These can be determined by calibrating with polymer standards of known molecular weights. A plot of Log($[\eta]$) versus Log(M), a Mark-Houwink-Sakurada plot, will be a straight line as long as the Universal Calibration is obeyed.



Values of α reflect the size of the molecule in solution and for a random coil polymer, range between 0.5 for a polymer dissolved in a theta solvent to about 0.8 in a very good solvent.

2.2 Universal Calibration

Benoit (1967) showed that polymers of different structure fall on the same calibration curve if the intrinsic viscosity is included as a calibration parameter. A calibration plot of the product of intrinsic viscosity and molecular weight ($[\eta]$.M) versus elution volume is a "Universal Calibration" as shown below in Figure 2.2.1. This allows the system to be calibrated with polymer standards of one type and then analyze polymers of another type and still obtain accurate molecular weight distributions.

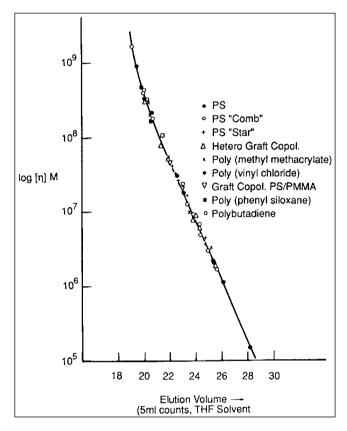


Figure 2.2.1 A Universal Calibration Plot

Therefore for an unknown polymer sample the Universal Calibration is used to convert the intrinsic viscosity, determined for each slice across the distribution from the viscometer, to molecular weight.

2.3 Polymer Branching

The determination of the degree of long chain branching has long occupied polymer chemists. In polymer solutions, the branching factor, g, is the starting point for many branching calculations.

Where $\eta_{\,b}$ is the intrinsic viscosity of the branched polymer and $\eta_{\,l}$ is the intrinsic viscosity of the linear polymer of the same molecular weight. ϵ is the "structural model" parameter, which is user defined.

2.4 Four Capillary Viscometry Theory

The PL-BV 400 HT Viscometer employs the fluid flow equivalent of the analogous Wheatstone bridge electrical circuit. Solvent travels down a bridge of four capillaries of equal resistance arranged as shown in Figure 2.4.1.

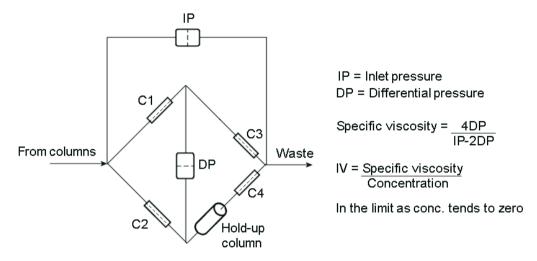


Figure 2.4.1 Plumbing Diagram of PL-BV 400 HT Viscometer

The differential pressure transducer monitors the pressure drop across the bridge, ΔP , and the inlet pressure transducer measures the pressure drop through the bridge, P_i . When the sample is introduced across capillary 4 (C4) of the bridge, an imbalance is caused due to the hold-up columns. This change in pressure, ΔP is proportional to the specific viscosity, η_{SD} , according to the following equation.

Where ΔP is the differential pressure across the bridge and P_i , is the pressure of the flow through the capillary bridge. The instrument makes a true differential measurement directly, making it very sensitive to dilute solution viscosity measurements.

The intrinsic viscosity, [η] is ideally obtained by measuring the specific viscosity values at several finite concentrations and extrapolate to zero concentration.

However, the concentrations used in GPC are low enough for the equation to be valid over a typical chromatogram, and thus an extrapolation to the true intrinsic viscosity is not required, since the concentration is close enough to the limiting concentration.

All of the data acquisition and data processing are accomplished using Cirrus[™] Multi software, which enables calculation of molecular weight and intrinsic viscosity

distributions using algorithms for Universal Calibration. Branching information can also be obtained from $\mathsf{Cirrus}^\mathsf{TM}$ Multi.

3 Operation

3.1 Powering the Viscometer ON/OFF

The PL-BV 400 HT is powered on and off using a mains power socket with the supplied 5V DC power supply unit and mains power cable. Switching this on provides power to the electronic control module. Confirmation of power on is provided when the output display on the module displays a value and one of the output display indicators is illuminated.



Powering the control module off and on will reset the output offsets.

3.2 Purging the Viscometer

3.2.1 For The PL-GPC 120 and PL-XT 220 Instruments

To purge the inlet pressure (IP) and differential pressure (DP) transducers, ensure the solvent is flowing through the system. Then on the front of the side compartment turn the knob labelled IP to *PURGE*. The IP transducer and its connecting tubes will then be flushed through with solvent. After purging the IP transducer turn the IP knob to *RUN* and the knob labelled DP to *PURGE* this will switch the solvent flow so that the DP transducer and its connecting tubes are flushed with solvent. After purging the DP transducer turn the DP knob to *RUN* this will switch the solvent flow back to normal operation, i.e. through the viscometer bridge.



