

# **HTHS Series II**

# High Temperature High Shear Viscometer

Instruction & Operation Manual







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## INTRODUCTION

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### **Overview**

	The <b>CANNON</b> <sup>®</sup> High-Temper cometer is designed to determ oils under conditions of high s	rature High-Shear (HTHS) Series II Vis- ine the viscosity of engine oils and other hear at high temperatures.
	The HTHS Series II is capable specified in ASTM D 5481. For set as low as 25°C above amb	e of testing at a temperature of 150°C as or other applications, temperature may be ient.
	Oil samples are introduced int through the glass capillary tub shear rates. The HTHS Series Data can then be analyzed via be displayed and printed.	o the viscometric cells and then propelled es at the test temperature to obtain various II monitors the test and records the results. the computer software and test results can
Manual	This manual provides informed	tion about
	This manual provides morma	tion about.
	<ul> <li>Functions and operation of</li> <li>HTHS testing method</li> <li>Basic maintenance and re</li> </ul>	of the HTHS Series II pair
Components	For additional details regardin data analysis software package for Windows <sup>®</sup> provided with the HTHS Series II, consult the software manual.	g the High Shear Viscosity Calculator, a
Components	The HTHS Series II vis- cometer includes 5 visco- metric cells in a thermostated block with an internal temperature control system, a digital pressure measuring system, auto- matic timer, vacuum/ pressure controls and waste receiver.	
Patents	U.S. Patent Numbers: 4,658,636; 4,723,442	The <b>CANNON</b> <sup>®</sup> High-Temperature High- Shear (HTHS) Series II Viscometer

### **HTHS Specifications**

	HTHS SERIES II SPECIFICATIONS
Instrument Model	High Temperature High Shear (HTHS) Series II Viscometer
Dimensions	521 mm wide x 387 mm deep x 686 mm high (20.5 x 15.25 x 27 in)
Weight	40.5 kg (89 lbs)
Shipping Weight	75 kg (165 lbs)
Operating Conditions	15°C-30°C, 10%-90% RH non-condensing; Installation category II Pollution degree 2
	Fuse Replacement
F1 & F2 (line)	110/115V unit: M250V 5A; 1¼" x ¼" (P61.3312) 230V unit: M250V 2.5A; 1¼" x ¼" (P80.4034)
F3 (circuit board)	F250V 200 mA TR5 (P61.3292)
F4 (circuit board)	Cartridge fuse, F250V, 100 mA 5x20 (P61.3606)
F5 (heater)	M250V 2.25A; 1¼" x ¼" (P61.3339)
F6 (DEC valve) F7 (INC valve) F9 (vent fan)	M250V 300mA; 1¼" x ¼" (P61.3340)
F8 (air pump)	M250V 2.25A; 1¼" x ¼" (P80.4034)
Compliance	EMC directive (89/336/EEC); Low voltage directive (73/23/EEC) HI-POT (1900 VDC, 60 sec.)
Catalog #	Item Description
9728-C30	Model HTHS Series II, 100 volts, 50/60 Hz, 500 watts
9728-C35	Model HTHS Series II, 115 volts, 50/60 Hz, 500 watts
9728-C40	Model HTHS Series II-F, 230 volts, 50/60 Hz, 500 watts
9728-C60	Thermometer, 148.5 to 151.5°C. Subdivisions 0.05°C

NOTE

The **CANNON®** High Temperature High Shear Series II Viscometer meets the high shear testing requirements of ASTM D 5481. SAE J300 (DEC99) requires HTHS viscosities from 2.6 to 3.7 mPa•s. Because the HT75 standard has a nominal viscosity of 2.7 mPa•s and the HT150 standard has a nominal viscosity of of 3.7 mPa•s, **CANNON®** recommends that the users calculate cell volumes using either of these standards.

	Calibration Standards:
Catalogue #	Item Description:
9727-U50	HT39, 2.0 mPa•s (cP) at 150°C
9727-U55	HT75, 2.7 mPa•s (cP) at 150°C
9727-U60	HT150, 3.7 mPa•s (cP) at 150°C
9727-U65	HT240, 5.0 mPa•s (cP) at 150°C
9727-U70	HT390, 7.0 mPa•s (cP) at 150°C
9727-U92	HTNN-1, Non-Newtonian reference standard, 3.6 mPa•s (cP) at 150°C and 10 <sup>6</sup> sec <sup>-1</sup>
9727-U9 <b>4</b>	HTNN-2, Non-Newtonian reference standard, 3.1 mPa•s (cP) at 150°C and 10 <sup>6</sup> sec <sup>-1</sup>

### **Operator safety**

Please observe the following safety procedures and notices for proper operation of your HTHS Series II instrument. Deviation from the installation, operation or maintenance procedures described in this manual may result in a hazardous situation and may void the manufacturer's warranty.

- Make sure that your unit is operated only by qualified personnel
- Make sure that you read and understand all operating instructions and safety precautions listed in this manual before installing or operating your unit. If you have questions regarding instrument operation or documentation, contact **CANNON**<sup>®</sup> Instrument Company.
- Transport the unit with care. Sudden jolts or drops may cause damage to components.
- Observe all warning labels.
- Never remove warning labels.
- Never operate damaged or leaking equipment.
- Never operate the equipment with damaged mains power cables.
- The instrument power cord should only be connected to a suitable AC mains power source (with protective earth ground) matching the specifications of the S/N label on the instrument rear panel.
- Position power cords so that they are not likely to be walked on or pinched by items placed on or against them. Keep all connections as neat as possible.







WARNING

 $\sim$ MAINS

AC Power Input Symbol



- To disconnect the power cord, pull it out by the plug. Never pull the cord itself.
- Always turn off the unit and disconnect the mains cable from the power source before performing approved service or maintenance procedures, or before moving the unit.
- Refer all service and repairs to qualified personnel.
- Do not attempt to service the unit beyond the service and/or repair procedures detailed in this manual or other documentation provided by the manufacturer. Contact **CANNON**<sup>®</sup> Instrument Company for all additional service/repair needs.

In addition to the cautionary statements listed previously, additional cautions may be posted throughout this manual. These warnings may be designated by an appropriate symbol inside an equilateral triangle. General cautions are indicated with an exclamation point (see diagram, left). Read and follow these important instructions. Failure to observe these instructions can result in permanent damage to the unit, significant property damage, and personal injury.

