

OPERATOR'S MANUAL

FOR

PL-BV 400 RT VISCOMETER

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Agilent Technologies

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DECLARATION OF CONFORMITY

We, Varian, Inc.
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declare that the product:

PL-BV 400 RT Viscometer Part # PL0810-3060

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

Safety: EN61010 - 1

EMC: EN55022

EN50082

Conducted emissions: Class B

Radiated emissions: Class B*

ESD (C), Radiated immunity (A),

Fast Transient Burst (B), Surge

immunity (B), Conducted immunity (A).

Dr. S.O'Donohue
Head of Instrumentation, Church Stretton
8th June 2010

PL-BV 400 RT 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 RT 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.

Access covers should not be removed by anyone other than properly trained personnel. No attempt should be made to service the detector without authorization from Varian's 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 local 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 assumes no liability for the customer's failure to comply with these requirements.

Signs and Pictograms Used in this Manual



WARNING

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

ATTENTION:

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



NOTE

NOTE:

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



NOTE

General Precautions

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.

Perform periodic 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



WARNING

Cleaning

The individual or group responsible for the use and maintenance of this equipment must carry out appropriate decontamination 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.

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1. General Information

1.1 Introduction

The PL-BV 400 RT viscometer is based on the proven four-capillary design and is intended for online measurement of viscosity when integrated into the PL-GPC 50 Plus instrument. 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 accessible from a concentration detector alone.

Due to the four-capillary technology, the differential pressure (DP) signal is very stable to flow rate fluctuations. A continuous monitor of the pressure through the bridge network also provides a consistent reference of the system flow; the inlet pressure (IP). The instrument can be used in both a batch mode, which yields an average intrinsic viscosity, and in 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 RT Viscometer consists of three components.

1. Precision engineered 4-capillary bridge
2. High sensitivity pressure transducers
3. State of the art low-noise electronics

The viscometer is located in the detector compartment section of the PL-GPC 50 Plus with the inlet and outlet of the detector accessed within the column oven compartment. The differential pressure (DP) and inlet pressure (IP) signals are obtained from the rear panel of the PL-GPC 50 Plus, analog output channels 3 (DP) and 4 (IP).

1.2 Specifications

PL-BV 400 RT Viscometer	Linearity	0.5% FS
	Shear rate (typical)	3000 s ⁻¹
	Sensitivity η_{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. L
	Recorder output differential pressure	1mV/Pa
	Recorder output inlet pressure	10mV/Pa
	Cell volume	120 μ L

1.3 Installation Procedures

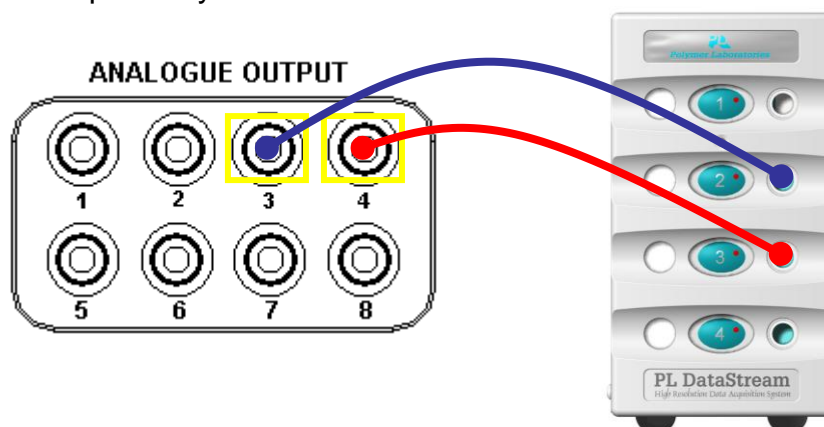
1.3.1 Configuring the PL-BV 400 RT Viscometer

The PL-BV 400 RT Viscometer needs to be added to the instrument configuration in order to control it from the PL Instrument Control software. To add the viscometer to the configuration open the PL Instrument Configuration Editor program and select *Add Component* from either the Configure menu or toolbar icon. From the Add Component dialog select the PL-BV 400 RT component. Once the viscometer component has been added assign the correct Com port.

