ThermoFisher
SCIENTIFIC

Title: OQ Chromeleon GCMS ISQ TSQ Series

Number: ULS-RPG-QM-4245 Revision: 019

Qualification #: 50559

Effective Date: 25 Jul 2024

CHANGE SUMMARY

(Only changes within the last 18 months are listed below.)

Revision Number	Effective Date	Description of Changes
019	See coversheet	• Added RPG Reports 3 Support. • Added support for TriPlus RSH SMART VOC Sample Prep Station. • Appendix A: Added for TriPlus RSH VOC Workflow Repeatability test. • Section 5.2 and 8.1.3: Added alternative to micropipette. • Section 6.1: MS AEI Repeatability renamed MS AEI Checkout. • Section 7.1: Added instructions for using RPG Reports 2 and 3. • Section 9.3: Added instructions for reporting data in RPG Reports 2 and 3. • Sections 10-15: Updated sections to match latest RPG qualification document template.
018	12 Mar 2024	• Removed reference to TSQ 9000 and ISQ 7000 on Section 6.1
017	28 Nov 2023	• Clarified TriPlus LS Autosampler Repeatability with MS OQ Limit is the same as other LS models (6.0% RSD). • Added Peak of Interest for HS, SPME, ITEX injections (Ethanol).

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OQ CHROMELEON GCMS ISQ AND TSQ SERIES

1 INTRODUCTION

Documented evidence must be provided to demonstrate the integrity of data collected and validate the results obtained on laboratory instrumentation.

Many laboratories achieve this by formal quality systems, which are generally implemented in accordance with one or more of the following recognized quality standards and other quality guidelines:

- Good Laboratory Practice (GLP)
- Current Good Manufacturing Practice (cGMP)
- ISO guidelines
- USP / EP

These standards and guidelines are written in broad terms, to make them as widely applicable as possible. All stipulate general requirements specifying instruments must be fit for purpose, properly maintained, and calibrated to national or international standards. This procedure used for Thermo Fisher Scientific Operational Qualification is adapted to these standards.

This procedure is intended to guide trained and certified individuals through Operational Qualification of Thermo Scientific products.

2 CDS DATA INTEGRITY AND GXP RESPONSIBILITY

The Instrument Testing is undertaken in the customer environment; this will utilize the customer CDS software package. Implementation of user management and data auditing may have been applied to the system and is the responsibility of the system owner. An appropriate user account may be required and all actions in the CDS can be recorded by the CDS depending on the audit settings applied. It is the responsibility of the Thermo Fisher Scientific representative to ensure that all data collected is identified using appropriate naming, saved location, and recorded in the RPG Reports UI or other provided checklist. A discussion with the customer and their quality department may be necessary to ensure that any data is named, stored, and handled appropriately.

All work carried out should comply to GxP Good Documentation Practice as followed by Thermo Fisher Scientific and the customer organization.

2.1 Performing the Qual (MS Documents)

NOTE: This may require test optimization injections to be run either directly using a syringe or infusion prior to carrying out the Operational Qualification. This optimization can be undertaken in the MS Tune program or the CDS directly and must be identified by name and recorded in the appropriate comment section of any checklist or RPG Reports UI.

3 OPERATIONAL QUALIFICATION SCOPE

A hardware operational qualification (OQ) is documented verification that a system operates according to written and preapproved limits throughout all specified operating ranges.

Performing an OQ is recommended at any of the following occasions:

- After initial installation of the instrument
- When a new module is added to an existing instrument
- Following a firmware update

- Moving instruments to a new location
- After replacing a non-consumable hardware component that has a serial number

OQ documents the following items:

- **Qualification Service Representative Information**
- **Customer Information**
- **System Information:** Instrumentation, computer, and software information as installed.
- **OQ Limits:** List of manufacturer-recommended limits for ensuring that the system is operating as expected.
- **Operational Tests and Results:** Testing of important functions to verify that the system operates as intended by the manufacturer and as required by the user. This is a group of selected, important parameters for testing according to the intended use of the system.