Purge the IP and DP transducers for at least 5 minutes, at a flow rate of 1.0 mL/min, or until the IP/DP meter shows a steady signal.



For the PL-GPC 120 the waste line for the DP transducer is connected internally to the "Purge Out" port on the front right hand side of instrument and therefore a waste line needs to be connected to this port.



For the PL-XT 220 the waste line for the DP transducer is connected internally to the "Purge Out" port on the rear panel of the PL-XT GPC module and therefore a waste line needs to be connected to this port.

3.2.2 For the PL-GPC 220 Instrument

To purge the inlet pressure (IP) and differential pressure (DP) transducers, ensure the solvent is flowing through the system. Then in the solvent module at the front right hand side of the compartment turn the knob labelled IP to *PURGE*. The IP transducer and its connecting tubes will then be flushed through with solvent. After purging the IP transducer turn the IP knob to *RUN* and the knob labelled DP to *PURGE* this will switch the solvent flow so that the DP transducer and its connecting tubes are flushed with solvent. After purging the DP transducer turn the DP knob to *RUN* this will switch the solvent flow back to normal operation, i.e. through the viscometer bridge.



Purge the IP and DP transducers for at least 5 minutes, at a flow rate of 1.0 mL/min, or until the IP/DP meter shows a steady signal.



The waste line for the DP transducer is a permanent waste line found in the solvent module.

4 Troubleshooting

4.1 General Troubleshooting Guide

DP/IP Baseline Drift

Allow sufficient time for system to equilibrate.

Ensure the instrument is located in a thermally stable environment.

Degas solvent.

Purge DP/IP transducers.

Ensure laboratory temperature not changing dramatically.

DP/IP Baseline Noise

Purge DP/IP transducers thoroughly to remove any air bubbles.

Purge pump head to remove any air bubbles.

Degas solvent.

Low Sensitivity

Purge DP/IP transducers.

Check flow split.

Long Retention Times

Check pump flow rate.

Check for leak on system.

Appendix 1 - Spare Parts List

Viscometer Spares

| Part Number | Product Description |
|-------------|---|
| | |
| PL0810-3071 | PL-BV 400 Bridge Assembly for BV 400HT (PL-GPC 120/220) |
| PL0810-3087 | PL-BV 400 Bridge Assembly for BV 400RT (PL-GPC 50) |
| PL0810-3072 | PL-BV 400 Transducer Assembly (PL-GPC 220) |
| PL0810-3073 | PL-BV 400 Transducer Assembly (PL-GPC 120) |
| PL0810-3074 | PL-BV 400 Electronics Control Module |
| PL0810-3076 | PL-BV 400 Delay Columns (x2) |
| PL0810-3077 | PL-BV 400 Pulse Dampener |
| PL0810-3078 | PL-BV 400 IP Transducer |
| PL0810-3079 | PL-BV 400 DP Transducer |
| PL0810-3080 | PL-BV 400 IP Purge Valve |
| PL0810-3081 | PL-BV 400 DP Purge Valve |
| PL0810-3082 | PL-BV 400 Transducer to Bulk Head Cable |
| PL0810-3083 | PL-BV 400 Control Cable |
| PL0810-3084 | PL-BV 400 Display PCB |
| PL0810-3085 | PL-BV 400 Main PCB |
| PL0810-3086 | PL-BV 400 PSU |

Appendix 2 - Cleaning & Decontamination Procedures



SWITCH OFF AND DISCONNECT POWER CORD FROM INSTRUMENT BEFORE CLEANING OR DECONTAMINATION.

Cleaning

The exterior of the instrument should be cleaned by wiping down with a soft cloth moistened with dilute detergent solution, followed by wiping down with a cloth moistened with deionized water. Do not attempt to clean any of the connectors on the front or rear panels; water may gain ingress and accessible pins may become bent. Ensure that no moisture enters the instrument. Allow instrument to dry off completely before reconnecting power.

Decontamination

The operator should wear appropriate personal protective equipment for this operation (gloves, safety glasses, lab coat and respirator if level of hazard has been risk-assessed to be sufficiently high).

Disconnect the instrument completely and remove it to a fume cupboard if necessary. Open the case of the control unit (undo the 4 cross-head screws on the rear panel) to see if any solvent has contaminated the interior. The PSU module cannot be serviced and should be replaced if it becomes contaminated.

Excess quantities of liquid spilt on or inside the instrument should be mopped up using absorbent cloths, followed by repeated wiping down with soft cloths moistened with Acetone or deionized water (as appropriate to the spilt solvent) until the last traces of the hazardous liquid have been removed.

Identify and if possible correct source of the leak. Inspect cabling, parts and surfaces to determine whether any damage has occurred. If in doubt, contact your local Varian Service Dept for assistance.

Allow the interior and exterior of the instrument to dry out completely before closing the case and reconnecting power.

Dispose of contaminated waste appropriately.