Agilent CrossLab Compliance Services

Agilent CrossLab
From Insight to Outcome

# GC HARDWARE
# OPERATIONAL QUALIFICATION

## Standard OQ Test Suite

This document describes the test program for qualifying GC systems, and the following table lists all OQ tests.

**Note**: Headspace tests apply only if a headspace sampler is an integral part of the system; Injection Carry Over is included in the standard OQ for GCs with headspace configurations but not for liquid sampler configurations (it can be ordered as EXTRA COST TEST).

**Key:** Fixed setpoints/limits | Variances allowed

| Test | Setpoints and Parameters | Limits |
|---|---|---|
| System Inspection and Basic Safety and Operation | N/A | Gases, chassis electric grounding, interlocks, hydrogen shutdown, and so on all correct |
| GC Oven Temperature Accuracy and Stability (Agilent Intuvo 9000) | Column connector = 250.0°C<br>Oven 1 = 230.0°C<br>Oven 2 = 100.0°C<br>Stability measured at oven 2 | Accuracy ≥ -5.0% and ≤ 5.0% of setpoint in K (oven)<br>Accuracy ≥ -1.0% and ≤ 2.0% of setpoint in K (column connector)<br>Stability ≤ 0.5°C |
| GC Oven Temperature Accuracy and Stability | Temperature 1 = 230.0°C<br>Temperature 2 = 100.0°C<br>Stability measured at temperature 2 | Accuracy ≥ -1.0% and ≤ 1.0% of setpoint in K<br>Stability ≤ 0.5°C (Agilent)<br>Stability ≤ 1.0°C (Others) |
| Headspace Leak (7697A only) | N/A | Valve functions properly and HSS is leak tight |
| Headspace Vent and Pressurization Valve Integrity (G1888A and older) | N/A | Valve functions properly |
| Headspace Heated Zones Temperature Accuracy | Tline: 115.0°C<br>Sample Loop: 110.0°C<br>Syringe Heater: 110.0°C<br>Oven: 100.0°C<br>Agitator: 100.0°C<br>(Applicable zones vary by model;<br>* TurboMatrix 40, TurboMatrix 16, TurboMatrix 110, HS40XL, HS110, HS110XL models only)<br><br>Tline is transferline. | Tline accuracy ≥ -1.8 and ≤ 5.2% of setpoint (7697A, 7697A w/tray)<br>Tline accuracy ≥ -4.3 and ≤ 4.3 % of setpoint (Others)<br>Sample loop accuracy ≥ -4.0°C and ≤ 4.0°C (G1883, G1888A, 7697A, 7697A w/tray)<br>Sample loop accuracy ≥ -5.0°C and ≤ 5.0°C (Others)<br>Syringe heater accuracy ≥ -2.0°C and ≤ 2.0°C (CTC)<br>Syringe heater accuracy ≥ -5.0°C and ≤ 5.0°C (Others)<br>Oven accuracy ≥ -6.0°C and ≤ 6.0°C (7694, G1289B, G1290B)<br>Oven accuracy ≥ -4.0°C and ≤ 4.0°C (G1883, G1888A)<br>Oven accuracy ≥ -5.0°C and ≤ 5.0°C (PerkinElmer*)<br>Oven accuracy ≥ -4.0°C and ≤ 4.0°C (PerkinElmer other models)<br>Oven accuracy ≥ -4.0°C and ≤ 4.0°C (7697A, 7697A w/tray)<br>Oven accuracy ≥ -5.0°C and ≤ 5.0°C (Others)<br>Agitator accuracy ≥ -2.0°C and ≤ 2.0°C (CTC) |
| Vial Heater Temperature Accuracy | Temperature 1: 60.0°C<br>Setpoints for temperature 2 and 3 are variable | Diff. from setpoint ≥ – 2.0°C, ≤ 2.0°C |
| Inlet Pressure Decay (EPC or manual control only) | Inlet gas flow control | Pressure change / 5 minutes ≥ –2.0 psi and ≤ 0.5 psi |
| Inlet Pressure Accuracy (EPC or manual control only) | Inlet pressure: 25.0 psi | Accuracy ≤ 1.2 psi |
| Inlet Flow Stability (EFC control only) | Inlet flow: 4.0 ml/minute | Accuracy ≤ 10.0%<br>Precision ≤ 5.0% |