Hot surface cautions (see diagram, left) may be attached on or near hot surfaces of the instrument. Avoid touching these surfaces during instrument operation above  $50^{\circ}$ C.

The Protective Conductor Terminal symbol is used to indicate required ground connections for your instrument electrical supply.

When supplying power to this instrument, connect the protective ground (earth) terminals of the instrument to the protective conductor of the (supplied) line (MAINS) power cord. The main plug for the power cord should only be inserted in a socket outlet (receptacle) provided with a protective ground (earth) contact. Do not use an extension cord (power cable) without a protective conductor (grounding).

The ~MAINS symbol indicates instructions or connections for the AC power supply. The AC Power input must match the electrical specifications listed on the label on the rear panel of the instrument. The supplied AC Mains power cord must be attached to the connector labelled ~MAINS. This connection serves as a means of disconnect and should be readily accessible.

The  $(\mathbf{O})$  symbol indicates the OFF position for the electrical switches for your unit (AC Mains or accessories).

Do not use this equipment in a manner not specified by the manufacturer. If you do, the protection provided by the equipment may be impaired, and you may void the manufacturer warranty.

### Apparatus description



The HTHS has been designed for testing of hot oil samples under high pressure. Care should be taken to perform sample tests per the procedures described in this manual.

The High-Temperature High-Shear Series II Viscometer consists of a heating device (an aluminum thermostatic block) with cavities housing five viscometric cells. The block permits temperature regulation of the

test sample from  $30^{\circ}$ C to  $150^{\circ}$ C,  $\pm 0.1^{\circ}$ C. Components and accessories include the waste-oil receiver, Vacuum Pressure Unit (built-in), digital timer mechanism, soak cycle timing watch, thermal sensors and safety devices.

The waste-oil receiver (see photo) consists of a recess underneath the block with apertures for the exit tubes



HTHS with waste oil receiver

from the five test cells. A plastic container is placed under the tubes to catch the oil sample as it is expelled from the viscometer cells. The container slides into place at the left front of the HTHS Series II. A hinged door provides protection from hot oil. At the rear, an internal fan draws vapors into the exhaust manifold.

Each viscometric cell contains a borosilicate-glass capillary sealed with Viton<sup>®</sup> gaskets at each end. A capillary support seats the capillary tube in the cell.

Matching of capillary lengths from one test cell to the next within 1 mm will allow the individual cell calibrations to be more closely matched, making



The HTHS cell capillary

subsequent runs of identical sample in different cells more repeatable. Matched capillary sets supplied with the instrument at initial purchase and are also available for purchase from **CANNON**<sup>®</sup>.

Nitrogen supply

Waste-oil receiver

Cell capillaries

NOTE

The user-supplied nitrogen gas cylinders should be fitted with a pressure regulator to reduce the pressure supplied to the HTHS Series II to no more than 500 psi. Testing pressures will range from 100 to 500 psi.



Nitrogen is the only gas recommended as a source of pressure for the HTHS Series II viscometer.

### Operation

Oil is introduced into the cells via the syringe-like charge cylinder(s) (see photo). The oil sample is filtered through a screen, then warmed to test temperature in the viscometric cell and finally propelled under high pressure from the cell through a narrow exit tube. The oil sample is expelled into a plastic container in the waste-oil receiver. A digital timer tracks the duration of the test run.

### Safety features:

### Protective housing

The HTHS components are protected from damage by a sturdy metal housing. The housing also prevents direct operator contact with the heater block assembly.

### **Pressure relief**

The HTHS ballast tank stores pressurized gas from the user nitrogen supply for the HTHS. If pressure in the tank exceeds 700 psi, the internal HTHS pressure relief rupture disc will vent gas to atmo-

Charge cylinder in Luer fitting

sphere until the gas source is turned off or depleted. <u>A failed rupture disc</u> <u>must be replaced prior to resuming HTHS operations</u>. See APPENDIX B for additional information on rupture disc replacement.



HTHS internal rear view including pressure relief rupture disc assembly

### Thermostat

The HTHS Series II is equipped with a thermostat that prevents the instrument from overheating. If temperature rises to an unacceptable level ( $160^{\circ}C \pm 5^{\circ}C$ ), power will be removed from the HTHS and will not be restored until the instrument has cooled to a safe state.



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The **CANNON®** High Temperature-High Shear (HTHS) Series II Viscometer is shipped with only minor assembly required. The five viscometric cells are shipped in place. Several accessories are included and must be installed prior to HTHS operation.

Follow the directions below to set up the HTHS:

### Unpacking/inspection

	1.	Remove all components from the shipping container(s).	
	2.	Remove any and all packing materials (styrofoam, etc.) from the components.	
	3.	Compare and verify shipped materials by comparing equipment items with packing/parts list(s). Report missing items to <b>CANNON</b> <sup>®</sup> Instrument Company immediately.	
	4.	Inspect each component for signs of damage. Report damages to the shipper and to the <b>CANNON</b> <sup>®</sup> Instrument Company immediately.	
Damaged items	Re tion Ins ori ins Un on	etain all packing materials until the instrument is connected and func- oning properly. If any component(s) must be returned to <b>CANNON</b> <sup>®</sup> strument Company, the damaged item(s) should be packaged in the iginal shipping container. Refer to the final chapter of this manual for structions on returning defective equipment. Customers outside the nited States should contact the local <b>CANNON</b> <sup>®</sup> dealer for procedures returning products to <b>CANNON</b> <sup>®</sup> .	

CAUTION

The HTHS is a precision instrument. Great care should be taken in handling the equipment.

### Installation procedure

- 1. Attach the included power cord to the connector marked MAINS on the lower rear panel of the HTHS. Do NOT plug the power cord into a power outlet at this time.
- 2. Place the 2-piece panel collar over the circular opening on top of the HTHS housing.
- 3. Place the cell cover over the viscometer cells and slide it into place inside the panel collar.

### **Connecting the Syringe Hose Adapter**

The Syringe Hose Adapter is an adapter plate with a hose connection
which permits introduction of both vacuum and air pressure to the
syringe-like charge cylinders. The adapter is designed to slide over the
upper aperture of the charge cylinder and provide an airtight seal when
the sponge pad on the underside of the adapter is pressed against the lip
of the charge cylinder.

The adapter is shipped with a length of clear 1/16 inch plastic tubing. Follow the procedure below to connect the adapter to the built-in Vacuum Pressure Unit of the HTHS.

