CANNON INSTRUMENT COMPANY

CANNON® Ravenfield® BS/C+ HTHS

Autosampler Manual

High Temperature High Shear Rate Viscometer

ASTM D4741, IP 370, CEC L36 A90

80.5993

Ravenfield® BS/C+ HTHS Autosampler Manual

P/N 80.5993

CANNON Instrument Company
2139 High Tech Road
State College, PA 16803

Copyright

Trademarks

Contact

Address: CANNON Instrument Company
2139 High Tech Road
State College PA 16803, USA

Phone: 814-353-8000; 1-800-676-6232

Fax: 814-353-8007

Website: www.cannoninstrument.com

Email:
Sales: sales@cannoninstrument.com
Service: service@cannoninstrument.com

Contents

Contents **1**

Autosampler with Windows **1**

Simple Workflow: Calibration 1

Simple Workflow: Routine Measurements 2

Installation **3**

User Interface **6**

Configuration of Options 6

Procedure 6

Calibration **8**

Selecting Calibration Oils 9

Temperatures of Test (Test Temp) 9

Sample Preparation 10

Cursor Hover 12

Normal Sample Operation **13**

Procedure 13

Test Status 13

Sample Failure 14

Review Samples **15**

Review Check Oil **16**

Halt on Bad Oil Check 16

Appendix 1 ..... **17**

Configuration Options 17

Parameters 17

Timings 17

Check Oil Parameters 18

Viscosity and Temperature 19

Bath Properties 19

Appendix 2 ..... **20**

Setting Up the Automatic Compensation System 20
Procedure for Setting Up Automatic Compensation System 20

List of Figures

Figure 1: Autosampler Connections 3
Figure 2: Diagram of Rear of Autosampler 4
Figure 3: Vacuum Pump Connections 4
Figure 4: Vacuum pump connections 5
Figure 5: Diagram of Autosampler 5
Figure 6: Samples and Calibrate Tabs 6
Figure 7: Cal Oil Table Screen 7
Figure 8: Calibrate Table 8
Figure 9: Set Test Temperature Screen 9
Figure 10: Performed Gap Setting Screen 9
Figure 11: Select Calibration oils 10
Figure 12: Sample Preparation Screen 10
Figure 13: Autosampler is Ready to Run Samples 11
Figure 14: Calibration data 11
Figure 15: Selected Points 12
Figure 16: Test Screen 13
Figure 17: Test Status Tab 14
Figure 18: Review Sample Data Tab 15
Figure 19: Typical Display 16
Figure 20: Configuration Screen 17
Figure 21: Timing Screen and Sample Repeat Pump Time 18
Figure 22: Check Oil Parameters 18
Figure 23: Check Oil Batch Screen 18
Figure 24: Bath Properties Screen 19
Figure 25: Expect the Scale to be -ve and the offset +ve 22

Autosampler with Windows

The Ravenfield model DR-IV autosampler is designed to provide a simple automatic loading mechanism to the BS/C+ HTHS viscometers.

The unit consists of a carousel, sampling arm and sample pump with an integrated PC running Microsoft Windows 10 and a provided touch screen. A small ticket printer is included to produce test reports. Also provided is a Standard RJ45 100 Base-T connection to laboratory networks.

The software incorporates a powerful database to store test results, allowing users to follow the results of any named samples over time. Graphs of sample viscosity and check oil can be routinely produced on the integrated printer, or any networked printer.

The version 2 program incorporates the ability to automatically process the whole suite of calibration oils, and to automatically trim the bath temperature, removing the need for the operator to intervene during test.

After switching on, the PC will automatically start the DR operating system, if it does not; click the "DR" shortcut on the main screen.

Simple Workflow: Calibration

Note: The BS/C+ has to have a previous manual calibration stored internally.

1. Switch on the PC. If the autosampler DR operating system does not start up automatically, click the DR shortcut on the main screen.
2. Without erasing the existing calibration, set the rotor/stator gap manually.
3. Exit calibration, and enter measurement mode. Press C. Continue to test to restart motor.
4. Connect the autosampler feedpipe to the side arm.
5. Place viscosity standard samples in vials on the carousel.
6. Identify standard oils samples on carousel in autosampler software. Make sure the samples are loaded in the correct positions – see sample test tab.
7. Verify the temperature is stable.

Note: The temperature is slightly lower than the test temperature in order to account for shear heating.

8. Press **Start**.
9. Wait until all standard oils have been tested. Review data on screen and check for outliers or other issues.
10. Select points on the displayed graph to be used for calibration.
11. Click **Calculate Constants**.
12. Check returned value of check oil for any issues.
13. If calibration is acceptable, save calibration to internal computer by clicking **Save Calibration**.

Simple Workflow: Routine Measurements

Note: The BS/C+ has to have a previous manual calibration stored internally, but will automatically retrieve the last calibration stored internally.

1. Calibrate as described in Simple Workflow: Calibration.
2. Click on **Measurement Mode** on the screen.
3. Connect the autosampler feedpipe to the side arm. Refer to Figure 1 (next page).
4. Pour samples into vials and place on the carousel. For a full carousel, ensure that at least three samples are check oils.
5. Enter the **Sample IDs** for oils into the software.
6. Press **Start**.
7. Wait until all samples have been tested.
8. Review data on screen.
9. Check performance of check oil over run.
10. Print report.

