



MPI: Customer Note. Calibrating OVG-4 Temperature & Flow

Issue/Version	Date	Author	Details
AAA	10/01/11	Richard Muffett	The original

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1 Introduction

1.1 Document Purpose

The purpose of this document is to provide instructions for checking and calibrating the temperature of the Permeation Oven and Sample Outlet flow of the Owlstone Vapour Generator (OVG-4), P/N 00-0003.

The document is based upon Owlstone's Factory Acceptance Testing procedure, document 90-0103.

1.2 Nomenclature

Term or abbreviation	Definition
OVG-4	Owlstone Vapour Generator
MFC	Mass Flow Controller

2 Health and Safety

2.1 Hazards

- Compressed air
- Chemical Hazard

It is the responsibility of the customer to assess chemical hazards / COSHH associated with this procedure. Owlstone cannot be held responsible for the chemical safety of offsite equipment

2.2 Required Training

- Persons must read and understand all aspects of this procedure before attempting to check or to calibrate the OVG-4 Sample Flow and oven temperature.
- Any questions should be directed to Owlstone technical support before or during this process.

3 Equipment required

3.1 Flow Meter

Must have an operating range greater than 0 - 500ml/min. Accuracy should be better than +/-1% reading.

Owlstone use a volumetric flow standard instrument manufactured by Bios inc. called a Definer 220+

www.biosint.com

3.2 Digital thermometer & temperature probe

The minimum operating range of must be 20 - 120°C. Accuracy should be better than +/-2% reading.

Owlstone use a Digitron 2046T digital thermometer and H0334D rigid air probe.

www.digitron.co.uk

Bios Definer 220 :-



Digitron 2046T coupled with HO334D rigid air probe :-



Tape aids location of probe handle in Permeation Oven

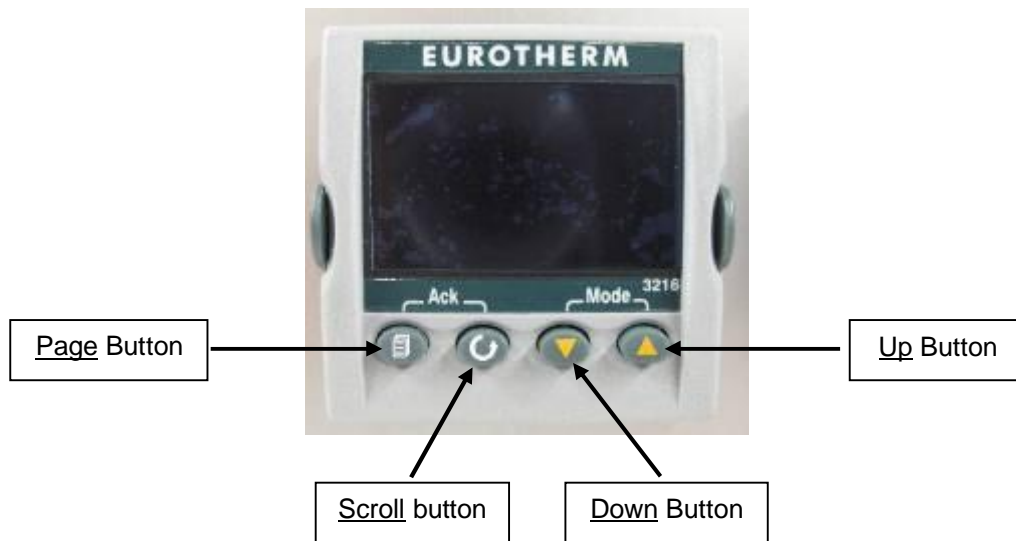
PTFE sleeve with end plug prevents probe touching oven wall

Important . rigid air probe must be assembled inside a length of $\frac{1}{4}$ PTFE tube as shown. This ensures that the air temperature measured is equivalent to that experienced by the gas phase of the analyte.

4 Procedures

4.1 Note on Eurotherm Controller buttons

The following button naming convention is used throughout this procedure.



4.2 Auto Tune

4.2.1 Why auto tune?

The Auto Tune function is a sophisticated Eurotherm hardware procedure that optimises PID settings in firmware so that the users mechanical system does not overshoot the set-point or fluctuate wildly.

Owlstone recommends the Auto Tune procedure should be undertaken..