- 1. Locate the adapter plate and the length of clear 1/16 inch plastic tubing.
- 2. Slide one end of the plastic tube over the fitting on the adapter plate.
- 3. Slide the other end of the plastic tube over the hose fitting on the quick-connect hardware.
- 4. Push the hardware into the connector atop the HTHS by pushing downward.

**NOTE** To release the hardware, press down on the knurled portion of the connector.



Syringe Hose Adapter installed

### Connecting the nitrogen supply

Connect the regulated nitrogen supply to the brass intake at the back of the HTHS unit, using the procedure below:

- 1. Locate the 10' length of 1/8 inch copper tubing and Swagelok<sup>®</sup> connectors provided.
- 2. Secure the tubing to the brass intake at the left rear of the HTHS unit using the Swagelok<sup>®</sup> connectors (see photo). To tighten, insert the tubing into the fitting, tighten finger-tight and then tighten an additional 3/4 turn with a wrench. The intake is located to the left of the MAINS power cord connector.
- 3. Connect the other end of the copper tubing to your nitrogen supply.



Swagelok™ connections



The nitrogen supply must be equipped with a pressure regulator preventing the release of gas at pressures above 500 psi.

### $\sim$ MAINS

- 1. Connect the power cord to an appropriate AC power source (see rear panel label for power input requirements).
- 2. Twist the front panel POWER switch to the right to provide power to the HTHS unit.
- The timer and pressure displays will light up.
- The exhaust fan will begin operating.
- The Watlow<sup>®</sup> temperature display will indicate ambient and target temperature.
- The instrument will begin heating the block to the target temperature.

### Verifying VPU operation

Check the operation of the built-in Vacuum Pressure Unit (VPU) as follows:

- 1. Turn the front panel PUMP switch ON and toggle the PRESSURE/VACUUM switch to PRESSURE. You should hear the pump operating.
- 2. Place your finger over the end of the black syringe hose adapter that is connected to the top of the unit by a clear plastic tube. You should feel the pressure of the air flow. The rate of flow is not adjustable.
- 3. Flip the PRESSURE/VACUUM switch to VACUUM and check the adapter for a slight suction with your finger. The VPU has been carefully adjusted at **CANNON**<sup>®</sup> but it may be necessary to readjust the force of the vacuum by turning the ADJUST dial on the front panel of the unit. The amount of force determines the relative speed at which oil is drawn into the charge cylinder. You may experiment with an oil sample to fine-tune the adjustment (see *Soaking the sample*, Chapter 5).

To increase the force of the vacuum, rotate the dial clockwise; to decrease the force of the vacuum, rotate the dial counterclockwise.



The flow of oil should be regulated in such a way that the oil is not drawn into the charge cylinder too quickly. If this happens, the oil may be drawn into the VPU via the syringe hose adapter, possibly contaminating future samples and damaging the unit.

4. Turn the pump OFF and press the HTHS power switch in to turn it off.

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## HIGH SHEAR VISCOSITY CALCULATOR

Software features	The High-Shear Viscosity Calculator is a standalone software package that calculates viscosity of a sample in mPa·s (cP) using efflux time and pressure values obtained during testing with the CANNON <sup>®</sup> High-Temperature High-Shear Viscometer (HTHS). The Calculator also provides a routine for ensuring the accuracy of constants used in viscosity calculation.
	Designed for use in conjunction with the HTHS, the Calculator incorporates a 15-minute temperature equilibrium timer for samples heating in the test cells. The Calculator can also recommend a Target Pressure, based on the user's best guess for sample viscosity, to achieve the desired shear rate with the HTHS test—this feature may considerably reduce the time required to achieve an acceptable shear rate with the HTHS instrument.
	A computer is used to run the software for the High-Temperature High-Shear Viscometer, but is not physically attached to the instrument. The HTHS software program is for data analysis only, and <i>NOT</i> for controlling or monitoring HTHS tests.
	For more information on using the High Shear Viscosity Calculator with your HTHS instrument, and for details on the calibration procedure, see the <i>High Shear Viscosity Calculator Instruction Manual</i> .
Computer	The table below provides minimum specifications for successful opera- tion of the High Shear Viscosity Calculator.
	Computer Specifications (minimum configuration)
	Intel® Pentium® or equivalent (166 MHz or better)
	MS Windows <sup>®</sup> 95/98/2000/NT <sup>®</sup> /XP <sup>®</sup>
	32 megabytes RAM memory
	Keyboard, monitor (color recommended) and mouse
	Windows <sup>®</sup> -compatible printer with cable

20 megabytes of hard disk space

### NOTE

### **Program Installation**

	Before installing the High-Shear Viscosity Calculator software, turn on your computer and make certain that the Windows <sup>®</sup> operating system is performing normally. Close all other software programs before beginning the installation procedure below:	
Checklist	1. Place your High-Shear Viscosity Calculator installation CD-ROM in your computer's CD-ROM drive (usually D:).If the installation program is not initiated automatically, click Start/Run and click the Browse button to select the SETUP program on the CD-ROM drive from the RUN window. Click Open. Then click OK.	
	2. Follow the installation program prompts to complete the installation.	
	The default path for installation is C:\Program Files\CANNON Instrument Company\HTHS.	
	The setup program will install the High-Shear Viscosity Calculator files to the drive and directory specified. The program will also create a shortcut icon on your Desktop.	
Initial software configuration		

### **Configuration disk**

Your High-Temperature High-Shear Viscometer is shipped with a Configuration floppy disk with initial software calibration information derived from HTHS factory tests.

Configuring software Prior to recording test data with the High-Shear Viscosity Calculator software, copy the configuration files from the floppy disk to the installation directory for the High-Shear Viscosity Calculator (the default directory is:

C:\Program Files\Cannon Instrument Company\HTHS).

Then start (or restart) the software to load the configuration data.

For more information on configuring your software to conform to HTHS characteristics, consult the *High Shear Viscosity Calculator Instruction Manual*.



### HTHS PRE-OPERATION CHECKLIST



The HTHS has been designed for testing of hot oil samples under high pressure. Care should be taken to perform sample tests per the procedures described in this manual.

To verify that the HTHS is functioning correctly, follow the checklist below:

Checking nitrogen supply

1. Turn the HTHS power switch clockwise to turn it on. Ensure that the regulated nitrogen supply is providing pressurized nitrogen gas to the HTHS via the rear panel connection (see photo). Listen for signs of gas leakage.



