Agilent CrossLab Compliance Services

Agilent CrossLab
From Insight to Outcome

GC HARDWARE
OQ OPTIONAL TESTS AND VARIANCES

Extra Tests for OQ

The following tests are NOT INCLUDED in the standard OQ for GC but can be ordered as EXTRA COST TESTS. Select the check boxes on the right and attach this document to your OQ EQP documentation for a record of qualification conditions.

Note: LTM and LTM II tests apply only if those modules are installed.

Key: Fixed setpoints/limits | Variances allowed

Test	Setpoints and Parameters	Limits	Include
Injection Carry Over (ALS only)	Injection volume on column: 1.0 ul	Area carry over ≤ 1.00%	☐
Response Linearity (FID, TCD only)	Certified reference standard with multiple peaks known concentration	Coefficient of determination (r2) ≥ 0.99900 R/F precision RSD ≤ 10.00%	☐
GC Heated Zones Temperature Accuracy (Agilent Intuvo 9000)	Inlet temp. = 250.0°C (SSL, MMI) Inlet trap temp. = 300.0°C (SSL, MMI) Detector zone temp. = 300.0°C	Inlet accuracy ≤ 10.0°C (inlet) Inlet accuracy ≤ 30.0°C (inlet trap) Detector accuracy ≤ 20.0°C (all zones)	☐
GC Heated Zones Temperature Accuracy (uECD/MS not supported)	Inlet temp. = 200.0°C/250.0°C (OC/all others) Detector temp. = 200.0 - 300.0°C (varies by detector type)	Inlet accuracy ≤ 15.0°C / 10.0°C (OC/all others) Detector accuracy ≤ 15.0°C	☐
GC Oven Temp. Ramp: Accuracy, Linearity, Precision	Initial temperature: 50.0°C Ramp: 100.0°C/minute (Intuvo 9000 GC) Ramp: 30.0°C/minute (other Agilent GCs) Final temperature: 280.0°C	Ramp accuracy ≤ 10.0°C/minute (Intuvo 9000) Ramp accuracy ≤ 1.0°C/minute (Others) Ramp linearity ≥ 0.99990 Ramp precision ≤ 2.00%	☐
LTM Basic Operation	Temperature: 60°C	Self test completes without errors Reference voltage (TP7) = 784 +/- 10 mV Transfer lines 1, 2 (TP3, TP5) = 794 +/- 50 mV Column temperature (TP1) = 784 +/- 10 mV	☐
LTM Oven Temperature Accuracy and Stability	Temperature 1: 230.0°C Temperature 2: 100.0°C Stability measured at temperature 2	Difference from setpoint ≤ 1.0% in K Stability ≤ 0.5°C	☐
LTM Oven Temperature Ramp	Initial temperature: 50.0°C Ramp: 100.0°C/minute Final temperature: 280.0°C	Ramp accuracy ≤ 2.0°C/minute Ramp linearity ≥ 0.99900 Ramp precision ≤ 2.00%	☐
LTM II Basic Operation	Set column set to 50.0°C to avoid damaging it without carrier	Self test completes without errors All LTM II heated zones heat up	☐
LTM II Oven Temperature Accuracy	Temperature: 60.0°C	Transfer lines 1, 2 accuracy ≤ 4.0°C Column temperature ≤ 3.0°C	☐
LTM II Oven Temperature Ramp	Initial temperature: 50.0°C Ramp: 100.0°C/minute Final temperature: 280.0°C	Automated test verifies that the ramp test is completed without any not ready statuses.	☐

Test Design and Rationale

Injection Carry Over (ALS)

Description: Low carry over from a previous injection is critical for accuracy and reliability of qualitative analysis. For liquid samplers, carry over performance is contingent on many variable factors independent of the engineering condition of the GC system. Many different syringe wash programs that can eliminate carry over are user selectable and may be application specific. The condition of the injection syringe is the only controllable engineering factor, and it is typically replaced during PM before OQ. Therefore, the carry over test for liquid samplers is offered only as an optional extra fee test in a customer-configured EQP.