Installation

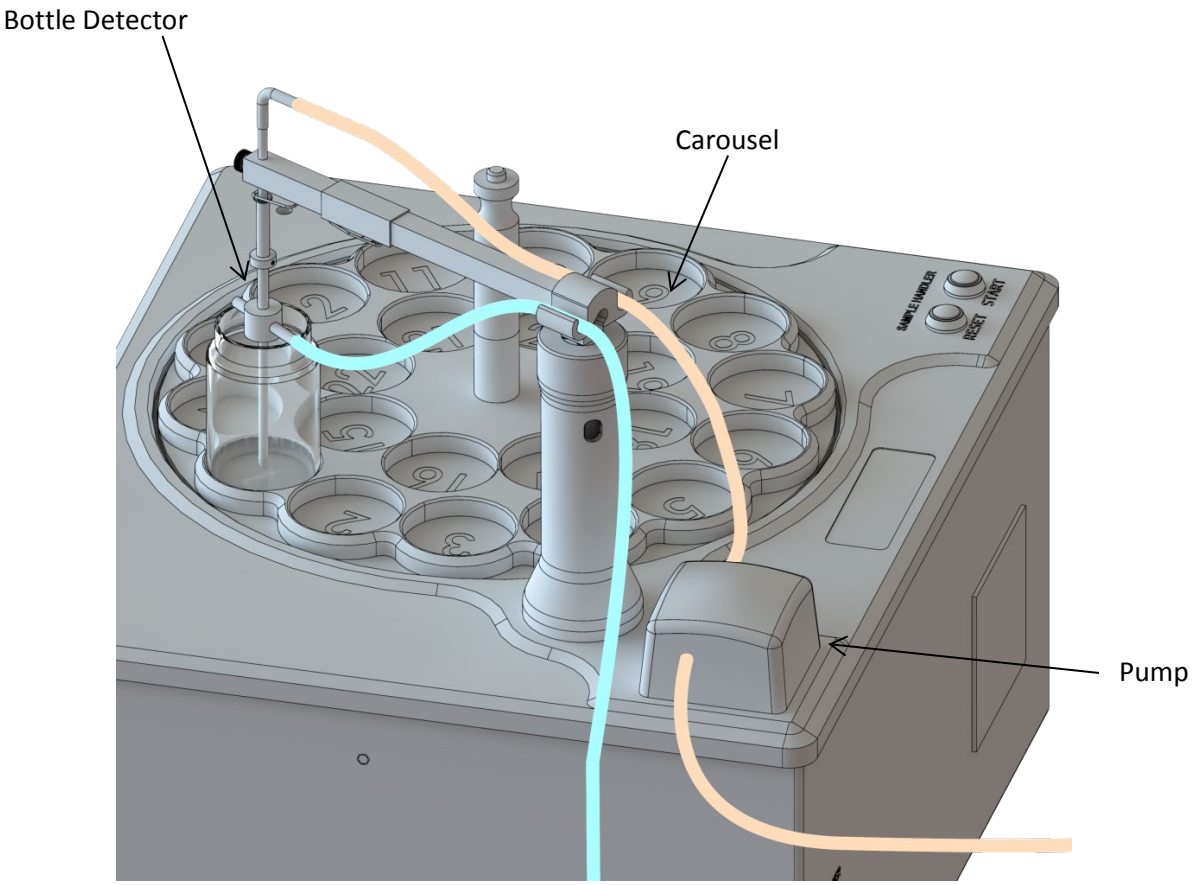

Figure 1: Autosampler Connections

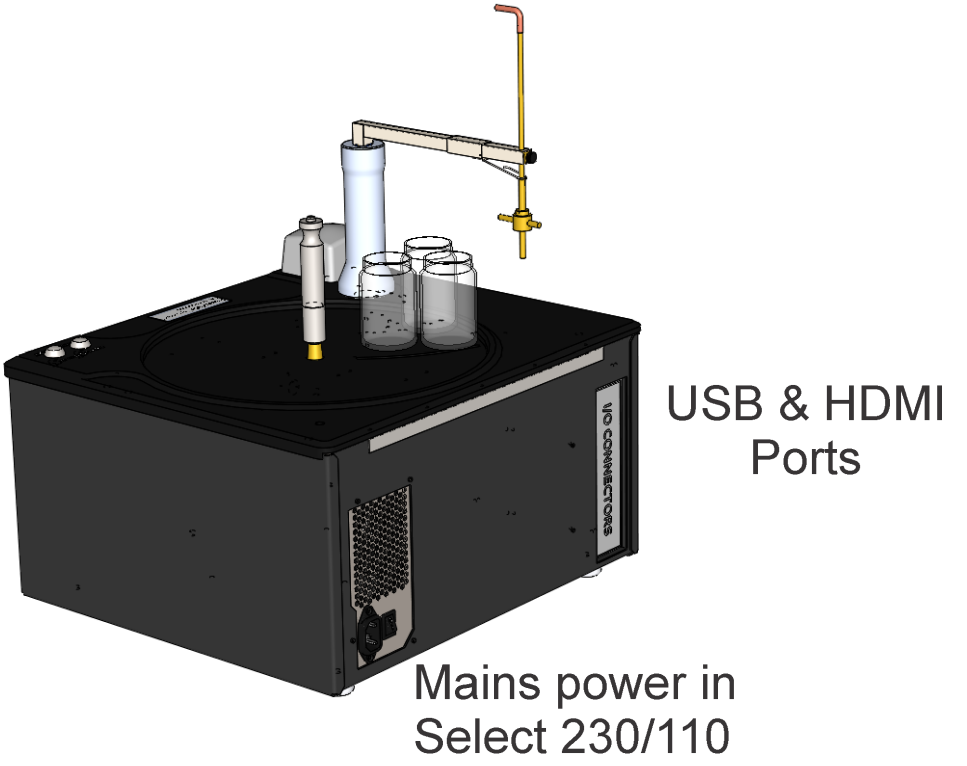

Figure 2: Diagram of Rear of Autosampler

The main parts of the instrument are identified in Figure 2. Plumbing of components is required for the initial installation. This is explained further in following sections.