HTHS Series II nitrogen connection to internal valve system (panel removed to provide internal detail)

Checking solenoid valves

2. If there is no audible indication of gas leakage, depress and hold the front panel push button switch labeled INCREASE (▲). This will actuate the solenoid valve that admits nitrogen from the regulated nitrogen supply source to the ballast tank. As gas flows into the ballast tank, the reading on the front panel digital pressure indicator should steadily increase. Increase pressure to about 100 psi. To stop the flow of gas, release the INCREASE switch. If the pressure in the ballast tank rises above the desired level, push and hold the button solenoid switch labeled DECREASE (▼) to vent gas from the ballast tank to the atmosphere. Release the button when the desired pressure has been obtained.

NOTE	There will be a time delay after the switch is released and before the HTHS digital pressure display stabilizes. This is normal during filling and venting the ballast tank. You may need to practice working with the valve settings to establish the desired pressure in a relatively short time.	
	The internal valve system for the HTHS Series II includes a replaceable rupture disc as a safety measure to prevent pressure from rising above 700 psi. <u>HTHS pressure settings above 500 psi are not recommended.</u>	
	3. Ensure that all of the plug valves at the top of the viscometric cells are closed. When you are done, all valve handles should be in the <i>horizontal</i> position.	
	4. Select a test cell using the Cell Selection switch. Then press the Start/Stop button to release nitrogen gas into the selected cell. Listen for the hiss of nitrogen gas escaping from the exit tube of the viscometric cell. The digital display will indicate a drop in pressure, and the digital timer will start.	
	5. Press the Start/Stop button again to stop the flow of nitrogen.	
	6. Repeat steps 4 and 5, changing the <b>Cell Selection</b> switch to each of the other four cells to ensure that the valve system is working properly. Note that the digital timer will reset for each test.	
	7. Check the Watlow <sup>®</sup> temperature display. The heater for the aluminum thermostatic block is energized when the power switch is turned on. The display indicates current and target block temperatures. After the power has been on for a while, the temperature indicator should show that the temperature of the block is rising.	
	8. Use the ▼ and ▲ buttons on the Watlow <sup>®</sup> temperature meter to select the target temperature (normally 150°C). Allow the temperature to stabilize at that setting. The temperature can be set for any value up to 150°C. It is necessary for the HTHS to stabilize temperature at the desired temperature before proceeding with actual viscosity tests. This may require more than two hours.	
NOTE	For temperature meter calibration and other meter options unrelated to HTHS testing, consult the Watlow® meter Operation Manual.	
	9. Insert a glass charge cylinder onto each of the five Luer fittings on the top of the viscometric cells and rotate the cylinder clockwise to lock it in place.	
NOTE	The HTHS will not begin a test unless all plug valves are in the closed (horizontal) position).	



### STANDARD HTHS TEST PROCEDURE

The procedure below should be followed whenever taking measurements with the HTHS Viscometer. Before meaningful data can be obtained, however, each of the viscometric cells must be calibrated with the standards provided (see CALIBRATION chapter). Before calibrating the HTHS, complete several test measurements with a readily-available oil to become familiar with the operation of the unit. Frequency of recalibration is at the user's discretion. Always recalibrate if changing the test temperature (usually 150°C).

### **Preliminary checklist**

Follow the preliminary checklist below to prepare a sample run for the HTHS:

- 1. Open the flow valve on your pressurized nitrogen supply to the HTHS (an internal ballast tank provides a pressure reservoir for efficient HTHS operation).
- Make sure that the waste oil container is in its position at the base of the HTHS under the viscometer cell exit tubes. Cap the HTHS exit tubes to prevent oil leakage during the preliminary part of the test. Satisfactory stoppers are available from CANNON<sup>®</sup> (Catalog #9726-N98; package of 12).





Plug valve open with Syringe Hose Adapter installed

When removing and replacing stoppers, always use a protective glove rated for protection at 150°C to prevent burns.

- 3. Attach the syringe tube (charge cylinder), which will contain the oil sample, to the Luer fitting on the desired viscometer cell holder at the top of the HTHS.
- 4. Turn the **Cell Selection** switch on the HTHS front panel to the number of the desired test cell. Pour approximately 5 ml of sample into the charge cylinder. This quantity of oil sample will be used to flush any oil remaining from the previous HTHS sample run.
- 5. Open the plug valve for the desired viscometer cell by turning the green plug valve handle counterclockwise to a vertical position.
- 6. Place the Syringe Hose Adapter over the top aperture of the charge cylinder and apply pressure to seal the sponge pad against the charge cylinder opening (see photo).

8. After the oil has been forced into the cell, close the green plug valve by moving it to the horizontal position.

DO NOT PROCEED TO THE NEXT STEP OF THE RUN PROCEDURE IF THE PLUG VALVE FOR THE TEST CELL IS IN THE OPEN (HANDLE VERTICAL) POSITION. The HTHS Series II test cycle will not begin unless the plug valve is closed.

- 9. Remove the adapter from the charge cylinder.
- 10. Using the toggle switch, turn the Pressure/Vacuum pump OFF.
- 11. Using the DECREASE and INCREASE push buttons, adjust the ballast tank pressure to between 100 and 150 psi.
- 12. Using a protective glove, carefully remove the stopper from the HTHS exit tube for the desired cell. Then press the START/STOP button to force the oil through the capillary. The sample will be flushed through the cell. An audible hiss indicates that the oil has been discharged and gas is flowing through the capillary.
- 13. After the test has been completed, toggle the START/STOP button on and off for a few seconds to remove as much excess oil from the cell as possible.
- 14. Using protective gloves, replace the exit tube stopper. You are now ready to prepare the sample for HTHS testing.

### Soaking the test sample

NOTE

1. To integrate testing with the software analysis package, load the High-Shear Viscosity Calculator software and select the desired test cell by clicking the graphic representation or typing the cell number in the appropriate field. You can also type the Cell Number in the appropriate field.



- 2. Fill the charge cylinder with the test sample to approximately the 10 to 12 ml level. Verify that the **Cell Selection** switch is turned to the number of the desired test cell.
- 3. Open the plug valve by turning the green plug valve handle counterclockwise to the vertical position.