This procedure supports the following Thermo Scientific hardware/software configurations:

- **GC:** Trace 1300, Trace 1310, Trace 1600, and Trace 1610
- **Autosampler:** AI/AS 1310/1610/3000 Series, TriPlus Classic, TriPlus 100LS, TriPlus 300, TriPlus RSH series (TriPlus RSH, TriPlus RSH SMART Advanced, TriPlus RSH SMART Standard, TriPlus RSH SMART VOC sample prep station), and TriPlus 500
  - **Injection Modes**: Liquid, Headspace, SPME, SPME Arrow, ITEX
- **MS:** ISQ Series (ISQ, ISQ LT, ISQ QD, ISQ 7000, ISQ 7610) and TSQ Series (TSQ, TSQ DUO, TSQ EVO, TSQ 8000, TSQ 9000, TSQ 9610)
- **Probes:** Direct Exposure Probe (DEP), Direct Insertion Probe (DIP)
- **Controlling Software:** Chromeleon 7.2.3 or later version
- **RPG Reports:** 2.x or 3.4.x or later with GCMS Systems license

4 RECOMMENDED DOCUMENTS

Use this document along with the gas chromatograph and mass spectrometer operator manual, Installation Procedure, and individual component operator/service manuals.

5 RECOMMENDED MATERIALS

5.1 Common Materials

- TG-SQC 15 m x 0.25 mm x 0.25 µm Test Column (P/N 26070-1300)
- GC/MS Test Kit (P/N 1R120150-PERF) – ISQ Only
- GC/MS Test Kit (P/N 1R120540-PERF) – TSQ Only
- GC/MS Test Kit containing 100 pg OFN (P/N 1R120150-IQOQ)
- GC/MS Test Kit containing 5 fg OFN (P/N 1R76310-0105)
- 2-mL screw cap autosampler vials, caps, and septa
- Disposable transfer pipettes
- Suitable blanking solvent (e.g., iso-octane)
- Deionized Water (for TriPlus RSH SMART VOC sample prep station)

5.2 Additional materials for Headspace Sampler Injections

- TriPlus Headspace Test Mix (P/N 33819024)
  - 20-mL sample vials and 20-mm caps with silicon-Teflon septa
  - 5 µL disposable micropipettes (P/N 36520056) or 5 µL syringe (TriPlus 500 Only)

5.3 Additional materials for SPME Sampler Injections

- All Headspace injection materials
- Kit Supelco SPME (P/N 25401080)

5.4 Additional materials for ITEX Sampler Injections

- TriPlus Headspace Test Mix (P/N 33819024)
- 5 µL disposable micropipettes (P/N 36520056)

5.5 Additional materials for Direct Probes

- Solids Probe Test Mix (P/N 1R120339-0001)

6 OQ TEST SUMMARY

6.1 MS Tests

MS Sensitivity			
The MS Sensitivity Test verifies the response/sensitivity of the detector by calculating the signal-to-noise ratio.			
How Test is Performed			
A standard is injected two times in each EI and CI modes. The signal-to-noise ratio of the target peak is calculated for each injection. The calculated results are compared to the set limits. If there is a second inlet devoted to the MS, this test is then performed using that inlet as well.			
Module	**Test Name**	**Test Point / Standard**	**OQ Limits**
TSQ Series MS	MS Sensitivity - EI Mode (SRM)	Signal-to-Noise of two 1-µL injections of 100 fg/µL OFN	Helium Carrier ≥ 5000:1
	MS Sensitivity - PCI Mode (SRM)	Signal-to-Noise of two 1-µL injections of 5 pg/µL BZP	Helium Carrier ≥ 2500:1
ISQ Series MS	MS Sensitivity - EI Mode (Full Scan)	Signal-to-Noise of two 1-µL injections of 1 pg/µL OFN	Helium Carrier ≥ 450:1 Hydrogen Carrier ≥ 50:1
	MS Sensitivity - PCI Mode (Full Scan)	Signal-to-Noise of two 1-µL injections of 100 pg/µL BZP	Helium Carrier ≥ 300:1