| Test | Setpoints and Parameters | Limits |
|---|---|---|
| Detector Flow Accuracy | Flow rate varies by detector type (N/A for NPD) | Accuracy ≤ 10.0% of setpoint (or 0.5 ml/minute, whichever is larger) |
| Noise and Drift (FID) | Detector signal | Noise ≤ 0.10 pA<br>Drift ≤ 2.50 pA/hour |
| Noise and Drift (TCD) | Detector signal | Noise ≤ 0.15 DU (He or $H_2$ carrier and makeup [or no makeup])<br>Noise ≤ 0.25 DU ($N_2$ carrier and makeup [or no makeup])<br>Drift ≤ 2.20 DU/hour |
| Noise and Drift (NPD) | Detector signal (N/A for 5890) | Noise ≤ 0.15 pA<br>Drift ≤ 3.50 pA/hour |
| Noise and Drift (ECD) | Detector signal (N/A for 5890) | Noise ≤ 0.15 DU<br>Drift ≤ 1.00 DU/hour |
| Noise and Drift (uECD) | | Noise ≤ 3.00 DU<br>Drift ≤ 15.00 DU/hour |
| Noise and Drift (FPD new style) | Detector signal, sulfur (N/A for 5890) | Noise ≤ 5.00 DU<br>Drift ≤ 5.00 DU/hour |
| Noise and Drift (FPD+) | | Noise ≤ 4.00 DU<br>Drift ≤ 3.00 DU/hour |
| Noise and Drift (FPD new style) | Detector signal, phosphorous (N/A for 5890) | Noise ≤ 5.00 DU<br>Drift ≤ 5.00 DU/hour |
| Noise and Drift (FPD+) | | Noise ≤ 2.00 DU<br>Drift ≤ 1.50 DU/hour |
| Noise and Drift (NCD) | Detector signal | Noise ≤ 5.00 pA<br>Drift ≤ 50.00 pA/hour |
| Noise and Drift (SCD) | Detector signal | Noise ≤ 15.00 pA<br>Drift ≤ 50.00 pA/hour |
| Scouting Run | Injection volume on column: varies by configuration | N/A |
| Signal to Noise (FID/SS/MMI/ALS) | Signal height divided by ASTM baseline noise for known concentration and conditions. | S/N ≥ 300,000 ($N_2$ makeup gas)<br>S/N ≥ 240,000 (He makeup gas) |
| Signal to Noise (FID/SS/MMI/HSS) | | S/N ≥ 5,000 ($N_2$ makeup gas)<br>S/N ≥ 4,000 (He makeup gas) |
| Signal to Noise (FID/VI/HSS) | | S/N ≥ 4,000 ($N_2$ makeup gas)<br>S/N ≥ 3,200 (He makeup gas) |
| Signal to Noise (FID/non-SS/using 18710-60170) | | S/N ≥ 800 ($N_2$ makeup gas)<br>S/N ≥ 600 (He makeup gas) |
| Signal to Noise (FID/non-SS/using 5188-5372 | | S/N ≥ 200 ($N_2$ makeup gas)<br>S/N ≥ 160 (He makeup gas) |
| Signal to Noise (NPD) | | S/N ≥ 300 |
| Signal to Noise (TCD/SS/MMI) | | S/N ≥ 750 ($N_2$ makeup gas)<br>S/N ≥ 5,000 (He or H2 makeup gas) |
| Signal to Noise (TCD/non-SS/MMI) | | S/N ≥ 4 (N2 makeup gas)<br>S/N ≥ 100 (He or $H_2$ makeup gas) |
| Signal to Noise (uECD) | | S/N ≥ 1,500 |
| Signal to Noise (FPD new style) | | S/N ≥ 700 (sulfur)<br>S/N ≥ 1,000 (phosphorous) |
| Signal to Noise (FPD+) | | S/N ≥ 1,400 (sulfur)<br>S/N ≥ 2,400 (phosphorous) |
| Signal to Noise (NCD) | | S/N ≥ 600 (NCD) |
| Signal to Noise (SCD) | | S/N ≥ 180 (SCD)<br>S/N ≥ 18 (SCD on FID base) |