4.	Place the Syringe Hose Adapter over the top aperture of the charge cylinder and apply pressure to seal the sponge pad against the top of the charge cylinder (see photo).
5.	Ensure that the Pressure/Vacuum switch on the left side of the HTHS front panel is set to Pressure. Then turn the Pump switch ON to introduce sample into the viscometric cell.
6.	Close the plug valve.
7.	Let the HTHS oil sample soak in the heated viscometric cell for at least 15 minutes to achieve test temperature. You can use the Equilibrium Timer built into the High- Shear Viscosity Calculator. From the interface window, just click on the Equilib- rium Timer button to start and stop the timer. Enter initial test data (Cell Number and Sample ID Name) in the High-Shear Viscosity Calculator software program during this time.
8.	Check the cell temperature on the front panel display and verify with your temperature reference to ensure that the HTHS is controlling at the desired test temperature.
9.	(optional) Input the estimated sample viscosity in the field provided by the High-Shear Viscosity Calculator. The Calculator will display the optimum gas pressure for achieving the desired shear rate.
10.	Adjust the gas pressure to the level required for test (See <i>ASTM D</i> 5481 sections 9 and 11 for more information). To increase the gas

pressure, press and hold the **Increase** button. To decrease pressure, press and hold the **Decrease** button. Monitor the current gas pressure reading using the HTHS digital pressure display.

**NOTE** Set the pressure 1.5 to 2 lbs. higher than the desired optimum test pressure, since the pressure will fall slightly during testing.

11. Wait for the pressure reading to stabilize before proceeding to the next steps.

### Setting sample volume

Before the HTHS test can be run successfully, the volume of oil in the viscometer tube must be precisely regulated. This is accomplished by withdrawing excess sample from the cell immediately prior to the test run.

- 1. Before adjusting the volume of oil in the cell, recheck the pressure to ensure that it is still at the desired value. If necessary, adjust the pressure before proceeding to the next step.
- 2. Verify that the **Cell Selection** switch is turned to the number of the desired test cell. Then open the plug valve for the desired cell by turning the green plug valve handle to its vertical position.

	3. Toggle the Pressure/Vacuum switch to the Vacuum position and again place the adapter on top of the charge cylinder. Turn the Pump switch ON to withdraw excess sample from the viscometric cell into the charge cylinder. If necessary, the intensity of vacuum can be controlled by using the Adjust dial on the front panel. When nitrogen gas bubbles appear consistently in the charge cylinder, the sample has been adjusted to the proper volume.	
NOTES	At least 0.5 ml of excess oil sample should be drawn into the charge cylinder. If bubbles appear consistently before this volume has been attained, not enough oil was used when filling the cell. You will need to introduce additional sample and begin the soak procedure again.	
	Empirical testing has suggested that variability in the intensity of vacuum used to withdraw excess sample from the HTHS test cell prior to testing may alter the sample volume in the cell. For better determinability and/or repeatability, avoid adjusting the valve setting from test to test.	
	4. Immediately turn the pump switch OFF and turn the plug valve to the CLOSED (horizontal) position. Remove the adapter from the top of the charge cylinder.	
NOTE	The HTHS will not begin a test unless all plug valves are in the closed (horizontal) position).	
	5. Verify cell temperature by checking the front panel temperature display.	
	6. Recheck the gas pressure reading and make last-minute adjustments if necessary, using the INCREASE and DECREASE buttons.	
Testing sample(s)		
NOTE	The HTHS will not begin a test unless all plug valves are in the closed (horizontal) position).	
	1. Verify that the <b>Cell Selection</b> switch is set to the number of the desired test cell. Using a protective glove, carefully remove the stopper from the viscometer cell exit tube. Then close the hinged door and begin the test immediately by pressing the <b>Start/Stop</b> button.	
	Make certain to remove the cap from the exit tube before initiating the sample run. Make certain that the plastic waste container is in position under the exit tubes.	
	2. Precisely 10 seconds into the timed sample test, check the current pressure from the digital pressure display and write it down.	
	3. Wait for the test run to be completed. The end of the sample flow period will be indicated by an audible hiss as nitrogen gas escapes through the capillary tube. The internal timer gauge will trip, stopping the timer. The HTHS should automatically stop the flow of pressurized gas to the test cell.	

- 4. Write down the efflux time for the sample run, as indicated on the HTHS digital timer.
- 5. Use the High-Shear Viscosity Calculator to enter the values for Pressure and Efflux Time from the test run. Then click the Calculate Viscosity ... button. The Calculator will automatically determine the shear rate from the efflux time and stored cell volume data. Whenever an acceptable shear rate is obtained, the viscosity data will be displayed in tabular format in the Calculator display window.
- 6. If the shear rate is not within the user-determined minimum and maximum values for the cell (you can click Calibration/Cell Data to view parameters for each cell), the software will automatically calculate and display a new recommended value for pressure. Readjust the HTHS instrument to the new test pressure, and repeat the test until the desired shear rate is obtained.
- 7. Repeat the test procedure for each cell.

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## **DAILY MAINTENANCE**

Because the HTHS is performing high temperature runs with a variety of oils, it is possible for the capillary drain tubes to become clogged, particularly if high viscosity oil remains in the capillary for extended periods of time.

To prevent this occurrence, **CANNON**<sup>®</sup> recommends that all five cells be flushed at the conclusion of testing each day with approximately 5 ml of a low viscosity neutral oil (2 mPa·s at 150°C) or commercially-available Stoddard solvent or Naptha.