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MS AEI Checkout			
The MS AEI Checkout Test verifies the response repeatability of the detector by calculating the percent difference between two injections. This test also checks detector sensitivity.			
How Test is Performed			
A standard is injected two times in AEI mode. The percentage difference of area of the target peak of each injection is calculated for the set of injections. The calculated results are compared to the set limits.			
Module	**Test Name**	**Test Point / Standard**	**OQ Limits**
TSQ Series MS (AEI Model Only)	MS AEI Checkout (SRM)	Repeatability of two 1-µL injections of 5 fg/µL OFN	Helium Carrier Minimum Peak Area ≥ 1350 Peak Area Difference < 20%
ISQ Series MS (AEI Model Only)	MS AEI Checkout (Full Scan)	Repeatability of two 2-µL injections of 5 fg/µL OFN	Helium Carrier Minimum Peak Area ≥ 1800 Peak Area Difference < 20%

MS Direct Probe Verification			
The MS Direct Probe test verifies the DEP/DIP probe is functioning properly.			
How Test is Performed			
1.0 µL of a solids probe test mix is analyzed and the presence of cholesterol is identified through the NIST search libraries.			
Module	**Test Name**	**Test Point**	**OQ Limits**
Direct Insertion Probe (DIP)	MS Direct Probe Verification	Contents of solids probe test mix	2 of top 3 search results match cholesterol
Direct Exposure Probe (DEP)			

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6.2 Oven Tests

The GC Oven is typically qualified under a separate model specific or GC system OQ protocol. However, the following oven tests may need to be performed as part of the MS testing if no GC detectors are present.

Oven Temperature Ramp Repeatability			
The Oven Temperature Ramp Repeatability test demonstrates the holistic repeatability of GC conditions (temperature and gas flow) during analysis by injecting the same sample multiple times and evaluating the relative standard deviation of the retention times. Variations in the gas flow and oven temperature deviations affect primarily the repeatability of the retention time.			
How Test is Performed			
This test is a subset of the Sampler Repeatability test. The carrier gas flow and oven temperature ramp is specified in the method. The relative standard deviation of the retention times of the six injections indicates repeatability of the system temperature ramp.			
Module	**Test Name**	**Test Point**	**OQ Limits**
GC Oven	Oven Temperature Ramp Repeatability	Retention Time of 6 Repeatability Injections	≤ 1.0% RSD

6.3 Autosampler Tests

The GC Autosampler Tests are typically qualified under a separate model specific or GC system OQ protocol. However, the following oven tests may need to be performed as part of the MS testing if no GC detectors are present.

Repeatability with MS			
The repeatability test demonstrates that system injections are precise and consistent over a series. The relative standard deviation (%RSD) of sample peak area characterizes the repeatability of the injection volume.			
How Test is Performed			
A standard is injected multiple times (minimum six injections) and data are collected. The relative standard deviation (%RSD) of the peak areas is calculated. The %RSD is then compared to the set limits.			
Module	**Test Name**	**Test Point / Standard**	**OQ Limits**
AI/AS Series	Repeatability for Liquid Injections	Area of 6 Standard Injections	≤ 6.0% RSD
TriPlus Series	Repeatability for Liquid Injections		
TriPlus Series*	Repeatability for Headspace, SPME, SPME Arrow and ITEX Injections		≤ 10.0% RSD

* See Appendix A for Repeatability of the TriPlus RSH SMART w/ VOC Sample Prep Station and LS/HS sampler.

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7 PREPARING THE SYSTEM FOR OPERATIONAL QUALIFICATION

Prior to performing the Operational Qualification, completion of an Installation Qualification and/or preventive maintenance routine is highly recommended. This should include:

- Gas Chromatograph
  - Installation Qualification (for new installations)
  - Preventive Maintenance (for existing instruments)
- Autosampler
  - Installation Qualification (for new installations)
  - Preventive Maintenance (for existing instruments)
  - Operational Qualification (only if a GC detector is installed, it is preferable that it is used to qualify the autosampler)
- Mass Spectrometer
  - Installation Qualification (for new installations)
  - Preventive Maintenance (for existing instruments)
  - Tuning for the applicable ionization modes

7.1 Reporting Interface

All data and chromatograms for Qualification test results will be summarized in test reports against OQ limits of tolerance. Test reports will be generated using the OQ reporting user interface (RPG Reports) to discern the outcome of each test. In most cases, the project will have already been started with the IQ.