1. A tapping must be made in the vacuum line to the instrument constant level pipe. Refer to Figure 3.

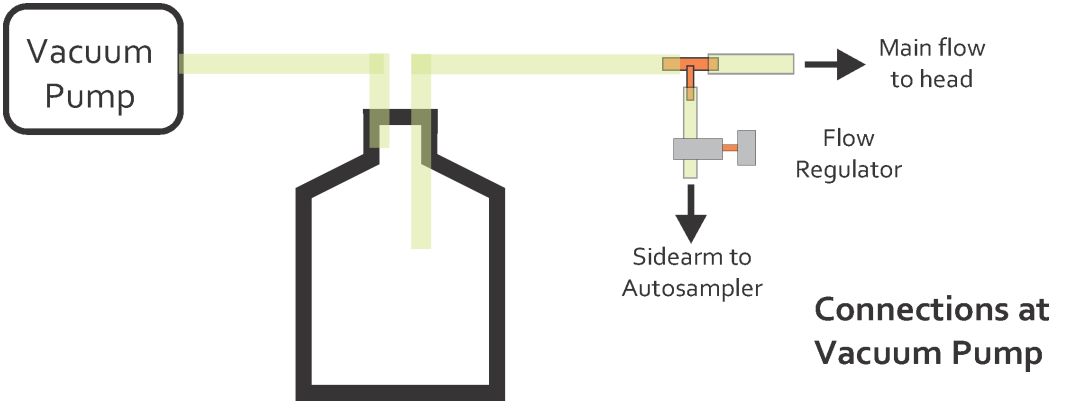

Figure 3: Vacuum Pump Connections

The flow regulator restricts the flow of air from the autosampler to ensure that there is adequate flow at the main flow to the head. This is a balance that has to be achieved during setup.

2. The main flow to the head is applied to the constant level vacuum pipe. Refer to Figure 4.

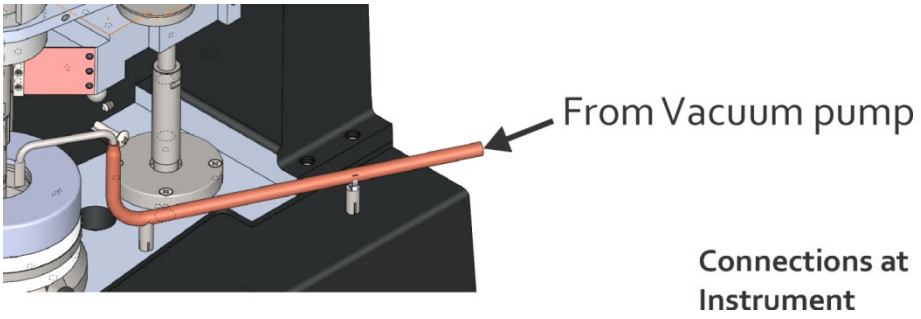

Figure 4: Vacuum pump connections

3. Attach the pipe from the sampling needle and the vacuum pipe attached to the "T" piece to the autosampler, in accordance with the diagram in Figure 5.

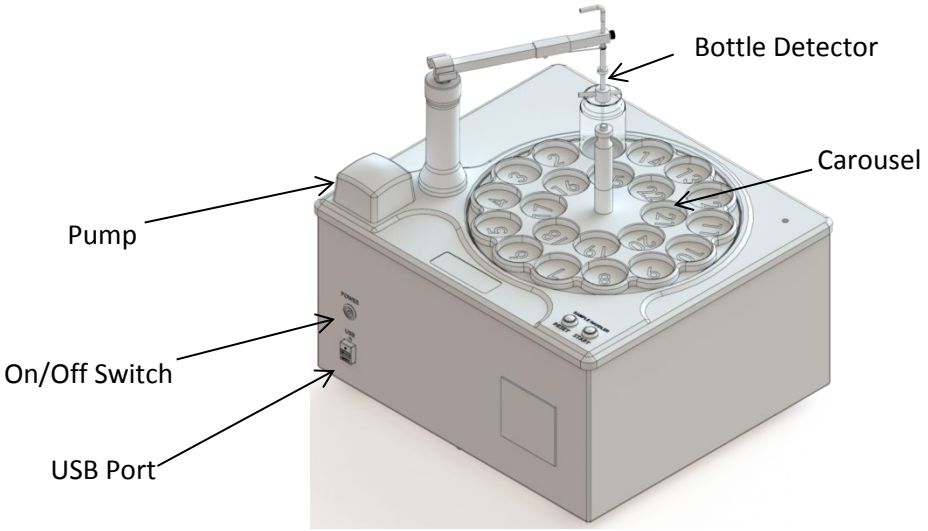

Figure 5: Diagram of Autosampler

The autosampler uses a peristaltic type pump. The white cover of the pump is lifted and the rollers of the pump withdraw to allow the sample pipe to pass in. The pipe must pass right to left through the pump to ensure the sample is pulled from the sample containers.

USB connections are made to the bath and instrument. They must be kept in the same USB connectors of the internal computer.

User Interface

The interface consists of the **Main Menu** bar at the top of the screen together with a series of tabbed sheets. Each tab is identified by a name at the top, for instance **Test**, **Test Status**, **Review Check Oil** and more. Click on the tab to see the contents of the sheet.