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### CALIBRATION



	Occasionally it may be necessary to recalculate the mathematical con- stants used by the High Shear Viscosity Calculator to calculate viscosity. A test protocol using CANNON-manufactured oil standards provides the data necessary to derive the constant values. We will refer to this protocol as a calibration. The procedure is described in detail in the <i>High Shear</i> <i>Viscosity Calculator for Windows® Instruction Manual</i> .
CANNON standards	CANNON Instrument Company produces a series of Newtonian viscos- ity standards designed specifically for calibration of the High Tempera- ture High Shear Viscometers such as the <b>CANNON</b> <sup>®</sup> High-Temperature High Shear Viscometer. They work equally well calibrating other instru- ments at this temperature. These standards cover the nominal range of 2.0 to 7.0 mPa•s at 150°C.
	Viscosities for all standards sold by <b>CANNON®</b> are based on the National Institute of Standards and Technology (NIST) value of 1.0016 mPa•s(centipoise) for water at 20°C (68°F). We have been granted ISO 9002 registration for the manufacture and certification of viscosity standards. Standards are available in 120, 470, and 940 mL bottles as well as 3.8 and 19 L containers. These sizes correspond to 4 ounce, pint, quart, gallon and 5-gallon containers.
	Each calibration oil should be run in each cell following the normal test procedure. For assistance in determination of appropriate test pressures for each cell, consult the <i>High Shear Viscosity Calculator for Windows</i> <sup>®</sup> <i>Instruction Manual.</i>
Capillary specifications	The Cell Calibration Record on page 35 of this manual contains capillary specifications necessary to complete the calibration. The Record should be updated by the user whenever a viscometer cell or capillary is replaced. Input current data from the Cell Calibration Record into the HTHS Calculator per instructions in the software manual.
NOTES	As per ASTM D 5481, samples are to be run with a shear rate of $1,400,000$ reciprocal seconds ( $1.40 \times 10^6$ sec <sup>-1</sup> ). Other shear rates are obtainable.
	Empirical testing has suggested that the volume of sample residue left on the cell walls following the HTHS test will vary as a function of sample viscosity. SAE J300 requires HTHS viscosities from 2.6 to 3.7 mPa•s. Because the HT75 standard has a nominal viscosity of 2.7 mPa•s and the HT150 standard has a nominal viscosity of of 3.7 mPa•s, <b>CANNON</b> <sup>®</sup> recommends that the users initially calculate cell volumes using either of these standards. It is not necessary to recalculate the volume unless the standards change or the viscometric cell(s) or capillaries are replaced.

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## **MAINTENANCE & REPAIR**

### **Flushing viscometer cells**

**CANNON**<sup>®</sup> recommends that all five cells be flushed at the conclusion of testing each day with approximately 5 ml of a low viscosity neutral oil (2 mPa·s at 150°C) or commercially-available Stoddard solvent. This is especially important if the HTHS viscometer is maintained at temperature for an extended period without running sample, as many automotive engine oils will carbonize if left in the cell for long periods at high temperatures.

### Clearing a blocked capillary tube

Erratic HTHS measurements may suggest a blocked or partially blocked capillary tube. If the capillary becomes blocked or partially blocked, it will usually need to be removed for cleaning. A solvent flush should be attempted first if the symptoms suggest partial blockage.

### Cleaning the capillary

If the flush fails to remedy the blockage, follow the checklist below to clean the capillary. You will need two wrenches, a small screwdriver and a narrow gauge wire (less than 0.15 mm) to perform portions of the operation. You will also need two replacement gaskets and a #400 mesh filter screen (available from **CAN-NON**<sup>®</sup>—see HTHS replacement parts list, Chapter 9):

Preliminary disassembly Prior to cleaning, follow the instructions in APPEN-DIX A for removal and disassembly of the viscometric cell.

After removing the capillary, remove and discard the gasket and filter screen from the capillary support. The screen and gasket (see photo) may remain in the cell even after the capillary has been removed. If so, you will need to use a small tool similar to a dental pick to remove these items from the interior of the viscometric cell.



- 1. Pass the narrow gauge wire through the capillary several times to break through any obstructions.
- 2. Clean the capillary with a suitable solvent, or chromic acid cleaning solution if necessary, to remove the contamination. Then repeatedly flush the capillary with distilled water to remove the cleaning agent.

Follow the instructions in APPENDIX A to reassemble and replace the viscometric cell.



## **SPARE PARTS LIST**

#### **Description**

#### Part Number

POWER SUPPLY +/-15/5V .3/.2/2A	25.4030
PUMP VACUUM 115V 50/60HZ	41.2057
MUFFLER/FILTER VACUUM PUMP	41.39
FILTER, 60-MICRON, B-2F-60	61.216
POWER SUPPLY 24VDC 1.1A UNI IN	61.3030
WASTE OIL RECEIVER PLASTIC	80.1111
VALVE NEEDLE	80.1190
TRANSDUCER, PRESSURE SENSOR	80.1231
SCREEN N400 MESH L\FILTER	80.1401
VALVE SHUT-OFF	80.1603
FITTING SYRINGE	80.1604
VISCOMETRIC CELL	80.1605
EXTENSION TUBE BOTTOM	80.1607
FILTER ADAPTOR BRASS	80.1608
SCREEN #180 MESH PRECAP	80.1609
FILL TUBE (7.0ml NOMINAL)	80.1615
GLASS CAPILLARY	80.1650
SYRINGE GLASS	80.1660
ADAPTOR, SYRINGE HOSE	80.1950
TUBING 1/16 CLR	80.1970
TIMER	80,1980
RS232 AT MODEM CABLE	80.2210
DOOR, LEXAN HTHSG2	80.4007
TOP COLLAR	80.4009
COVER, TOP	80.4010
VALVE, 3 WAY SOLENOID	80.4015
VALVE, 7 WAY SWITCHING	80.4016
PANEL, SIDE TRIM	80.4017
ADAPTOR, SYRINGE	80.4019
VALVE 4 WAY TOGGLE MTV-5	80.4020
ASSY, PROBE SENSOR HTHS2	80.4021
FITTING, QUICK DISCON 1/16	80.4022
FITTING, QUICK DISCON INSERT	80.4023
BUMPER BLACK -95 SHORE A	80.4024
PROBE ASSY, RTD HTHS	80.4026
PID CONTROLLER 24VDC	80.4029
RELAY, SOLID STATE 5A 60VDC	80.4031
HEATER, 175W .25D	80.4033
FUSE M250V X 2.5A 1/4 X 1 1/4	80.4034
VALVE MODIFIED FOR HOLDER	80.4039
VISCOMETRIC CELL-UNCALIBRATED	80.4041
SWITCH RSD 2100-1090-002	80.4042
MAGNET RSD 2100-4902-000	80.4043
RUPTURE DISC 700 PSI	80.4048
FITTING, MALE CONN B-200-1-4	80.4049
SWITCH, TOGGLE SPDT	80.4050
SWITCH, PUSH BUTTON	80.4051
SWITCH, MOMENTARY INC/DEC	80.4052
CCA, VALVE JUNCTION	80.4053
CCA, POWER CONVERTOR	80.4055
IC CMOS MICRO 8 BIT PROGRAMMED	80.4058