7.1.1 RPG REPORTS 2.X:

1. Open the RPG Reports UI and start a new project (an GCM Systems license must be used for the project).
2. In **Section 1, Settings** configure the following:
   a. Qualification Type: **GCMS Systems**
   b. Qualification Control Software: **Chromeleon**
3. In **Section 1,** choose the applicable instrument models and complete the rest of the configuration fields.
4. Tests will now be visible in **Section 4: OQ**.

7.1.2 RPG REPORTS 3.X:

1. Open the RPG Reports UI and start a new project (an GCMS Systems license must be activated for the project).
2. The following **Settings** are required before you can proceed:
   a. Group: ***Select applicable TFS Group (e.g., Analytical Instrument Group)***
   b. Service: **IQOQ** or **OQ**
   c. Hardware Configuration: **GC**
   d. Control Software: **Chromeleon** ***& software version (e.g., 7.3)***
3. On the **Settings** screen, you may now specify the components of the system to be tested by moving them from the **Available Modules** to the **Selected Modules** with the arrow button.

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Note: Instrument PC, GC Oven, Inlets, the MS, Samplers, and RSH Tools are all treated as individual Modules for testing.

4. Click the **Next** button to advance.
5. This will create a report page section for each selected module (with a numeric identifier in case more than one of the same instrument are added).
6. The individual module details are then completed, and once **Powered On Successfully?** is completed, tests can be selected per each module.

   Note: Recommended tests will already be checked, but this can be modified, multiple copies of a test can also be added (they will receive a similar numeric identifier to allow for adding the additional tests).
7. Selected test pages will be added under the instrument module.
8. **Test Equipment and Standards** and **Protocols** may be identified at this time on the applicable report pages, and any electronic files attached.
9. The project should be saved before then proceeding with testing.

7.2 Chromeleon eWorkflows

Automated tests are run using a pre-defined Chromeleon eWorkflow file. The file contains sequence and processing templates as well as built-in reports for generating chromatograms and exportable data.

7.3 Instrument Method Parameters

Instrument method parameters for the tests can be found in a method reference file. There is a method reference tab for standard injections. Recommended parameters may be modified as needed to be appropriate for the instrument configuration.

7.4 Importing the eWorkflow

1. From within the RPG Reports UI, select the appropriate eWorkflow (i.e., **OQ for GCMS EI Sensitivity.ewfx**). This will start the import process within Chromeleon.
2. When the eWorkflow import wizard opens, choose the desired Destination data vault, and click **Start**.
3. From the eWorkflow tab select the imported workflow from the left-hand column and the instrument to be tested from the right hand column; then click **Launch**.
4. When the **Method** window opens, click **Next**.
5. When the **Sampler Configuration** window opens, click **Finish**.
6. Browse to the appropriate location to save the sequence file and click **Finish** (if the name is already in use, Chromeleon will enumerate the sequence name).

   NOTE: Be sure not to run the sequence at this time.
7. Open the **Custom Sequence Variables** tab at the bottom of the sequence page.
8. Manually enter the Qualification Number.
9. Save and close the sequence.
10. Repeat these steps for all MS modes being tested.

7.5 Creating Instrument Methods

1. Create a **Standard** Instrument Method.
   a. Open the appropriate OQ sequence.
   b. Select Create and Instrument Method.

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c. Click on **Method Reference File** (Section3: FILES in the RPG Reports UI), set the instrument parameters according to the compound tab.

d. Save this instrument method as the target compound and MS mode name (i.e., **OFN EI**).

2. Repeat the previous step for all sequences imported.
3. In the sequence, assign the appropriate method to each sample injection.
4. Create additional methods for additional detectors and/or sampler modes as needed.

8 PERFORMING THE QUALIFICATION

8.1 EI Mode MS Sensitivity Tests

8.1.1 SAMPLE PREPARATION TSQ SERIES – LIQUID SAMPLES

1. Add 1.0 mL of a suitable solvent blank to an autosampler vial and label it **Blank**.
2. Transfer the contents of the 100 fg/µL OFN from the test kit into a 2.0 mL screw top vial or similar vessel.
3. Transfer the contents of the 100 pg/µL OFN from the test kit into a 2.0 mL screw top vial or similar vessel.
4. Transfer the contents of the 5 fg/µL BZP from the test kit into a 2.0 mL screw top vial or similar vessel.