- 1) Before undertaking a temperature or flow calibration
- 2) If the user observes unstable or fluctuating sample flow or oven temperatures

4.2.2 Prepare to Auto Tune

Before undertaking an Auto Tune both the permeation Oven temperature and the Sample Flow should be mid-range. Owlstone recommends the user sets the oven to 60°C and Sample Flow 250ml/min.

The Auto Tune procedure is the same for both the Temperature and the Flow Eurotherm controllers.

4.2.3 Auto Tune Procedure

- i. Press and hold down the Page button until Level 3 is displayed (approximately 5 seconds).
- ii. When prompted %Code+enter % by using the up arrow, wait approx. 3seconds
- iii. Now press the Scroll button repeatedly until A.Tune is displayed.
- iv. Press the up arrow. The display will state %n+. Wait approx 5 seconds.
- v. Press the Page button to return to the normal view.
- vi. When the auto tune is complete the %auto tune+message will clear.

4.3 System Leak Test

4.3.1 Basic leak test

- i. Set the flow controller to 500 ml/min.
- ii. Once the flow is stable at 500 ml/min fit a 1/8" Swagelok cap to the OVG sample outlet port. The process value ml/min should fall quickly, this is normal behaviour.
- iii. If after 15mins the flow display has not reached a value of less than 15ml/min then there may be a leak inside the OVG. Please contact Owlstone Technical Support.
- iv. Once this test is complete remove the blanking cap from the OVG sample outlet. The flow will return to the 500ml/min set point.
- v. Whilst this test is being carried out both Flow and Temperature controllers will alarm. The Flow Controller alarm can be reset once the blanking cap is removed and the process value within 50ml/min of the set-point.
- vi. The Temperature controller alarm can be reset once the process value has reached the set-point.

4.3.2 Comprehensive leak test

Contact Owlstone Technical Support for comprehensive leak testing or troubleshooting advice.

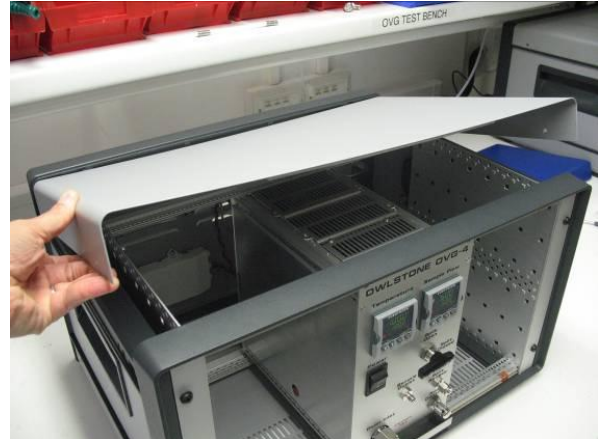
4.4 Temperature Calibration

To check and / or calibrate the OVG-4 you will need a Digital Thermometer and Rigid Air Probe.

The probe is placed inside the oven and a range of temperatures checked and recorded. If the measured temperature is not within $\pm 0.2^{\circ}\text{C}$ of the set-point then the OVG-4 should be re-calibrated.

4.4.1 Determining whether calibration is required?

- i. Set a set-point of 0ml/min on the Sample Flow Controller.
- ii. Remove the four screws holding the rack lid in place.



- iii. Next remove air pressure by disconnecting the quick connect fitting at the rear of the OVG.



OVG gas supply quick release fitting

- iv. Remove Oven Inlet cap and place the rigid air probe inside the oven as shown below.

The handle of the probe must be positioned so that the opening of the oven inlet is sealed. Any gaps here may lead to temperature losses and an invalid calibration.



- v. Set the OVG-4 set-point temperature to 30°C and wait for the process value to reach this temperature. If the oven is hot then cooling to 30 °C could take 1hour.
- vi. Once the displayed set-point temperature is stable record the temperature measured by the Digital Thermometer.
- vii. Repeat steps v, vi for temperatures of 60°C and 100°C.
- viii. If measured temperatures are more than $\pm 0.2^\circ\text{C}$ from the set-point value then Owlstone recommend that the OVG-4 is re-calibrated.

4.4.2 Calibration Procedure

Calibration is straightforward and achieved via user programmable High . Low offsets at 30, 100°C.

The reader must decide whether offsets should be applied at both 30°C and 100°C, often an offset is only required at one of the two values. This decision requires data to be collected as per 4.4.1 above.

The calibration process is time consuming and can be iterative. From experience Owlstone recommend that the first offset to be implemented is the Low offset (30°C) however this is not essential.