| Test | Setpoints and Parameters | Limits |
|---|---|---|
| Injection Precision (Split/Splitless) | Injection volume on column: 1.0/1000/250 ul (ALS/Agilent HSS/CTC HSS with split/splitless FID)<br>Injection time: 0.2 minutes (pressure-balanced HSS only) | Retention time RSD ≤ 1.00%<br>Area RSD ≤ 3.00% (ALS, Agilent HSS)<br>Area RSD ≤ 4.00% (CTC HSS)<br>Area RSD ≤ 8.00% (NCD, SCD; ALS only) |
| Injection Precision (Purged/Packed) | ALS with purged/packed injection port; without HSS | Retention time RSD ≤ 1.00%<br>Area RSD ≤ 3.00% (FID, TCD)<br>Area RSD ≤ 5.00% (other detectors) |
| Injection Carry Over (HSS only) | Same as Injection Precision | Area carry over ≤ 1.00% |

## Test Design and Rationale

### Overview

Many GMP/GLP enforcement agency inspectors now ask firms to provide a risk assessment of their equipment and computer systems plus a science-based rationale for subsequent validation and qualification testing.

GENERAL RISK STATEMENT: Any laboratory chemical system used for raw material testing or final drug product / medical device testing in GMP or used in formal GLP studies will likely fall into a HIGH RISK category. This risk assessment will imply the need for IQ & OQ & on-going qualification. ANY USER SPECIFIC RISK ANALYSIS SUPERCEDES THIS GENERAL RISK STATEMENT.

The rest of this section outlines the science-based rationale for each test in the Agilent hardware OQ plus a brief test design and procedure description.

The recommended set of hardware OQ tests described in this EQP derives from Agilent's interpretation of FDA, USP, and GAMP guidelines and other authoritative expert literature.

OQ test design incorporates both modular and holistic testing, which is a proven and regulatory acceptable approach. When applicable, direct metrology is used to test pump flow rates and thermal-controlled column compartments, for example. Holistic chemical testing is used to evaluate critical instrument characteristics

When applicable, certified reference standards and calibrated equipment are used.

Considering the number of setpoints, parameters, and conditions of each recommended OQ test, the proven concepts of worst case, range, and representative have been applied. If a property or characteristic is known to have its worst performance at one end of a range of use, this is the setpoint that should be tested and other setpoints are not required. If a property or characteristic has no known worst case, testing at the high and low points of the range of use is required. If there are too many possible use cases and conditions to realistically test (and none is a worst case), a representative sample for test is the best approach.

#### System Inspection and Basic Safety and Operation

Description: System must be in safe and operational condition before starting the OQ tests.

Procedure: The instrument is given a general inspection and its basic safety features are challenged to ensure proper operation.

#### GC Oven Temperature Accuracy and Stability

Description: Oven temperature accuracy is important for comparability between systems and transferring methods. Oven temperature stability is critical for qualitative and quantitative analysis.

Procedure: At two different temperatures, accuracy is measured using an external calibrated thermometer and expressed as the difference between found and setpoint values. At one of these, a statistically significant number of additional readings are taken during the total duration of the test and stability is expressed as the delta between the highest and lowest temperatures.

#### Headspace Leak

Description: Proper operation of the valves is critical for repeatable peak areas and carry over.

Procedure: This test verifies that the valves operate properly with no excessive leaks or restricted internal flow paths.