For further information on configuring the system then please see the on-line help within the PL Configuration Editor program and the PL-GPC 50 Plus Operator's Manual.

1.3.2 Connecting the Detector Outputs to the PL DataStream

Using the Detector output cables supplied, attach the BNC connector ends of the cables to the Ports labelled "Analogue Output 3 and 4" on the rear of the PL-GPC 50 Plus and the other ends to the Detector Input Channels (e.g. Channel 2 and 3) on the front of the PL DataStream. The differential pressure (DP) and the inlet pressure (IP) outputs from the PL-BV 400 RT viscometer are connected internally to analogue outputs 3 and 4 respectively.



The PL-BV 400 RT Viscometer also has a Specific Viscosity output, which is obtained from analogue output 8.



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

1.3.3 Connecting the PL-BV 400 RT Viscometer Inlet/Outlet

The PL-BV 400 RT Viscometer is connected in parallel to the RI detector. The plumbing connections are all situated within the column oven compartment. The PL-BV 400 RT Viscometer and RI detector should be connected as follows.

1. Connect the supplied 30 cm 0.010 in. ID stainless steel tube from the outlet of the column to one of the supplied Valco T-pieces.
2. From the same Valco T-piece connect the supplied 10 cm 0.010 in. ID stainless steel tube to the inlet of the viscometer (bottom connection of the viscometer) and connect the approximately 40 cm 0.010 in. ID stainless steel tube to the inlet of the RI detector (bottom connection for the PL-RI detector and top connection for the Knauer RI detector).



The length of tube to the RI inlet will vary so as to obtain a 50:50 flow split between the RI and viscometer. See below for flow split ratio calculations.

3. Connect the two supplied 10 cm 0.040 in. ID PTFE tubes from the outlets of the viscometer and RI to the other Valco T-piece.
4. Connect the PTFE tube labelled with the blue band, the waste tube to the same Valco T-piece.

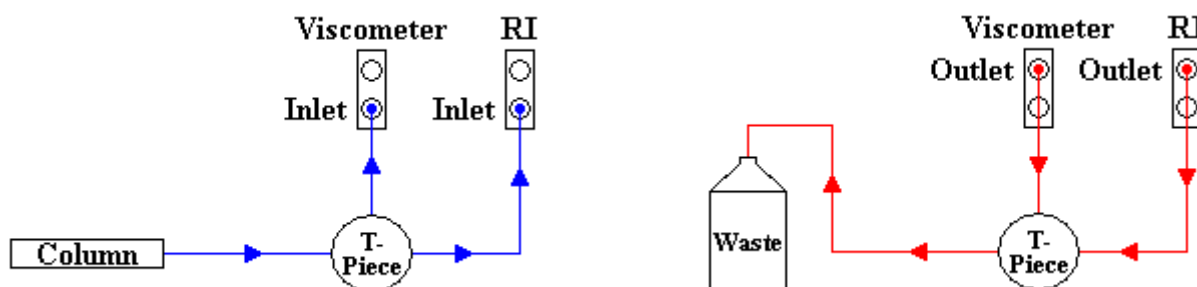


Figure 1.3.3.1 Schematics of the Inlet and Outlet Connections for the PL-BV 400 RT Viscometer and the PL-RI Detector in Parallel

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.

$$\text{Flow Split Ratio} = \frac{\text{IP}_{(\text{RI} + \text{Visc})}}{\text{IP}_{(\text{Visc Only})}} * 100$$

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 Valco T-piece and connect a column end plug to the T-piece. Autozero the IP output at zero flow and then set the same flow rate as used

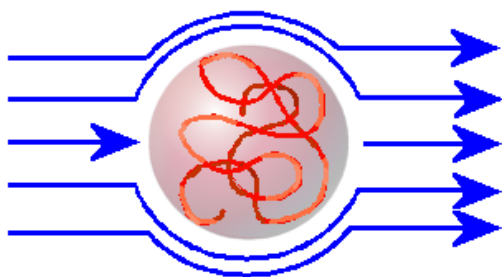
Chapter 1. General Information

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 value.

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.