8.1.2 SAMPLE PREPARATION ISQ SERIES – LIQUID SAMPLES

1. Add 1.0 mL of a suitable solvent blank to an autosampler vial and label it **Blank**.
2. Transfer the contents of the 1-pg/µL OFN from the test kit into a 2.0-mL screw top vial or similar vessel.
3. Transfer the contents of the 100-pg/µL OFN from the test kit into a 2.0-mL screw top vial or similar vessel.
4. Transfer the contents of the 100-pg/µL BZP from the test kit into a 2.0-mL screw top vial or similar vessel.
5. Transfer the contents of the 100-fg/µL OFN from the test kit into a 2.0-mL screw top vial or similar vessel.

Vial Label TSQ	Vial Label ISQ	Position	Sample	Test
Blank	Blank	1	Iso-Octane or suitable blanking solvent	Blank
100 fg/µL OFN	1 pg/µL OFN	2	Octafluoronaphthalene in iso-Octane	EI Sensitivity
100 pg/µL OFN	100 pg/µL OFN	3		Repeatability (liquid)
5 pg/µL BZP	100 pg/µL BZP	4	Benzophenone	PCI Sensitivity
5 fg/µL OFN	5 fg/µL OFN	5	Octafluoronaphthalene in iso-Octane	AEI Checkout

NOTE: The sample table above is for preparing all mode sensitivity and liquid repeatability tests (as applicable).

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8.1.3 SAMPLE PREPARATION – HEADSPACE SAMPLES

1. Prepare six 20-mL sample vials and one blank.
   a. For TriPlus 500:
      i. Using a 5 µL disposable micropipette (PN 36520056), dip to pipette into the TriPlus Headspace Test Mix (P/N 33819024), allow the micropipette to fill completely.

         Note: The samples can also be prepared by using a suitable micro syringe to transfer the 5 µL of the standard.

      ii. Place the entire micropipette filled with TriPlus Headspace Test Mix into the first 20-mL empty vial and cap the vial.
   b. For all other Headspace samplers:
      i. Draw 2 µL of Headspace Test Mix (P/N 33819024) into the syringe barrel followed by a small air gap (ensure that the syringe needle is empty).
      ii. Insert the syringe needle into the first 20-mL empty vial.
      iii. Inject the test mix into the vial, but make sure that the end of the needle touches the internal wall of the vial. Cap each vial after preparation.
2. Repeat these steps for the remaining sample vials.
3. Situate the seven vials loaded with test mix into the sampler tray positions listed below:

Vial Label	Position	Sample	Test
Blank	7	Empty	Blank
HS Test Mix	8	Ethanol	HS Repeatability
	9		
	10		
	11		
	12		
	13		

8.1.4 SAMPLE PREPARATION– SPME/SPME ARROW SAMPLES

1. Prepare six 20-mL sample vials and one blank.
2. Draw 2 µL of test mix into the syringe barrel followed by a small air gap (ensure that the syringe needle is empty).
3. Insert the syringe needle into the first 20-mL empty vial.
4. Inject the test mix into the vial; ensure that the end of the needle touches the internal wall of the vial. Cap each vial after preparation.
5. Repeat these steps for the remaining sample vials.

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6. Situate the seven vials loaded with test mix into the sampler tray positions listed below:

Vial Label	Position	Sample	Test
Blank	7	Empty	Blank
SPME Test Mix	8	Ethanol	SPME Repeatability
	9		
	10		
	11		
	12		
	13		

8.1.5 SAMPLE PREPARATION – ITEX SAMPLES

1. Prepare six 20-mL sample vials and one blank.
2. Cap the blank vial and place it into the autosampler tray.
3. Use a 5-uL disposable micropipette (PN 36520056): dip the pipette tip into the test solution and allow the micropipette to fill completely.
4. Place the entire micropipette filled with the test solution into an empty vial and cap the vial.
5. Repeat steps 3-4 for all remaining sample vials.
6. Situate the seven vials loaded with test mix into the sampler tray positions listed below:

Vial Label	Position	Sample	Test
Blank	7	Empty	Blank
ITEX Test Mix	8	Ethanol	ITEX Repeatability
	9		
	10		
	11		
	12		
	13		

8.2 Preparing the System

8.2.1 SYSTEM SETUP FOR EI SENSITIVITY

1. In **Chromeleon**, shut down the mass spectrometer via the status window.
2. Verify that the **source** and **transfer line temperatures** have cooled to **below 150 °C**.
3. De-energize the mass spectrometer.
4. Manually open the vent valve.
5. Perform a column evaluation and verify that it completes successfully.
6. Install the test column and plumb outlet to the mass spectrometer.
7. Close the vent valve.
8. Re-energize the mass spectrometer.