Procedure: This procedure must use a column. If a column is used for the Injection Precision test, the blank injection after the six repeat injections of the precision test is evaluated for carry over, and the result is expressed as a percentage of the evaluated peak of the final run from the IP sequence. If a column is specifically installed for this test, a single run of a standard followed by a blank is used to calculate carryover.

Response Linearity (FID, TCD)

Description: Response linearity is critical for quantitative analysis. It is often demonstrated in user applications and analytical methods typically using multi-level calibration standards and internal standards. Therefore, this is an optional extra fee OQ test. The response linearity test uses a certified chemical reference test mix that is validated to be challenging and representative of many applications.

Procedure: The response linearity test is executed using a single injection from a standard containing a number of n-alkanes with increasing concentrations. Response linearity can be calculated with just one injection of a standard for the following reasons.

- The difference in length of the n-alkanes (boiling-point increases) separates these components on the column.
- The increasing concentration gives an increasing detector response.
- GC theory states (and experiment confirms) that the response factors for these compounds are the same (within a very small variance). Therefore, a single injection of this multi-component /multi-level concentration sample can be used to calculate the response linearity of the detector.
- The single injection test design eliminates the contribution of injector precision to the linearity statistics evaluation.

GC Heated Zones Temperature Accuracy

Description: The precise temperature of the heated zones is not critical to quantitative or qualitative analysis. When the inlet zones are hot enough to vaporize but not so hot as to thermally decompose sample, this is adequate. When the detector zones are hot enough to evaporate sample and prevent condensation, this is adequate. Temperature accuracy of the heated zones may be important for comparing systems and transfer methods. Therefore, this is an optional test.

Procedure: This test demonstrates that the inlet and detector show an accurate temperature using proprietary novel design to overcome the inherent difficulties in gaining accurate and meaningful readings. The temperature is measured using an external thermometer. The probe is inserted as if it is a column with a pre-defined length above the column nut to get consistent measurements between different instruments. (Note: Due to the possible risk of radioactive contamination, ECDs are excluded from this service).

GC Oven Temperature Ramp: Accuracy, Linearity, and Precision

Description: Most GC analyses use a temperature program instead of an isothermal oven temperature program to complete the separation of the compounds in the sample. For retention time reproducibility, it is important that the temperature program is always executed in the same way.

This test uses a calibrated digital thermometer to determine the accuracy, linearity, and precision of the GC oven temperature program. Linearity is defined as the correlation coefficient (r) and uses data points that are part of the ramp. Ramp accuracy is defined as the absolute difference between the slope of the linear curve fit through the same data points used for linearity calculations (calculated ramp) and its setpoint. Precision is calculated as the RSD value over the three calculated ramps.

Procedure: In this test, a linear oven temperature profile is collected three times in a row using a digital thermometer and a data logger. For each run, the oven ramp accuracy and oven ramp linearity are calculated. After all three runs are completed, the oven ramp precision is calculated.

The ramp in use is a steep slope that challenges the GC to deliver high power in a reproducible way.

LTM Tests

Description: The RTD is a column packed in a heating foil. Although columns are generally considered to be consumables and not part of a hardware qualification, in this case the “oven” includes the column so tests are required to evaluate its functionality.

A direct temperature measurement (vs. indirect) is preferred but not feasible in this case given the LTM design: adding a temperature sensor to the metal foil introduces a cold spot and adversely affects temperature, and inserting a probe into the RTD requires taking the column apart.

One indirect temperature measurement is a direct measurement of the return voltage from the RTD, which can be converted to temperature using a known equation.

Another indirect temperature measurement would be chemical tests. If the system is not able to heat up in a reproducible way, you might see a shift in retention times. Because this kind of measurement is used by Agilent (and many other vendors) to evaluate system performance, it would be difficult for LTM to rework the complete chemical test suite: especially detector-specific tests like Signal to Noise and Signal Noise and Drift. The RTD can be any column and noise, in particular, is influenced by the column type.