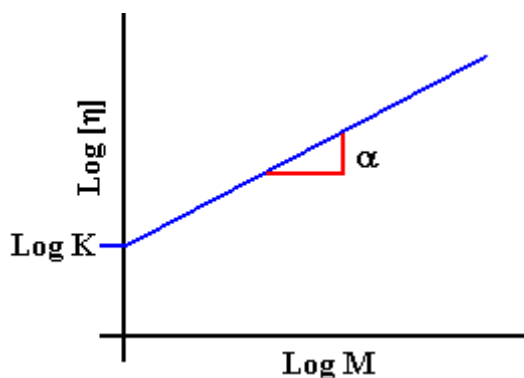
This was first recognized in 1930 by Staudinger, who found an empirical relation between relative magnitude of increase in viscosity and the molar mass of the polymer.

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, $[\eta]$ 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 $\text{Log}([\eta])$ versus $\text{Log}(M)$, 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 ($[\eta] \cdot M$) 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.

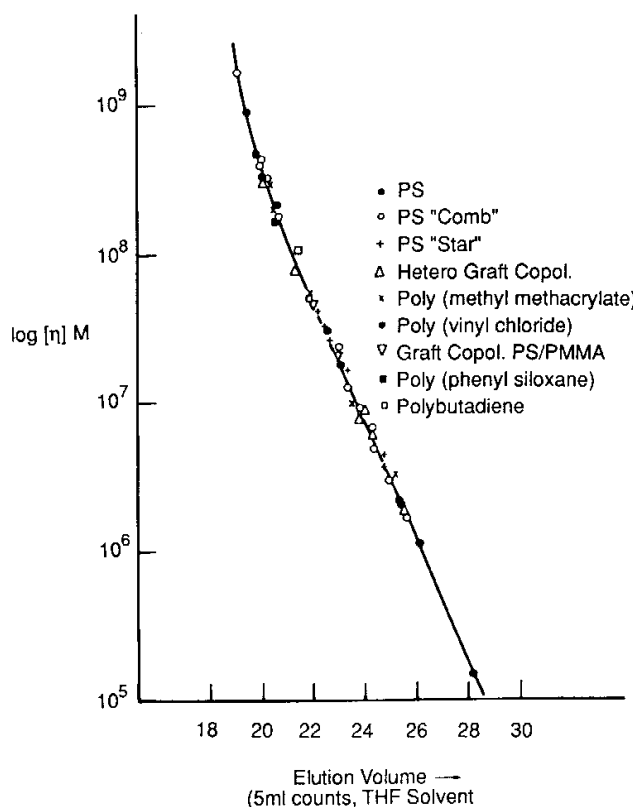


Figure 2.2.1 A Universal Calibration Plot

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.

$$g = \left(\frac{[\eta_b]}{[\eta_l]} \right)^{1/\varepsilon}$$

Where η_b is the intrinsic viscosity of the branched polymer and η_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 RT 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.