9. Set **source temperature** to **200 °C** and **transfer line temperature** to **250 °C**.
10. Allow the system to properly stabilize.
11. If using an autosampler for injection, ensure that the samples have been loaded in the tray.
12. Ensure that the **EI** ion volume has been installed and that the **EI Full Autotune** has been performed.
13. Verify that the proper vacuum level has been achieved.
14. Open **Chromeleon** and verify that the mass spectrometer is **Ready** in the status window.

8.2.2 PERFORMING THE EI SENSITIVITY TESTS

1. Open the EI Sensitivity sequence in Chromeleon (i.e., **OQ for GCMS EI Sensitivity**).
2. Verify that the sequence has been set up for one 1-µL blank injection and two 1-µL sensitivity injections of the Test Mix.
3. Verify that the vial position is correct for each injection and save the sequence.
4. Save the sequence and then start the sequence.
5. The sequence will begin, and the autosampler will perform the injections. If no autosampler is installed, the system will await the manual injection process.
    a. Manual injection process:
        i. Inject 1 µL (plus needle volume) of Blank and press **Start**.
        ii. Inject 1 µL (plus needle volume) of OFN sensitivity standard and press **Start**.
        iii. Allow acquisition to complete (~18 min).
        iv. Repeat for a total of two injections.
6. When the acquisition has completed, double-click on the samples to view the data in Chromeleon Studio.
7. If necessary, edit the integration parameters in the processing method to optimize for the OFN peak detection.
8. From the Chromeleon studio, select each sample, right-click and choose, **Export**, select **Raw file format (*.raw)** and click **OK**.
9. Open the Signal-to-Noise Test application (Start > All Programs > Thermo Xcalibur > Signal to Noise Test).
10. Disregard the first raw file of the batch, which is for the blank injection; using the Signal-to Noise-Test application, open the second raw file of the batch **EI_SN**, which is for the first of the two actual sample injections.

NOTE: If the application does not recognize the peak, change the detection drop-down from Automatic to the appropriate setting.

11. Transcribe the Signal-to-Noise (RMS) value into the corresponding field of the RPG Reports UI.
12. Using the Evaluate / Run Test function, run the RPG report.
13. Repeat this process for the second sample injection in the batch.

8.2.3 SECONDARY INLET

If the system has an additional inlet devoted to the mass spectrometer, it is recommended that the Sensitivity test be repeated on that inlet.

8.2.4 SYSTEM SETUP FOR AEI CHECKOUT

1. In **Chromeleon**, shut down the mass spectrometer via the status window.

2. Verify that the **source** and **transfer line temperatures** have cooled to **below 150 °C**.
3. De-energize the mass spectrometer.
4. Manually open the vent valve.
5. Perform a column evaluation and verify that it completes successfully.
6. Install the test column and plumb outlet to the mass spectrometer.
7. Close the vent valve.
8. Re-energize the mass spectrometer.
9. Set **source temperature** to **200 °C** and **transfer line temperature** to **250 °C**.
10. Allow the system to properly stabilize.
11. If using an autosampler for injection, ensure that the samples have been loaded in the tray.
12. Ensure that the A**EI** ion volume has been installed and that the A**EI Full Autotune** has been performed.
13. Verify that the proper vacuum level has been achieved.
14. Open **Chromeleon** and verify that the mass spectrometer is **Ready** in the status window.