Based on the above, the following qualification is executed when an LTM is installed:

1. A complete GC qualification without Injection Precision (IP) is run with standard oven procedures. An LTM Basic Operation test is scheduled to show the LTM is functional.
2. An LTM Oven Temperature Accuracy and Stability test is executed. This test is similar to the standard GC Oven Temperature Accuracy and Stability.
3. An LTM Oven Temperature Ramp test is executed, similar to the standard GC Oven Temperature Ramp test, but it uses a much higher ramp.
4. The IP test is run using the LTM module. Inlet, detector, and RTD modules are tested separately in steps 1-3, but this test verifies that all components work together. LTM runs in general are very short due to the high oven ramp and very fast cool down rate.

Procedure for LTM Basic Operation: After completing the self-test, four different temperatures (voltages) are measured: reference voltage (setpoint), return voltage (column temperature), and both transfer lines. This assures that all zones are functional, correctly installed, and connected.

Procedure for LTM Oven Temperature Accuracy and Stability: This test uses a calibrated voltmeter to determine temperature accuracy and stability of the LTM oven. (Voltages are measured and then converted to temperatures using a known relation. The converted temperatures are used in all calculations and limit comparisons.)

Procedure for LTM Oven Temperature Ramp: This test uses a calibrated digital thermometer to determine the accuracy, linearity, and precision of the LTM oven temperature program. Linearity is defined as the correlation coefficient (r) and uses data points that are part of the ramp. Ramp accuracy is defined as the absolute difference between the slope of the linear curve fit through the same data points used for linearity calculations (calculated ramp) and its setpoint. Precision is calculated as the RSD value over the three calculated ramps.

LTM II Tests

Description: Same as LTM tests.

Procedure for LTM II Basic Operation: After completing the self test, all heated zones of the LTM II column are measured to verify that they heat up. This assures that all zones are functional, correctly installed, and connected.

Procedure for LTM II Oven Temperature Accuracy: This test uses a controlled environment to heat the LTM II column without applying power (heat) to the LTM II module. When temperature is stabilized, the LTM II temperature sensors reported values are recorded and compared against the temperature setpoint of the controlled environment.

Procedure for LTM II Oven Temperature Ramp: This test uses a built-in test to evaluate the LTM oven temperature program.

Allowed Variance Ranges

The simplest and most common occurrence is the Agilent Recommended test program - whereby the acceptance and approval refers to the fixed standards qualification tests and setpoints as recorded in the Agilent Recommended EQP. In this case, verbal confirmation of approval after customer review is sufficient for Agilent service to proceed with scheduling and delivery.

Agilent defines variances as changes to the default recommended values (as stated in the Agilent Recommended EQP) that fall within a range of well-defined allowable changes. These changes are considered to be within the intended use range of the system under test. The following table shows the allowed variance ranges for the test setpoints that can be configured. Agilent reserves the right to warrant conformance only when the tests definition lay within the maximum and minimum values shown.

#	Test (A# are additional tests (not part of standard program); same minimum values apply to Agilent and (as applicable) non-Agilent systems.		Agilent Setpoint			Units	Non-Agilent Setpoint	
			Min	Default	Max		Default	Max
1	Oven Temperature Accuracy #1	(Intuvo 9000, oven)	40.0	230.0	300.0	°C	N/A	N/A
		(Others Agilent GCs)	40.0	230.0	350.0	°C	230.0	350.0
2	Oven Temperature Accuracy #2	(Intuvo 9000, oven)	40.0	100.0	300.0	°C	N/A	N/A
		(Others Agilent GCs)	40.0	100.0	350.0	°C	100.0	350.0
3	Oven Temperature Accuracy #3	(Intuvo 9000, col. connector)	150.0	250.0	300.0	°C	N/A	N/A
4	Oven Temperature Stability	(Intuvo 9000, oven)	40.0	100.0	300.0	°C	N/A	N/A
		(Others Agilent GCs)	40.0	100.0	350.0	°C	100.0	350.0
5	HS Heated Temp. Acc. Transferline	(7697A)	40.0	115.0	250.0	°C	N/A	N/A