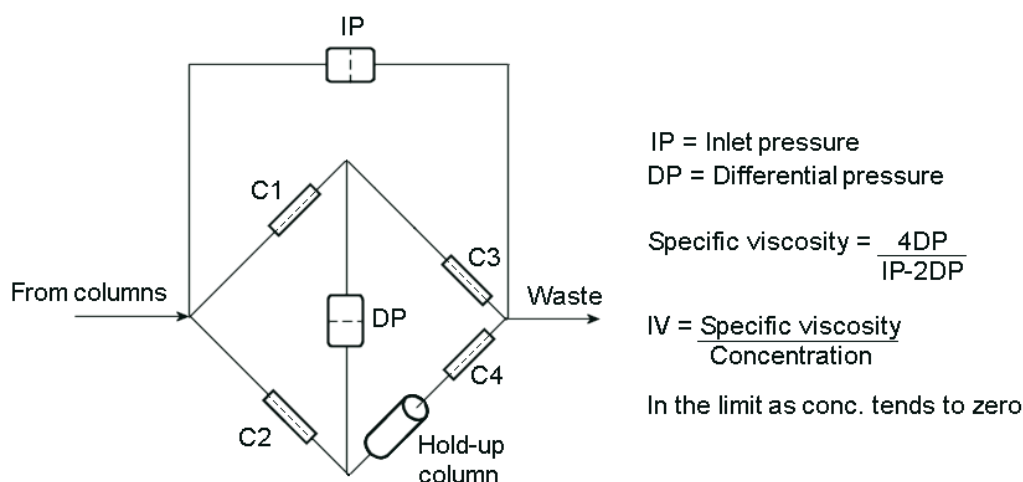


Figure 2.4.1 Plumbing Diagram of PL-BV 400 RT Viscometer

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 holdup columns. This change in pressure, ΔP is proportional to the specific viscosity, η_{sp} , according to the following equation.

$$\eta_{sp} = \frac{4\Delta P}{P_i - 2\Delta P}$$

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, $[\eta]$ is ideally obtained by measuring the specific viscosity values at several finite concentrations and extrapolate to zero concentration.

$$[\eta] = \lim_{c \rightarrow 0} (\eta_{sp}/c)$$

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 Cirrus Multi.


3. The Graphical User Interface

A Windows based Graphical User Interface (GUI) providing total instrument control is used to operate the PL-BV 400 RT Viscometer as well as the other components of the PL-GPC 50 Plus and the PL-AS RT & MT autosamplers. This intuitive interface provides simplistic control as well as a comprehensive monitoring system. The instrument status can be rapidly assessed for each of the instruments modules.

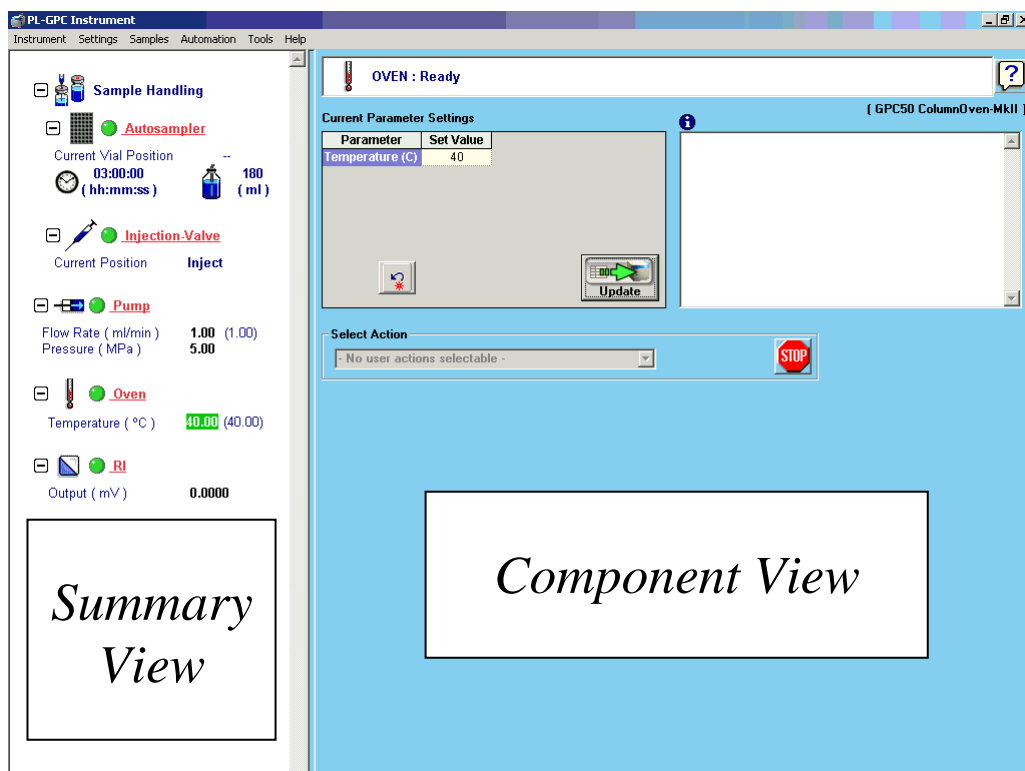


NOTE

For further information on the PL Instrument Control software please see the on-line help within the program.