#### Headspace Vent and Pressurization Valve Integrity

Description: Proper operation of the valves is critical for repeatable peak areas and carry over.

Procedure: This test verifies that the valves operate properly: with no excessive leaks or restricted internal flow paths.

### Headspace Heated Zones Temperature Accuracy

Description: Temperature accuracy of the heated zones is important for comparing systems and transferring methods. Oven accuracy is critical to quantitative headspace methods.

Procedure: The temperature is measured using an external calibrated thermometer with appropriate probe design. Accuracy is determined as the difference between found and setpoint values.

### Vial Heater Temperature Accuracy

Description: The 7693A vial heater option can be used during sample preparation. This test verifies that it heats accurately.

Procedure: The heater temperature is measured with an external thermometer and accuracy is calculated as the difference between the measured value and setpoint.

### Inlet Pressure Decay

Description: Inlet pressure integrity is critical for repeatable injection and retention times. The pressure decay and pressure accuracy tests combine to demonstrate pressure integrity.

Procedure: Inlet is capped, a pressure applied, and inlet flow turned off. The pressure decay is recorded over a specified time range.

### Inlet Pressure Accuracy

Description: Inlet pressure integrity is critical for repeatable injection and retention times. The pressure decay and pressure accuracy tests combine to demonstrate pressure integrity. This test checks for accurate pressure to the head of the column. Column flow is achieved by maintaining a constant pressure against a known restriction. Because the restriction is a function of the column geometry, measuring pressure in the inlet is the most accurate way to determine flow.

Procedure: The inlet is capped, a pressure is applied, and the inlet pressure is recorded using an external calibrated manometer connected to the inlet.

### Inlet Flow Stability

Description: Inlet flow stability is critical for repeatable injection and retention times. Inlet flow accuracy and precision tests combine to demonstrate inlet flow stability.

Procedure: Column flow setpoint is achieved, all detector flows are turned off, and calculations are made: flow accuracy as the absolute % difference of the mean of the ten flow readings and the setpoint; flow precision as the % RSD of ten flow readings.

### Detector Flow Accuracy

Description: Detector flow accuracy is critical for a stable detector signal. Incorrect flows may have an impact on detector performance.

Procedure: Flow accuracy is determined by measuring the flows with a calibrated mass flowmeter and then comparing the results to the test setpoints and the values displayed by the GC.

### Noise and Drift

Description: This test gives an indication of detector sensitivity and stability.

Procedure: The signal is monitored at specified conditions appropriate to the type of detector over a twenty-minute period. The signal noise is calculated based on ASTM E594-96 as the average peak-to-peak noise in a number of signal segments.

The drift is calculated as the slope of the linear regression for the signal. Detector type and the gases used all contribute to different performance and therefore different limits for each configuration.

### Scouting Run

Description: This test is used to determine the chromatogram for presence of expected peaks, sufficient run time, and proper integration events prior to the start of the actual qualification runs.

### Signal to Noise

Description: Sensitivity of GC detection is a critical performance feature in quantitative and qualitative analysis. A signal-to-noise value of a representative compound at known concentration provides sensitivity statistics.

Procedure: A traceable standard is injected and signal to noise is calculated.

### Injection Precision

Description: System precision is critical for quantitative analysis.

Procedure: An initial stabilizing injection is made, followed by six repeat injections of a traceable standard followed by a final blank injection. The % RSD of the six injections is calculated to provide precision statistics. There are separate dedicated instrument parameters and reference standards applicable to each inlet/detector combination. This test is performed with liquid and headspace sampler configurations.

### Injection Carry Over

Description: Low carry over from a previous injection is critical for accuracy of quantitative and reliability of qualitative analysis. For headspace samplers, the engineering condition contributes to carry over performance, so this is a core OQ test for these samplers.

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 are available that can eliminate carry over. These are user selectable and may be application specific. The condition of the injection syringe is the only controllable engineering factor. The injection syringe is typically replaced for new 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: 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.

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