8.2.5 PERFORMING THE AEI CHECKOUT TESTS

1. Open the AEI Checkout sequence in Chromeleon (i.e., **OQ for GCMS AEI Checkout**).
2. Verify that the sequence has been set up for one 1-µL blank injection and two repeatability injections of the Test Mix.
3. Verify that the vial position is correct for each injection and save the sequence.
4. Save the sequence and then start the sequence.
5. The sequence will begin, and the autosampler will perform the injections. If no autosampler is installed, the system will await the manual injection process.
6. When the acquisition has completed, double-click on the samples to view the data in Chromeleon Studio.
7. If necessary, edit the integration parameters in the processing method to optimize for the OFN peak detection.
8. Repeat the above step for both sensitivity injections and processing methods (note that there are individual processing methods for each injection).
9. Open the Chromeleon report in the **Report Designer** view.
10. Click on the appropriate **EI Sensitivity** tab of the report.
11. Verify that the results for the test have been properly generated.
12. If necessary, update the report header information.
13. From the **Chromeleon** button in the upper left-hand corner, select **Print**.
14. Transcribe the displayed Signal-to-Noise value from each injection into the corresponding field of the RPG Reports UI.
15. Attach the printed report to the qualification binder.

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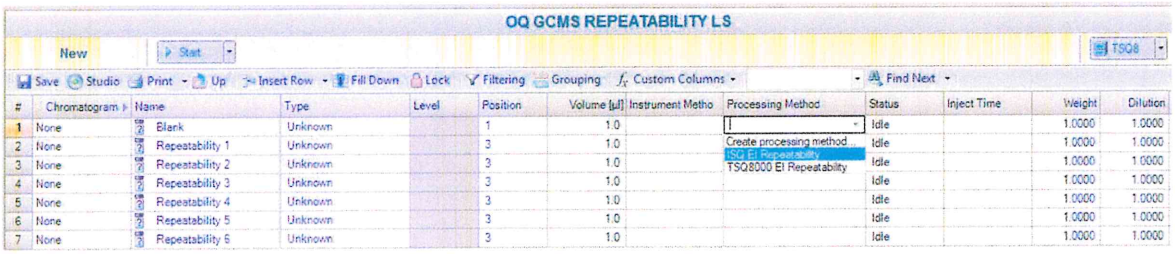
8.3 Performing the Liquid Sample / Oven Temperature Ramp Repeatability Test (with MS)

NOTE: This test is not necessary if Repeatability has previously been performed using a GC detector

It is best practice to ensure that a blank injection is made prior to injecting any samples.

1. Open the Repeatability sequence in Chromeleon (i.e., **OQ for GCMS LS Repeatability**).
2. In the **Processing Method** column of the displayed sequence, choose the appropriate processing method from the given list for all samples (the sequence will be pre-populated with a method for each supported mass spectrometer type).



#	Chromatogram	Name	Type	Level	Position	Volume [µl]	Instrument Metho	Processing Method	Status	Inject Time	Weight	Dilution
1	None	Blank	Unknown		1	1.0			Idle		1.0000	1.0000
2	None	Repeatability 1	Unknown		3	1.0			Idle		1.0000	1.0000
3	None	Repeatability 2	Unknown		3	1.0			Idle		1.0000	1.0000
4	None	Repeatability 3	Unknown		3	1.0			Idle		1.0000	1.0000
5	None	Repeatability 4	Unknown		3	1.0			Idle		1.0000	1.0000
6	None	Repeatability 5	Unknown		3	1.0			Idle		1.0000	1.0000
7	None	Repeatability 6	Unknown		3	1.0			Idle		1.0000	1.0000

3. Verify that the sequence is set up for one 1-µL blank injection and six 1-µL repeatability injections of the Test Mix.
4. Verify that the vial position is correct for each injection and save the sequence.
5. Save the sequence.

8.3.1 RUNNING THE SEQUENCES

1. Select the appropriate sequence and add it to the instrument queue.
2. Open the **Instrument View** window and display the **Queue** tab. As soon as the queue has started, Chromeleon will run the sequence.
3. When acquisition has completed, double-click on the samples to view the data in the **Chromeleon Studio**.
4. If necessary, edit the integration parameters in the processing method to optimize for the OFN peak detection.
5. Repeat these steps for any additional liquid samplers, if installed.

8.4 Performing the Headspace Sample / Oven Temperature Ramp Repeatability Test (with MS)

NOTE: This test is not necessary if Repeatability has previously been performed using a GC detector

It is best practice to ensure that a blank injection is made prior to injecting any samples.