The autosampler defaults to showing the **Tests** tab at start up. In the **Samples** tab, the new **Calibrate** tab is visible on the left as shown in Figure 6.

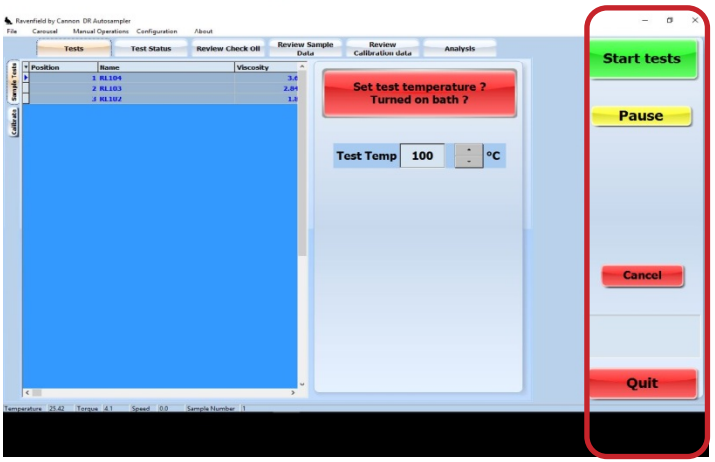

Figure 6: Samples and Calibrate Tabs

The main buttons on the right of the screen control the autosampler in bulk: starting tests, pausing and resuming tests, cancelling and quitting program operation.

Configuration of Options

Extensive facilities for controlling all the testing parameters are built into the operating program software. These are all noted in detail in Figure 7. The factory defaults are sufficient for most operations. The only routine access to configuration that is required is to amend the Calibration Oil Table (**Cal Oil Table**) is when new calibration oils samples are ran.

Procedure

1. Select **Configuration** from the Main Menu.

2. Select the tab **Cal Oil Table**. Refer to Figure 7.

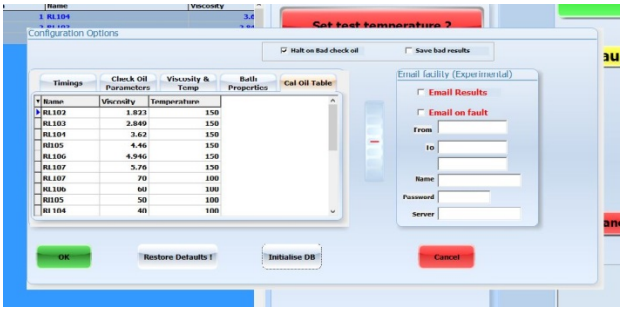

Figure 7: Cal Oil Table Screen

3. Click on the name column to change the name of a calibration oil. This is the same procedure for changing the value of the viscosity and temperature options.

Calibration

The new autosampler calibration function removes some of the routine work regarding calibration of the HTHS instrument by eliminating the need to cycle seven calibration oils through the instrument.

1. Select the **Calibration** screen from the Main Menu.
2. Select the **Preparation** tab at the top left of the **Calibrate** tab. Refer to Figure 8 and the red square.

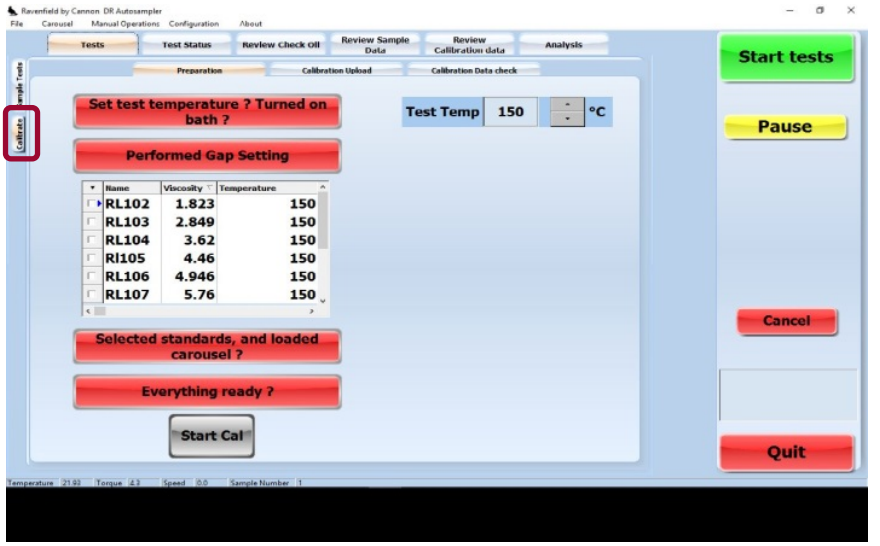

Figure 8: Calibrate Table

3. At this stage all tabs are red. The operator must satisfy each item when ready to proceed with calibration.
4. The first question, **Is the bath on?** Clicking the **Set Test Temperature? Turned on Bath?** will turn on the bath to the temperature specified in the **Test Temperature** box on the right of the tab. Refer to Figure 9.

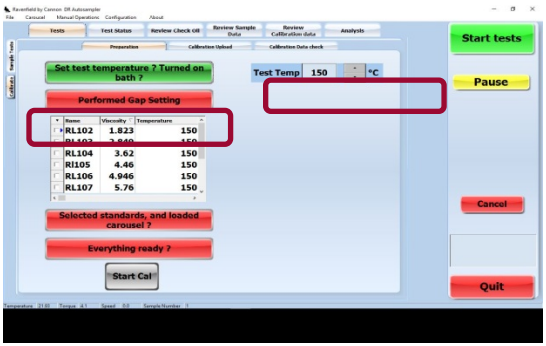

Figure 9: Set Test Temperature Screen

5. This temperature is the stated temperature under the **Test Temperature** on the **Configuration** tab. See *Configuration Options in Appendix 1* for more information.