#	Test (A# are additional tests (not part of standard program); same minimum values apply to Agilent and (as applicable) non-Agilent systems.		Agilent Setpoint			Units	Non-Agilent Setpoint	
			Min	Default	Max		Default	Max
		(Others)	40.0	115.0	300.0	°C	115.0	300.0
6	HS Heated Temp. Acc. Sample Loop	(G1888, G1883)	40.0	110.0	150.0	°C	N/A	N/A
		(7697A)	40.0	110.0	210.0	°C	N/A	N/A
		(Others)	40.0	110.0	300.0	°C	110.0	300.0
7	HS Heated Temp. Acc. Syringe Heater		40.0	110.0	150.0	°C	110.0	150.0
8	HS Heated Temp. Acc. Oven	(7697A)	40.0	100.0	210.0	°C	N/A	N/A
		(7694, G1888, G1883)	40.0	100.0	150.0	°C	N/A	N/A
		(Other loop-based)	40.0	100.0	300.0	°C	N/A	N/A
		(Others)	40.0	100.0	150.0	°C	100.0	150.0
9	HS Heated Temp. Acc. Agitator		40.0	100.0	150.0	°C	100.0	150.0
10	Vial Heater Temp. Accuracy		40.0	60.0	80.0	°C	N/A	80.0
11	Inlet Pressure Accuracy		5.0	25.0	50.0	psi	25.0	50.0
12	Injection Precision	(ALS)	0.2	1.0	3.0	µl	1.0	3.0
13	Injection Precision, Carry Over	(HSS)	250	1000	3000	µl	1000	3000
		(PB HSS)	0.01	N/A	N/A	minutes	0.02	0.10
A1	Injection Carry Over	(ALS)	0.2	1.0	3.0	µl	1.0	3.0
A2	GC Inlet Temp. Accuracy	(Intuvo 9000 trap)	50.0	300.0	300.0	°C	N/A	N/A
		(OC)	40.0	200.0	220.0	°C	200.0	220.0
		(Intuvo 9000 inlet, other inlets)	40.0	250.0	350.0	°C	250.0	250.0
A3	GC Detector Temp. Accuracy	(FID/TCD/NPD)	40.0	300.0	350.0	°C	250.0	350.0
		(FPD)	40.0	200.0	250.0	°C	200.0	250.0
A4	GC Oven Ramp Initial Temperature		40.0	50.0	100.0	°C	50.0	100.0
A5	GC Oven Ramp Rate	(Intuvo 9000)	2.0	100.0	100.0	°C/minute	N/A	N/A
		(Other Agilent GCs)	2.0	30.0	40.0	°C/minute	20.0	25.0
A6	GC Oven Ramp Final Temperature		150.0	280.0	350.0	°C	280.0	350.0
A7	LTM Oven Temperature Accuracy #1		40.0	230.0	350.0	°C	N/A	350.0
A8	LTM Oven Temperature Accuracy #2		40.0	100.0	350.0	°C	N/A	350.0
A9	LTM Oven Temperature Stability		40.0	100.0	350.0	°C	N/A	350.0
A10	LTM Oven Ramp - Initial Temperature		40.0	50.0	100.0	°C	N/A	100.0
A11	LTM Oven Ramp Rate		20.0	100.0	1500.0	°C/minute	N/A	1500.0
A12	LTM Oven Final Temperature		150.0	280.0	350.0	°C	N/A	350.0
A13	LTM II Oven Temperature Accuracy		45.0	60.0	125.0	°C	N/A	125.0
A14	LTM II Oven Ramp - Initial Temperature		40.0	50.0	100.0	°C	N/A	100.0
A15	LTM II Oven Ramp Rate		20.0	100.0	1500.0	°C/minute	N/A	1500.0
A16	LTM II Oven Final Temperature		150.0	280.0	350.0	°C	N/A	350.0

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