#### **Description**

FITTING, NIPPLE 1/8NPT X 3.0L	80 4059
FAN MUFFIN 4.5 IN	80 4062
LABEL START/STOP PHOTO NEG	80 4063
LABEL INC/DEC SW INSERT	80 4064
CCA TRIGGER HTHS2	80 4066
FITTING FI BOW BRASS 1/8NPT FF	80 4069
REDUCER 3/16TUBE X 1/8TUBE	80 4070
GASKET VITON FOR HTHS CAPILLARY	80 4073
GASKET HTHS VISCOCEL	80 4073
TRANSFORMER 600VA	80 4077
CCA CLOCK/COUNTER HTHS IL PRESSURE	80 4079
TRANSDUCER MODIFIED	80 4085
OPERATION MANUAL HTHS II	80.4000
MANUAL HIGH SHEAR VISC CALC	80 4995
TEST HIGH SHEAR VISCO CALC	80 4006
	00.4990



### WARRANTY/RETURN INFORMATION

### **Products limited warranty**

In addition to other manufacturers' warranties, **CANNON**<sup>®</sup> Instrument Company ("the Company") warrants all products (other than reagents and chemicals) delivered to and retained by their original purchasers to be free from defect in material and workmanship for one year from the date of the Company's invoice to the purchaser. For a period of one year from the date of such invoice, the Company will correct, either by repair or replacement at the Company's sole election, any defect in material or workmanship (not including defects due to misuse, abuse, abnormal conditions or operation, accident or acts of God, or to service or modification of the product without prior authorization of the Company) without charge for parts and labor. The determination of whether any product has been subject to misuse or abuse will be made solely by the Company.

The Company shall not be liable for any special, incidental, or consequential damages, or any damage to plant, personnel, equipment or products, directly or indirectly resulting from the use or misuse of any product sold by the Company except as set forth in and limited by the foregoing warranties. Representations and warranties made by any person, including dealers and representatives of the Company, which are inconsistent, in conflict with, or in excess of the terms of this warranty shall not be binding upon the Company unless placed in writing and approved by an officer of the Company.

### Reagent and chemical warranty

**CANNON**<sup>®</sup> Instrument Company ("the Company") warrants all reagents and chemicals sold by the Company and delivered to and retained by their original purchasers to conform to the weight, specifications and standards stated on the package. The Company will, at its sole option, either replace or refund the price (net of freight, handling charges and taxes), of any reagent or chemical sold by the Company which does not conform to such weight, specifications and standards upon the prompt return of the unused portion. Except for replacement or refund of the net price, the Company shall not be liable for any damages occurring as a consequence of the failure of any reagent or chemical sold by the Company to conform to the weight, specifications and standards stated on the package.

### Returning a product to **CANNON®**

Procedure	Before returning a <b>CANNON</b> <sup>®</sup> product for repair or service, make every attempt to identify the problem. If, after careful checking, the problem remains unidentified or unsolved, telephone <b>CANNON</b> <sup>®</sup> Instrument Company (or the local service agent) to consult with a product specialist. If the specialist cannot recommend a simple solution or repair, <b>CAN-NON</b> <sup>®</sup> will authorize the return of the product through the issuance of a Return Authorization number (RA).	
	<b>CANNON®</b> Telephone Number <b>CANNON®</b> Fax Number	814-353-8000 814-353-8007
	Products returned to <b>CANNON®</b> must be care to the following address:	efully packed. Ship prepaid
	CANNON Instrument Company ATTN: Return Authorization # 2139 High Tech Road State College, PA 16803 USA	
	Please include the following:	
Required information	<ul> <li>The Return Authorization number (RA).</li> <li>The name and telephone number of the person contact regarding the product.</li> <li>Shipping and billing instructions for the return location.</li> <li>A detailed explanation of the reason for the return of the reason for the return of the reason for the return of the reason for the rest of the reason for the return of the reason for the rest of the reason for the return of the reason for the rest of the rest of the reason for the rest of the rest of the rest of the reason for the rest of the rest of</li></ul>	on at your company to arn of the product to your return.
	If the product is not covered by warranty, the with an estimate of the repair costs and asked repairs are made. The customer will be requir for the cost of the repairs.	customer will be provided for approval before any ed to issue a purchase order
Hazardous materials	Stringent government regulations restrict the scontact <b>CANNON</b> <sup>®</sup> before returning a product contain mercury.	shipment of mercury. Please et that could possibly
Shipping notification	Products returned without prior notification (loss or without Cannon's authorization, will not be	by either telephone or fax), e accepted.
	The customer may be billed a testing fee if a p <b>CANNON</b> <sup>®</sup> and found to be working properly	product is returned to y.



## APPENDIX A—HTHS CAPILLARY REPLACEMENT

### **Replacing an HTHS capillary**

It may occasionally be necessary to replace a capillary for one or more of the HTHS test cells. The procedure below describes the steps necessary to accomplish this task.

### Parts/Tools required:

You will need wrenches (sizes 7/16", 9/16" and 1/2") and a small screwdriver to perform portions of this operation. You will also need two replacement gaskets (P80.4073) and a #400 mesh filter screen (P80.1401).



Do not attempt the capillary replacement procedure unless the instrument has been turned off for several hours to allow cells to cool to a temperature of 50°C or less.

### Removing the viscometric cell



Turn off or disconnect the main gas supply to the instrument before continuing with this procedure!

- 1. Make sure that the HTHS nitrogen gas pressure is near 0 and that the gas line connections to the HTHS are closed. If the cells are pressurized, press the DEC button on the HTHS front panel to vent pressurized gas to the atmosphere. If the instrument has been in operation, turn it off and allow several hours for the test cells to cool to 50°C or less. Unplug the power cord.
- 2. Remove the glass charge cylinder(s) from the top of the cell.
- 3. Remove the cell cover. Then lift and remove the two-piece collar on top of the HTHS.
- 4. Remove the 12 screws securing the top panel to the HTHS frame. Remove the top panel and set it aside.

**CAUTION** Do not touch internal HTHS components unless specifically instructed to do so in these instructions.

5. Using a 7/16" wrench, disconnect the Swagelok<sup>™</sup> fitting to the copper gas line connection for the cell requiring capillary replacement (see photo, next page).



Disconnecting cell gas line connection

- 6. Trace the electrical wiring from the cell to the valve junction connector and detach the connector from the corresponding numbered junction. Carefully lift the cell(s) out of the thermostating chamber.
- 7. Orient the cell horizontally and hold the cell with a wrench, using the flats on the brass fitting at the base of the cell.
- 8. Use a second wrench to loosen the hex nut attached to the exit tube at the base of the cell and remove this fitting by turning the hex nut counterclockwise.
- 9. Remove the exit tube assembly from the cell.



Do not attempt to remove the fill tube and plug valve assembly from the opposite end of the viscometric cell. Their removal may cause a change in volume when reassembled.