To start the control software select the PL Instrument Control item  in the PL Instrument Control program group of the Programs option in the Start menu.

3.1 Overview



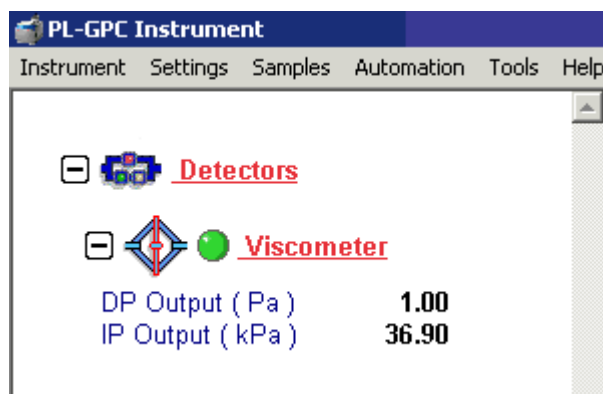
The Control screen is effectively divided into **two** main Views, these are:

1. The **Summary View**
2. The **Component View**

3.2 Summary View

The summary view displays all the components of your PL-GPC 50 Plus instrument as a "Tree" providing you with status for each of the modules and a quick way of access to the various components, parameters and options available within the PL Instrument Control software.

For the PL-BV 400 RT Viscometer the status information within the *Summary View* reports the differential and inlet pressure outputs, DP and IP.



Clicking on the component names displayed in red in the *Summary View* of the screen changes the control display shown in the *Component View*.

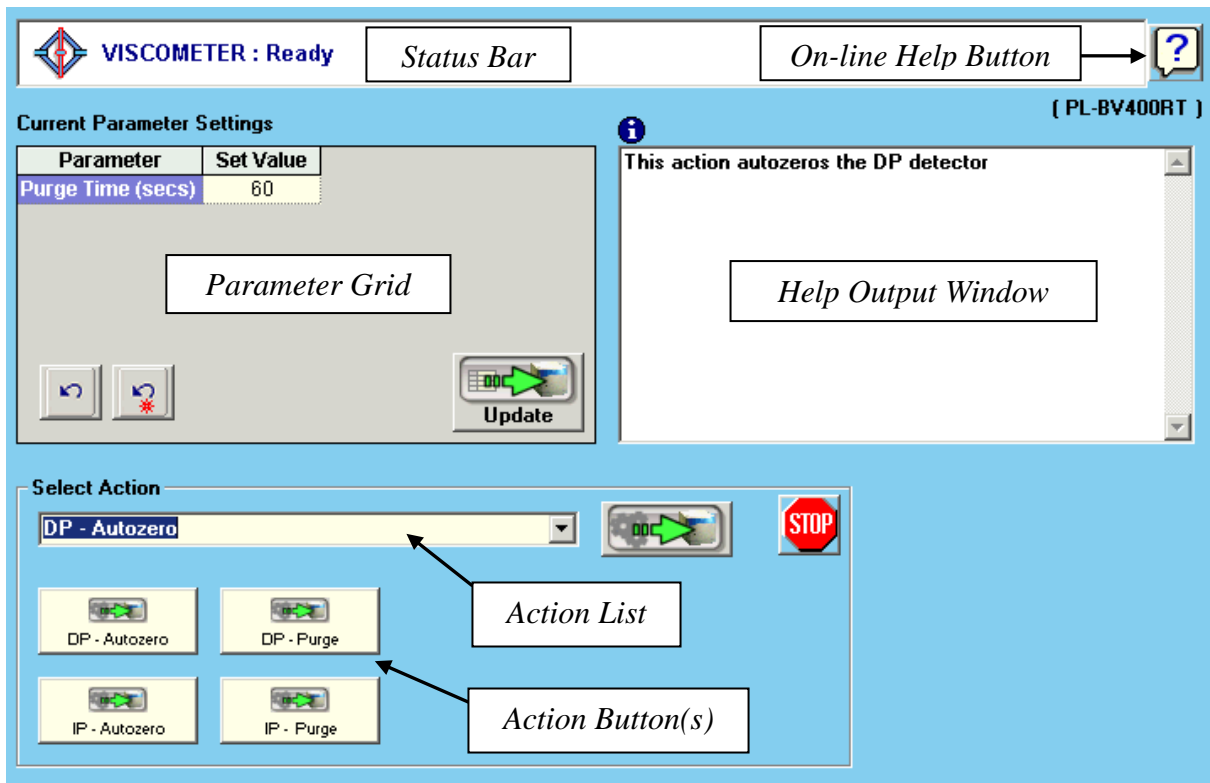
A green LED next to the viscometer component indicates the detector is being controlled and running whereas a red LED would indicate the detector is either not running or not controlled.