- i. Set the OVG to 30°C and allow the temperature to stabilise.
- ii. Once the temperature is stable at 30°C navigate to the calibration menu of the Eurotherm controller:-

- Press and hold the Page button until Level-3 is displayed. When prompted on the screen enter the access code 3 by using the up arrow. Wait a few seconds for it to be accepted.
- Press the Page button until the word CAL is displayed on the screen.
- Press the Scroll button until Lo UCAL is displayed on the screen.
- Use the up and down arrows to set the temperature to the value reported by the Digital Thermometer
- To return to the normal display press the Page and Scroll buttons together

The temperature displayed will now match exactly that shown on the Digital Thermometer.

- iii. Wait at least 15mins and check that the temperature is stable at the 30.0°C set-point.
- iv. Check that the temperature displayed on the Digital Thermometer is still within $\pm 0.2^\circ\text{C}$ of the displayed temperature. The Low offset adjustment is now complete.

If data collected in 4.4.1 indicates the 100degC set-point needs adjusting then the procedure is as follows..

- i. Set the oven to 100°C and allow the temperature to climb and stabilise.
- ii. Once at temperature navigate to the calibration menu in the same way as before but instead of going to Lo UCAL, press the up arrow button until the display shows Hi UCAL.
- iii. Alter the temperature using the up or down arrow keys so that the Eurotherm displays the same value as the Digital Thermometer at 100°C.
- iv. Wait at least 15mins and check that the oven temperature is stable at 100°C.

Following adjustment of High and Low offset values the calibration must once again be checked at 30°C, 60°C and 100°C. Again use your digital thermometer and follow the procedure described in 4.4.1.

If measurements at 30°C, 60°C and 100°C are all within $\pm 0.2^\circ\text{C}$ then the calibration is complete.

If the recorded temperatures are not within $\pm 0.2^\circ\text{C}$ then Hi . Low offsets will need modifying.

Once the calibration is complete:-

- i. remove the probe and close the oven inlet
- ii. Reconnect the quick release fitting at the rear of the OVG-4 and replace the rack lid.

Offsets entered, the s/n of the Digital Thermometer, Date and reason for re-calibration should be recorded and retained for future reference.

4.5 Flow calibration

4.5.1 General Note

- 1) It is essential that the pressure supplied to the GEN-SYS rack is at or near 40psi before attempting to calibrate the OVG-4 Sample Flow controller. Failure to do this may result in a poor calibration.
- 2) It is essential that a suitably accurate flow standard is utilised. Owlstone recommend the Bios Definer 220 volumetric flow standard which offers a standardised accuracy of +/-1%.
- 3) Connections between the OVG-4 hardware and flow standard must be leak tight.

4.5.2 Procedure

- i. Set the sample flow set-point to 100sml/min.
- ii. Connect your flow meter to the Sample Outlet port as shown above.
- iii. Wait a few minutes for the flow to stabilise. Record the flow reported by your flow meter.
- iv. Set the sample flow set-point to 500sml/min.
- v. Wait a few minutes for the flow to stabilise. Record the flow reported by your flow meter.

If measured flows differ significantly from flow reported by the OVG-4 then calibration is required. Owlstone recommend that the Sample Flow output is calibrated to within 1.5% of set-point.

Calibration uses the linear High . Low offset method as per the temperature calibration described in 4.4.2. Once again Owlstone recommend that the Low offset is implemented before the High offset.

- i. Using the Sample Flow controller input a set-point of 100sml/min. Wait a few minutes for the reported flow to stabilise at 100sml/min.
- ii. Press and hold down the Page button until Level 3 is displayed. When prompted on the screen enter the access code 3 by using the up arrow and wait a few seconds for it to be accepted.
- iii. Press the Page button until the word CAL is displayed on the screen.
- iv. Press the Scroll button until Lo UCAL is displayed on the screen.
- v. Use up and down arrows to set the flow to that reported by your flow meter
- vi. To return to the normal display press the Page and Scroll buttons together

Allow a few minutes for the flow to stabilise. Check that both the flow displayed by the OVG-4 and that reported by your flow meter read 100sml/min.

If the initial 500 ml/min reading was within 1.5% then it should not need to be calibrated. Otherwise repeat steps i. . vi. above but this time modify the Hi UCAL parameter.

Owlstone recommend that following calibration the Sample Flow is checked at 100, 200, 300, 400 and 500sml/min. The output flow at all data points should be < +/-1.5% error.

Record adjustments made, the s/n of the flow standard used, the date and reason for re-calibration.