6. Confirm there is an existing set gap between the rotor and stator in the instrument. This has to be set manually, as an intermediate step, when the instrument is being calibrated in the manual manner. Refer to Figure 10.

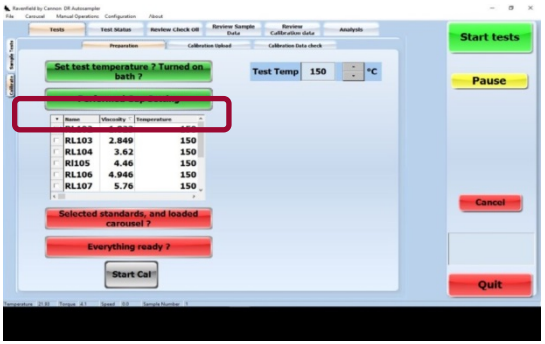

Figure 10: Performed Gap Setting Screen

Selecting Calibration Oils

Calibration oils must be previously setup in the **Configuration** option, tab **Cal Oil Table** (see *Configuration Options in Appendix 1*). The operator must select at least three calibration oils; these oils are then prepared and loaded onto the carousel in the correct order.

In Figure 11, the first three oils have been selected: RL102, RL103 and RL104. It is not possible to revise any stored values from this screen.

Temperatures of Test (Test Temp)

This is NOT selectable from this screen, only from the **Configuration** options. Refer to Figure 11.

Figure 11: Select Calibration oils

Sample Preparation

Now load calibration oil samples onto the carousel that is ready for testing.

The bottom of each cell is clearly marked with its position on the carousel. It is vital that each sample is placed correctly - sample 1 goes in the cell for sample 1, sample 2 in the cell for sample 2, etc.

Once the carousel is loaded, press the **Reset** button on the top of the autosampler, then press the **Start** button. The carousel turns automatically until the system locates sample 1, and then stops. The autosampler is ready to start; however, there are some final questions for the operator.

Figure 12: Sample Preparation Screen

1. When the carousel is loaded the **Start Cal** button becomes active. Clicking on it turns it green. Refer to Figure 13.

Figure 13: Autosampler is Ready to Run Samples

2. Click **Start Cal** to set the autosampler into calibration mode, then press **Start Tests**.

The autosampler will begin to test sample 1. The arm will swing in to sample 1 and descend into the sample vial. The pump then propels the first flush of sample 1 into the Ravenfield HTHS test cell.

After the carousel of oils is tested and the results provided, the operator must examine the calibration data to ensure a satisfactory result has been reached.

3. The **Calibration Data Check** tab of the calibrate option tab must be selected. Refer to Figure 14.

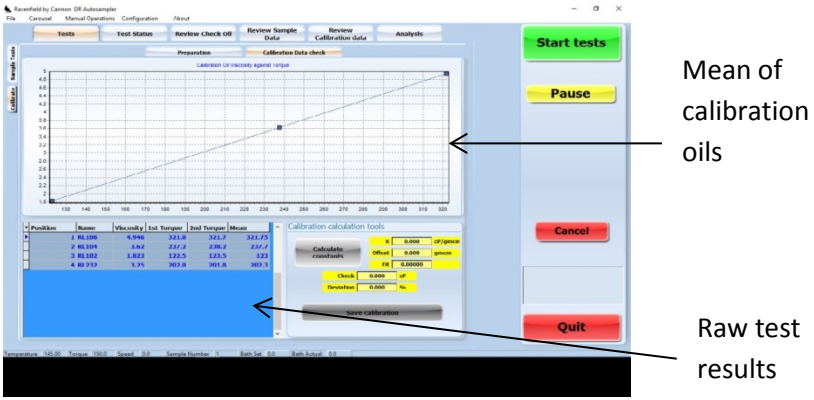

Figure 14: Calibration data

The **Calibrate Data Check** screen shows the results of testing the carousel of calibration oils. At the bottom left of the screen is a table showing raw test result. At the top is a graph displaying the viscosity of the calibration oils against the average of the two accepted test torque results, or the "mean" as shown in Figure 14.

4. Closely examine the line, and select all the tested data points. There must be more than three points. More than three points must be selected.

5. Selected points will become yellow. When the points are selected, click the **Calculate Constants** button to view the results of a linear regression and least squares fit through the data. Observe the quality of the fit. Refer to Figure 15.

Figure 15: Selected Points

Note: The calibration is not accepted if it is greater than 0.9995.

6. The final oil on the carousel is a check oil. Its value must be closely monitored and compared to the nominal value on the label of the oil container. The limits of the validity of the result are also marked there.

The yellow box marked **Check** contains the value of the check oil determined from the new calibration. Beneath it the box **Deviation** shows the difference between the determined value and the stored value, kept in the **Configuration** option under the **Check Oil Parameters** tab.

If the check oil doesn't agree by less than the amount specified in **Check Oil Tolerance**, the calibration is rejected.

Cursor Hover

If the data correlation is poor, it is possible to check the results and find the errant point.

1. Click the cursor in the **Mean** column of the table at the bottom left of the screen. A small box opens showing the back calculated value of the calibration oil using the calibration constants.
2. A large difference between the value that appears and the value under **Viscosity** is an errant data point, meaning the test should be repeated.

Normal Sample Operation

Procedure

Normal sample operation is nearly identical to calibration operations.

