# Model 4660 Eclipse Instructions: Rinsing the Sample Pathway

Many performance problems associated with purge-and-trap (P&T) methods are symptomatic of either a leak in the system or the normal wear-and-tear on the instrument and can be corrected or prevented by incorporating a few simple steps into your routine preventative maintenance procedures.

This instruction sheet describes a routine procedure for rinsing the P&T sample pathway with methanol. OI Analytical recommends that this procedure be performed on a regular basis (either monthly or quarterly, depending on actual sample load) or as indicated by the presence of one or more of the performance problems listed. This procedure should be performed after determining that the system is free of any leaks. Once each year, during the annual Preventative Maintenance, the Eclipse transfer line should also be rinsed with methanol.

## Symptoms Indicating the Need to Rinse the Sample Pathway with Methanol

- Poor recoveries of late eluting or brominated compounds
- High %RSDs for compounds eluting after the xylenes in an initial calibration, caused by low response on some standards (low response of standards during a calibration can also be caused by a dirty ion source in the MS)
- Excessive carryover
- Low response for acrolein or 2-chloroethyl vinyl ether (2-CLEVE)
- Low response for ketones or alcohols
- Poor peak shapes, especially for ketones and alcohols
- Purge pressure above 14 psi

## **Required Equipment**

- 1. A Blank trap (#0, part number 228114)
- 2. A 10-mL syringe
- 3. A "rinsing tool" to connect the syringe to the outlet side of the water management fitting.
- 4. A 3/8" wrench
- 5. HPLC- or reagent-grade methanol

If desired, a "P&T Rinsing Kit" can be purchased from OI Analytical as part number 324536. The kit contains all of the tools necessary to rinse the sample pathway, as well as the instruction sheet. If the analytical trap will be changed as part of the procedure, it must be purchased separately.

WARNING: Methanol is toxic. Take appropriate precautions.

#### Rinsing the Eclipse Sample Pathway (not including the transfer line)

1. From the Maintenance screen, step the instrument to either the "Standby" or "Purge Ready" state (refer to Figure 1).



Figure 1. Instrument in "Purge-Ready"

2. Verify that the 6-port valve is in the "Load" position (refer to Figure 2).



Figure 2. 6-Port Valve in the "Load" Position

- 3. Disconnect the P&T transfer line from the GC injection port.
- **NOTE:** Be sure that the GC oven and inlet are both cool and that the carrier gas has been turned off. Follow the GC manufacturer's instructions.
- 4. Turn off the power to the P&T and unplug the power cord. Allow the system to cool.

5. Using the 3/8" wrench, remove the analytical trap and connect the rinsing tool to the outlet (trap) side of the water management fitting (see Figure 3).



Figure 3. Connecting the Rinsing Tool

6. Using the rinsing tool and a syringe, rinse 10 mL of methanol through the sample pathway, collecting it in the sparge vessel (see Figure 4).



Figure 4. Rinsing the Sample Pathway with Methanol

7. From the 4-way valve above the sparge mount, use the syringe to remove the methanol from the sparge vessel (see Figure 5).



Figure 5. Removing the Methanol from the Sparge Vessel

- 8. Repeat steps 6 and 7 twice with clean aliquots of methanol.
- 9. Rinse 10 mL of air through the sample pathway using the syringe and the rinsing tool. Repeat twice.
- 10. Disconnect the rinsing tool from the water management fitting and install a Blank trap (#0, no packing material).
- 11. Connect the P&T power cable and turn the instrument on. Verify that the purge gas supply is on.
- 12. From the 4-way valve above the sparge mount, use the syringe to add 5 mL (or 25 mL) of clean reagent-grade water to the sparge vessel.
- 13. From the Maintenance screen, run the Purge/Bake procedure through 2 cycles to remove any remaining methanol from the system (refer to Figure 6).



Figure 6. Setting the Number of Purge/Bake Cycles

- 14. Install a new analytical trap and condition it for 30 minutes at the recommended temperature (refer to the trap installation instructions, document part number 287250, provided with the trap).
- 15. Re-connect the transfer line to the GC inlet and turn the carrier gas back on.
- 16. Perform the automated Leak Test procedure and confirm that the P&T system is leak free.
- 17. Run a normal system blank with water and Internal/Surrogate standards.

#### **Rinsing the Eclipse Transfer Line**

1. From the Maintenance screen, step the instrument to either the "Standby" or "Purge Ready" state (see Figure 7).



Figure 7. Instrument in "Purge-Ready"

2. Rotate the 6-port valve to the "Inject" position (see Figure 8).



Figure 8. 6-Port Valve in the "Inject" Position

- 3. Disconnect the P&T transfer line from the GC injection port. Place the end of the transfer line in a beaker to catch the rinsate.
- **NOTE:** Be sure that the GC oven and inlet are both cool and that the carrier gas has been turned off. Follow the GC manufacturer's instructions.
- 4. Turn off the power and unplug the power cord. Allow the system to cool.

5. Using the 3/8" wrench, remove the analytical trap and connect the rinsing tool to the outlet (trap) side of the water management fitting (refer to Figure 9).



Figure 9. Connecting the Rinsing Tool

6. Using the rinsing tool and the syringe, rinse 10 mL of methanol through the transfer line (see Figure 10). Repeat twice more with clean aliquots of methanol.



Figure 10. Rinsing the Sample Pathway with Methanol

- 7. Using the rinsing tool and the syringe, rinse 10 mL of air through the transfer line. Repeat until no methanol is visible exiting the transfer line.
- 8. Remove the rinsing tool and re-install the analytical trap (refer to the trap installation instructions, document part number 287250, provided with the trap).
- 9. Connect the power cable and turn the instrument on.

**CAUTION:** Do NOT reconnect the transfer line to the GC injection port.

10. From the Maintenance screen, select Trap Condition and condition the trap for 30 minutes to allow the entire system to bake and remove any remaining traces of methanol (refer to Figure 11).



Figure 11. Conditioning the #10 Trap

- 11. Re-connect the transfer line to the GC inlet and turn the carrier gas back on.
- 12. Perform the automated Leak Test procedure and confirm that the P&T system is leak free.
- 13. Run a normal system blank with water and Internal/Surrogate standards.



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