For further information on the *Summary View* please see the on-line help within the PL Instrument Control software.

3.3 Component View

The component view displays the component view of the selected component providing you access to parameters and/or direct commands for that specific component. In general each component view contains a number of common items and options. The actual items available in a view are dependent on the component selected.

The component view for the PL-BV 400 RT Viscometer is shown below.





Status Bar – This displays information about the viscometer component and it's current status.

On-line Help Button – This is a direct link to the on-line help for the viscometer component view.

Parameter Grid – This displays the current set purge time for both the DP and IP purges. To set and update the purge time enter the required value in the *Set Value*

column and press the Update button, .

To undo the new set value prior to pressing the Update button press the Undo button, .

To reset the purge time back to the default value press the Factory Reset button, .

Help Output Window – This displays a simple summary help for the purge time parameter and viscometer actions. To view this information, select the parameter or action. The information displayed in the *Help Window* will be a short description about the action or parameter, the Factory Default Value and the Minimum and Maximum Values for the purge time.


Action List – This is a drop down list of the actions that are available for the viscometer. To run an action, select the required one from the *Action List* and press



the Submit Action button.

Action Button(s) – The actions available for the viscometer are displayed as *Action Buttons*, as well as being found in the *Action List*. To run an action, press the required Action button.



Press the Stop button,  to stop all the procedures currently running on the viscometer.

For further information on the viscometer component view please see the on-line help within the PL Instrument Control software.

For information on the Toolbar menus please see the on-line help within the PL Instrument Control software.

4. Troubleshooting

4.1 Errors

Any error(s) that occur with communications or operations (running component actions, updating parameters etc.) with the viscometer, the *Diagnostic Output Window* will automatically open with the error(s) displayed in the *Component Error Status Tab* of the *Diagnostic Output Window* as shown below. Each error is uniquely identified with a number that can be referenced back to the control code.