1. Set the instrument to **Temperature**.
2. Lower the head to the operating position.
3. Flush the cell.
4. From the Main Menu, select **Carousel** then **Clear ALL Data**.
5. Enter the new sample IDs of the sample oils.
6. Press the green **Start Tests** button and begin testing. Refer to Figure 16.

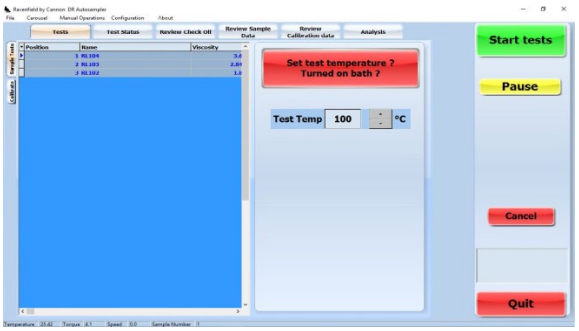

Figure 16: Test Screen

Test Status

The autosampler automatically flushes the previous sample out with a “chase” flush in the same way the instrument is operated manually. Click the **Test Status** tab to monitor the test. Refer to Figure 17.

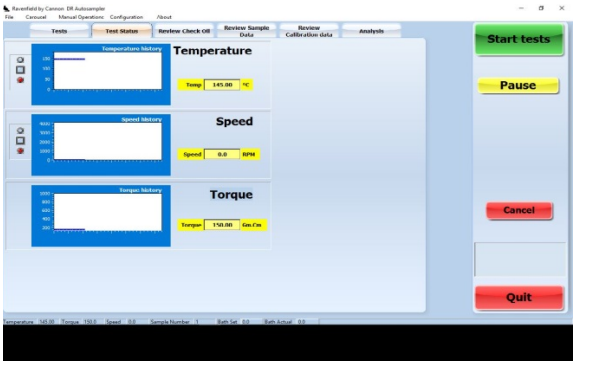

Figure 17: Test Status Tab

The Test Status tab displays and allows the operator to monitor the leds and graphs for the three key variables in the instrument's test: temperature, speed and viscosity. Temperature and speed are controlled by the instrument and their status indicators show high (red), nominal (green), low (red).

Note: The test is likely to fail if the instrument is operating outside of the nominal parameters.

Sample Failure

The version 2 autosampler software provides a safety for proper testing. The temperature of the control bath is automatically adjusted to allow the correct rate of rise for the sample throughput. However, if the system fails, or a test takes longer than five minutes to reach target temperature, then the test is automatically aborted and the next sample is loaded.

Review Samples

If the same type/specification of oil is regularly tested, the data can be monitored by using the built-in SPC functions. The **Review** tab is useful for finding any sample tested in the instrument by using the sample name to find it.

1. Select the **Review Sample Data** tab. Refer to Figure 18.

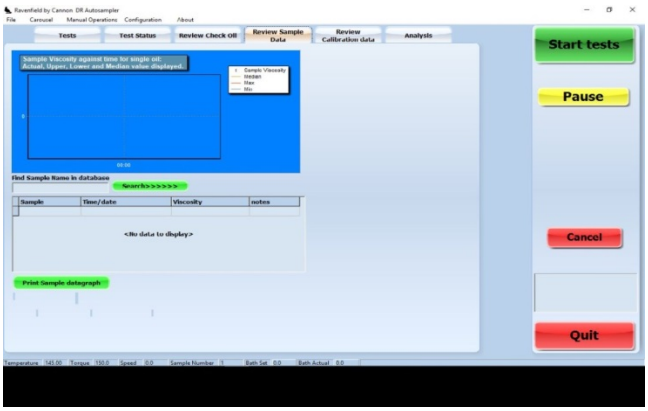

Figure 18: Review Sample Data Tab

2. In the edit box beneath **Find Sample Name in Database** enter part of the name of the sample to review.
3. Hover the mouse cursor over any box that shows only a truncated piece of the data and the hint box will show the full data content.

The software displays the viscosity of all samples in the grid box in the graph at the top of the page combined with three lines representing the median, maximum and minimum value of the grid.

Review Check Oil

The results of testing any oil that has the name RL232 in the sample reference field is stored in a separate table in the internal database for easy examination and quality control. The check oil is a standard oil whose viscosity is tightly controlled by CEC procedures and monitored by CEC statisticians.

1. Samples must be placed on the carousel and labelled carefully. The instrument recognizes any combination or configuration of RL232 in a sample reference name (i.e. rl232, rL232, Rl232).
2. The top, nominal and bottom limits for the check oil viscosity are clearly shown in the typical display screen. The black crosses mark the current test result. Refer to Figure 19.

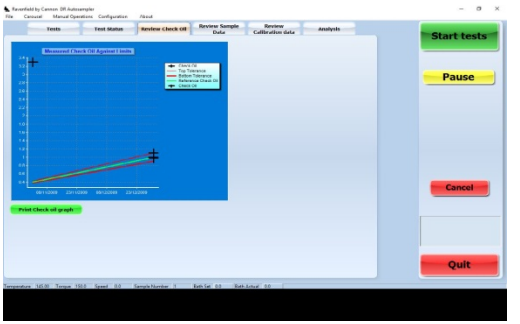

Figure 19: Typical Display

3. If the check oil results are consistently higher or lower than the nominal figure from the check oil, then the instrument generally requires recalibration.

Halt on Bad Oil Check

If the **Halt on Bad Oil** check box is checked in the configuration screen, the autosampler automatically looks for and acts on the result of a test on the instrument check oil RL232.