### Removing the capillary

- 10. Maintaining the horizontal position of the cell, remove the gasket\* from the opening at the base of the cell, being careful that the glass capillary does not fall out of the cell. Discard the used gasket.
- 11. Carefully orient the cell in a vertical position over a soft cloth or paper and allow the glass capillary to slide out of the tube onto the cloth/paper. If the capillary does not drop out readily, tap the base of the cell gently to jar the capillary loose. If the capillary cannot be dislodged by this procedure, try heating the base of the cell with a laboratory hot plate or heat gun.



Disassembling the cell



12. After removing the capillary, remove and discard the gasket and filter screen (see photo). The gasket and screen may remain in the test cell even after the capillary has been removed. If so, you will need to use a small screwdriver or other tool similar to a dental pick to remove these items from the interior of the viscometric cell.

### **Reassembling the cell**

Follow the procedure below to reassemble the viscometric cell:



Reassembly sequence (left to right)

- 13. Orient the viscometric cell vertically with the plug valve assembly down.
- 14. Place a new filter screen in the cell and gently tamp into place with the tip of the exit tube assembly. Be careful not to deform the screen.
- 15. Place a new gasket in the cell and make sure it is seated against the screen.
- 16. Insert the capillary into the cell. The capillary is bilaterally symmetrical; either end may be inserted first.
- 17. Replace the gasket\* at the bottom end of the capillary and replace the exit tube fitting. Rotate the fitting until finger tight and then turn approximately one full turn with a wrench to seal the viscometric cell.
- 18. Carefully orient the assembled cell with the exit tube pointing down; replace the assembly in the HTHS thermostating chamber.
- 19. Reconnect the Swagelok<sup>TM</sup> fitting to the copper gas line connection for the cell. Tighten the fitting by hand; then secure with a 1/8" turn of a wrench.
- 20. Replace the two-piece collar and cell cover.
- 21. Replace the glass charge cylinder(s) at the top of the cell and plug in the power cord.
- 22. Reconnect or turn on the main gas supply to the instrument.
- 23. Turn on the HTHS and increase the gas pressure to approximately 50-100 psi.
- 24. Set the selector switch for the correct cell and press the START/ STOP switch.

- 25. Verify that gas can be heard discharging from the exit tube (a hissing sound should be clearly audible).
- 26. When you have verified that the new capillary is free of blockage, press the START/STOP switch again to stop the flow of gas. You are now ready to calibrate the cell and resume normal HTHS operations.

### High-Temperature High-Shear Rheometer Cell Calibration Record

Instrument Serial #			
HTHS Te	echnician:		Date:
Cell # (1-5)	Cell volume	Capillary diameter	Capillary length
	(ml)	(mm)	(mm)
1			
2			
3			
4			
5			

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## APPENDIX B—HTHS RUPTURE DISC REPLACEMENT

### Replacing an HTHS rupture disc

WARNING	There is no guarantee of rupture disc life. Disc life span is affected by corrosion, creep and fatigue, and physical damage. These conditions will derate the rupture disc to a lower set pressure. The user should be prepared to handle a premature failure of the rupture disc. Do not allow any buildup or solidification of media to occur on a rupture disc. This may increase the pressure setting of the rupture disc.
	The ½" STANDARD rupture disc is a precision instrument and must be handled with extreme care. Rupture discs should be installed only by qualified personnel familiar with rupture discs and proper piping practices.
	Do not install rupture disc if there is any damage in the dome area. A damaged rupture disc is any rupture disc with visible nicks or dents in the dome.
	The rupture disc manufacturer does not recommend reinstalling a rupture disc that has been removed from the holder. Always reinstall the entire rupture disc assembly, as reinstallation of the disc alone may adversely affect the joint sealing capabilities and/or performance of the rupture disc.
NOTE	The angular seat rupture disc design is rated for up to 700+ psig.
Installation	
	The rupture disc for the HTHS Series II instrument is sold and installed as an assembly that includes the rupture disc itself, vacuum support and holddown ring, conical threaded outlet and inlet connector. The rupture disc assembly is connected inline with the HTHS pressure system via a threaded connection on the inlet hex (see images, next page).
	The SCREW TYPE rupture disc assembly is permanently stamped with a maximum burst pressure rating. Ensure that the unit being installed is compatible with current operating conditions. Install the unit "upside down" with the outlet hex toward the floor of the compartment. In the event of a rupture disc failure, this will direct the gas pressure away from more sensitive internal components.
CANNON® High-Temperature High-Shear Viscometer Series II Instruction & Operation Manual Revision 1 Od—June 2011: CANNON® Instrument Company	

- 1. Before placing the assembly into the system, turn off the gas supply. Press the DECREASE button until the gas pressure is close to 0.
- 2. Remove power from the instrument.
- 3. Remove the 12 screws securing the top panel to the HTHS frame. Remove the top panel and set it aside.



Rupture disc installed inline

4. Clean the mating threads of all grit, dirt, or other foreign material to ensure a proper seal.

FLOW

OUTLET

HEX

INI FT

HEX

- 5. Make certain that the flow arrow stamped on the inlet body points in the proper system flow direction.
- 6. Apply PTFE tape or other appropriate sealer to the threads.
- 7. Place wrench on the thicker INLET HEX (see figure) and tighten with a torque wrench to 40 Ft·Lbs (54 N·M).



Do NOT use the narrower OUTLET HEX for tightening the assembly into the system, as you may damage or unseat the rupture disc.

### Preventive maintenance

Replace the rupture disc every year under normal conditions. More frequent changes may be necessary due to corrosion, fatigue, temperature or other adverse conditions. These factors should be evaluated through actual service experience.

To avoid operation downtime, a spare rupture disc should remain in stock at all times for each holder in use. The number of spares required will be determined by actual service conditions.



If the rupture disc is not replaced periodically when exposed to adverse conditions, premature failure of the rupture disc may occur, thereby discharging process media.

Once the integrity of the rupture disc has been compromised, gas will leak from the line until the assembly is replaced.





### APPENDIX C-FIGURES & DIAGRAMS

### **Plug Valve construction**



### Viscometric cell and exit capillary (cutaway view)



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### **CANNON INSTRUMENT COMPANY®**

2139 High Tech Road | State College, PA 16803 | USA 800-676-6232 | 814-343-8000 | Fax: 814-353-8007 sales@cannoninstrument.com | www.cannoninstrument.com