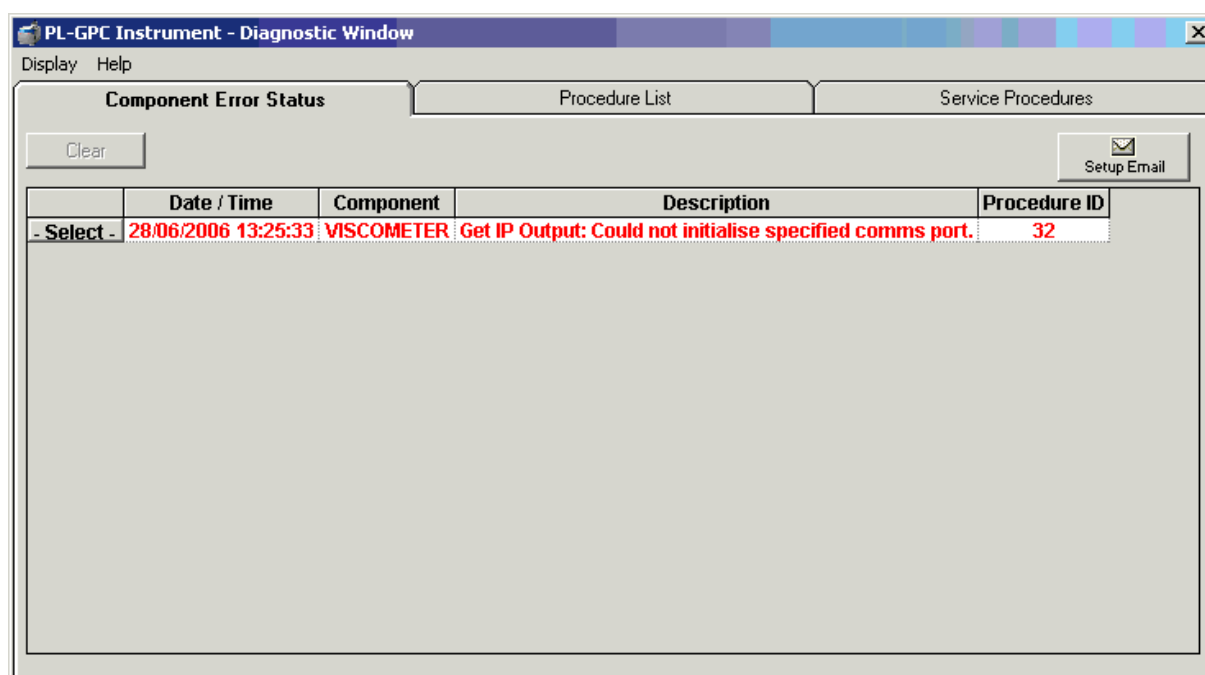


Figure 4.1.1 Diagnostic Output Window

Note: The information displayed in last two Tabs is primarily for service diagnostics and the Window does not need to be open for normal operation of the instrument.

If an error is displayed it needs to be cleared in order for the diagnostic window to be closed, allowing access back to the PL Instrument Control software. To clear an error press the Select button to highlight the row and press the Clear button. Multiple errors can be selected at a time. Once all errors have been cleared the diagnostic window can be closed.

Clearing an error will stop all procedures that are running and attempting to communicate with the viscometer. To re-establish communications with the viscometer either select the *Reconnect option* from the *Instrument menu* or return to the component view and resend the parameter or repeat the required action.

The errors that can be displayed from the software for the viscometer are listed on the following pages.

4.1.1 PL-BV 400 RT Viscometer Errors

Error	Cause(s)	Action
Could not initialize specified comms port	Incorrect Com port assigned to the component, the PL-GPC 50 Plus is not powered on or the USB cable is not connected.	Ensure the correct Com port has been assigned within the PL Instrument Configuration Editor program. Ensure the PL-GPC 50 Plus is powered on and the USB cable is connected.
No response received from the device	Incorrect Com port assigned to the component or communications lost with the component, e.g. power failure.	Ensure the correct Com port has been assigned within the PL Instrument Configuration Editor program. Ensure the PL-GPC 50 Plus and/or component is powered on. If assigned Com port is correct and the system is powered on then contact Varian or your local agent.
Unrecognized response from the device		Turn the PL-GPC 50 Plus off and then on again (ensure the control software has been closed before turning the instrument off). If this error persists then contact Varian or your local agent.
The device rejected the last command	Incorrect component assigned to the Com port.	Ensure the correct component has been assigned to the correct Com port within the PL Instrument Configuration Editor program.
Autozero operation could not completely cancel the transducer offset	The Viscometer failed to autozero the DP signal correctly.	Purge the DP Transducer and ensure it is flushed with solvent. If this error persists then contact Varian or your local agent.
The device returned an error	The component failed to complete an action.	Reinitialize the component from the PL Instrument Control software and ensure the initialization is completed successfully. Otherwise turn the PL-GPC 50 Plus off and then on again (ensure the control software has been closed before turning the instrument off). If unsuccessful and no obvious cause for the error then contact Varian or your local agent.

4.2 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 - Cleaning & Decontamination Procedures



**CLEANING AND DECONTAMINATION PROCEDURES
ARE ONLY TO BE CARRIED OUT BY TRAINED
SERVICE PERSONNEL**

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 deionised water. Do not attempt to clean any of the connectors on the rear panel; water may gain ingress and accessible pins may become bent. Ensure that no moisture enters the instrument. Allow instrument to dry off completely before reinstalling into the PL-GPC 50 Plus and 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 cover of the instrument to see if any solvent has contaminated the interior.

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 the 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 replacing the cover and reinstalling the PL-BV 400 RT into the PL-GPC 50 Plus.

Dispose of contaminated waste appropriately.

Appendix 2 – Parts List

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