Samples must be placed on the carousel and labelled carefully. The instrument recognizes any version of the name RL232 (as stated above). If the check oil exceeds the limits in **Check Oil** tolerance the instrument will pause, sound an alert, and place a large red box on the screen displaying the results of the bad test.

The **Pause** button will change to **Paused**.

Appendix 1

Configuration Options

A fundamental part of the autosampler operation is the configuration of its operating parameters. These are stored in the windows registry and can be edited/viewed by clicking on **Configuration** from the Main Menu. Refer to Figure 20.

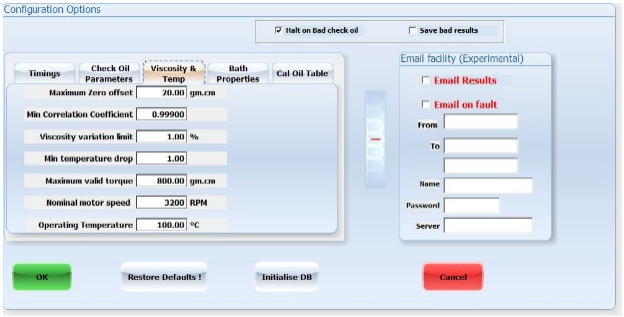

Figure 20: Configuration Screen

There are many parameters that can be changed.

A set of defaults is saved inside the program and it is possible to revert to the factory settings by clicking the **Restore Defaults** button.

Parameters

Timings

Intersample Pump Time: Indicates it is time to switch on pump between different bottles.

Sample Repeat Pump Time: Indicates it is time to switch on pump between different flushes of the same sample bottle. Refer to Figure 21.

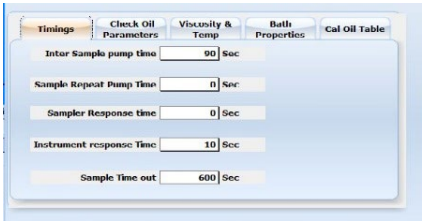

Figure 21: Timing Screen and Sample Repeat Pump Time

Samper Response time: Time permitted before a communication fault between the sampler and the internal computer is declared.

Instrument Response Time: Time permitted before a communication fault between the instrument and the internal computer is declared.

Sample Timeout: Time permitted for sample temperature to rise to test point.

Check Oil Parameters

The **Check Oil Parameters** must be set correctly for HTHS tests to remain within specifications. Refer to Figure 22.

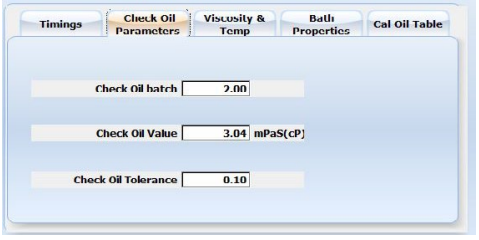

Figure 22: Check Oil Parameters

Check Oil Batch: Ensures that the batch of oil used is the same as that specified in this field. Refer to Figure 23.

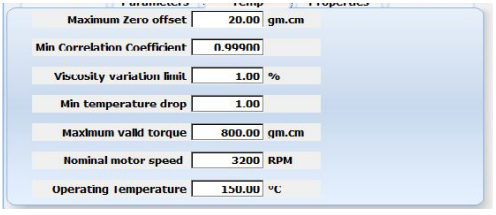

Figure 23: Check Oil Batch Screen

Check Oil Value: The nominal value of the check oil at 10^6 s^{-1}, determined by inter-laboratory cross check program.

Check Oil Tolerance: Maximum permissible error (±, expressed as a percentage). Routine tests on the check oil must agree within the tolerance specified, or the carousel will pause until a fault condition is repaired.

Viscosity and Temperature

Maximum Zero Offset: The instrument returns three calibration factors, a "quality of fit" or **correlation coefficient**, a **scale** and an **offset term**. The offset term is expressed as a torque offset. A measurement quality can be assigned to the torque offset. A low torque offset is desirable. This limit is tested during the automatic calibration process, and specified as **Maximum Zero Offset**.

Min Correlation Coefficient: A low quality fit through the measurements of torque against calibration standard indicates a poor calibration. A *minimum* quality of fit is specified here.

Viscosity Variation Limit: Maximum acceptable variation of determinations of the viscosity of samples of the same bottle.

Min Temperature Drop: This parameter is actually held within the instrument, but it is the minimum drop in temperature which a measurement may become valid. Tests where the temperature in the cell drops by less than this figure between pump cycles are not acceptable.

Maximum Valid Torque: The maximum permissible torque for a test.

Nominal Motor Speed: If the speed drops by more than 1% from the figure entered, an invalid test will be declared. In all but exceptional test conditions, this will be 150 °C.

Operation Temperature: The nominal test temperature. Usually 150 °C.

Bath Properties

This tab is responsible for the successful operation of the automatic temperature compensation system, and is described in depth in *Configuration Options*. Refer to Figure 24.

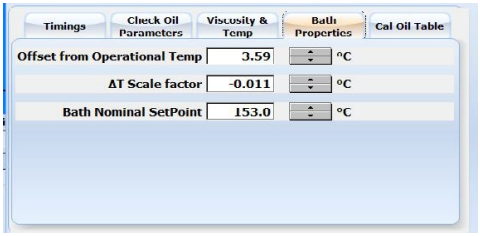

Figure 24: Bath Properties Screen

Appendix 2

Setting Up the Automatic Compensation System

The Ravenfield by Cannon autosampler incorporates sophisticated control algorithms to replace the manual adjustment of bath setpoint during testing. While in routine testing this was not an issue since the usual variability between oils acceptable under SAE standards is within a narrow range, it is an issue during the full range operation of the calibration process. Therefore no attempt to “autocalibrate” had been made.

Automatic compensation relies on making several careful measurements of oils and the creation of constants. This is likely to be further automated in later releases of the autosampler DR V2 code, but is not yet automatic. This calibration will generally only be needed to be performed annually as the parameters are properties of the bath, pipework, and heating bath oil.

The compensation system makes temperature corrections based on a measurement of the instantaneous power input to the sample at the instant that the test cell is at its coldest, after a volume of fresh oil has been pumped to the cell for the **Intersample Pump Time** as defined in the **Configuration** options.

Three parameters will be created and preserved in the **Configuration | Bath** properties and then set in the application registry:

- **Bath Nominal Setpoint**: The temperature at which that the bath needs to be set so that the temperature in the cell delivers the correct cusping* response for the oil selected
- **ΔT scale factor**
- **Offset from Operational Temp**

Note: The latter two properties, **ΔT scale factor** and **Offset from Operational Temp**, are the subject of the procedure described on the next page.

*cusp(ing) – that the temperature in the cell should be rising at no more than 0.1 °C to 0.2 °C in 10 seconds.

Procedure for Setting Up Automatic Compensation System

1. Prepare a full container of RL106 and place in position 1 of the carousel.

2. Prepare a full container of RL102 and place in position 2 of the carousel.
3. Start the Autosampler.
4. Using **Manual** mode, pump RL106 for the ISP time – **Automatic in Manual Operation | Sampler** operations from Main Menu.
5. Record the **Torque** for RL106, T_{106} as the pump stops.
6. Watch the bath temperature and adjust to get the correct cusping* performance with the oil under test.
7. Record bath temperature. This will become **Bath Nominal**.
8. Let bath settle to **Bath Nominal**.
9. Repeat these steps for RL102. Pump sample of RL102.
10. Record **Torque** for RL102, T_{102} as the pump stops.
11. Adjust bath to correct with bath controls in instrument access panel to get RL102 to cusp* correctly.
12. Record the Delta T from **Bath Nominal** (bath for RL106) and now.
13. Plug numbers in equations below to get a slope and offset term. Refer to Figure 25.

*cusp(ing) – that the temperature in the cell should be rising at no more than 0.1 °C to 0.2 °C in 10 seconds

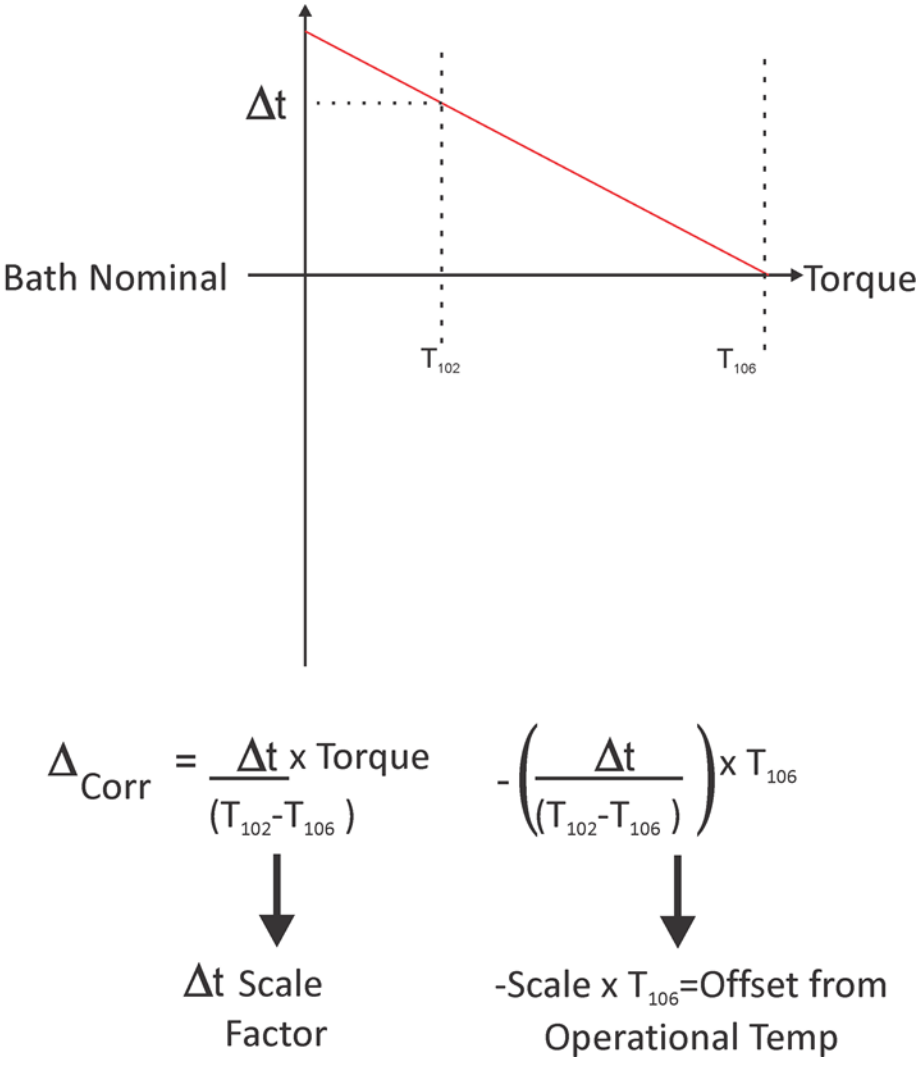

Figure 25: Expect the Scale to be -ve and the offset +ve

CANNON INSTRUMENT COMPANY®

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