1. Open the Repeatability sequence in Chromeleon (i.e., **OQ for GCMS HS Repeatability**).
2. In the **Processing Method** column of the displayed sequence, choose the appropriate processing method from the given list for all samples (the sequence will be pre-populated with a method for each supported mass spectrometer type).
3. Verify that the sequence is set up for one 1-µL blank injection and six 1-µL repeatability injections of the Test Mix.
4. Verify that the vial position is correct for each injection and save the sequence.
5. Save the sequence.

8.4.1 RUNNING THE SEQUENCES

1. Select the appropriate sequence and add it to the instrument queue.
2. Open the **Instrument View** window and display the **Queue** tab. As soon as the queue has started, Chromeleon will run the sequence.
3. When acquisition has completed, double-click on the samples to view the data in the **Chromeleon Studio**.
4. If necessary, edit the integration parameters in the processing method to optimize for the peak of interest.

8.5 Performing the SPME/SPME Arrow Sample / Oven Temperature Ramp Repeatability Test (with MS)

NOTE: This test is not necessary if Repeatability has previously been performed using a GC detector.

It is best practice to ensure that a blank injection is made prior to injecting any samples.

1. Open the Repeatability sequence in Chromeleon (i.e., **OQ for GCMS SPME Repeatability**).
2. In the **Processing Method** column of the displayed sequence, choose the appropriate processing method from the given list for all samples (the sequence will be pre-populated with a method for each supported mass spectrometer type).
3. Verify that the sequence is set up for one 1-µL blank injection and six 1-µL repeatability injections of the Test Mix.
4. Verify that the vial position is correct for each injection and save the sequence.
5. Save the sequence.

8.5.1 RUNNING THE SEQUENCES

1. Select the appropriate sequence and add it to the instrument queue.
2. Open the **Instrument View** window and display the **Queue** tab. As soon as the queue has started, Chromeleon will run the sequence.
3. When acquisition has completed, double-click on the samples to view the data in the **Chromeleon Studio**.
4. If necessary, edit the integration parameters in the processing method to optimize for the peak of interest.

8.6 Performing the ITEX Sample / Oven Temperature Ramp Repeatability Test (with MS)

NOTE: This test is not necessary if Repeatability has previously been performed using a GC detector.

It is best practice to ensure that a blank injection is made prior to injecting any samples.

1. Open the Repeatability sequence in Chromeleon (i.e., **OQ for GCMS ITEX Repeatability**).
2. In the **Processing Method** column of the displayed sequence, choose the appropriate processing method from the given list for all samples (the sequence will be pre-populated with a method for each supported mass spectrometer type).
3. Verify that the sequence is set up for one 1000-µL blank injection and six 1000-µL repeatability injections of the Test Mix.
4. Verify that the vial position is correct for each injection and save the sequence.
5. Save the sequence.

8.6.1 RUNNING THE SEQUENCES

1. Select the appropriate sequence and add it to the instrument queue.
2. Open the **Instrument View** window and display the **Queue** tab. As soon as the queue has started, Chromeleon will run the sequence.
3. When acquisition has completed, double-click on the samples to view the data in the **Chromeleon Studio**.
4. If necessary, edit the integration parameters in the processing method to optimize for the peak of interest.

8.7 MS Direct Probe Verification

NOTE: NIST Library must be installed prior to running this test.

1. Ensure that the **EI** ion volume and probe ion volume holder are installed into the source assembly.
2. Open the Direct Probe Verification sequence in Chromeleon (i.e., **OQ for GCMS HS Direct Probe**).
3. Program the probe controller method, as described in the Direct Probe User Guide (e.g., p/n 119327-0001 *for DIP*).
4. Open **Chromeleon**. Modify the **MS** method end run to indicate probe run time.
5. On the front panel of the probe controller, modify the probe controller method to match the parameters indicated in the Direct Probe User Guide.
6. Prepare a sequence to analyze approximately 1 µL of probe test mix.
7. Start the sequence and wait for the mass spectrometer to be **Waiting for Contact Closure**.
8. Press the **Start** button on the probe controller to initiate the scan.
9. When the acquisition has completed, double-click on the samples to view the data in the **Chromeleon Studio**.

8.8 PCI Mode MS Sensitivity Tests

8.8.1 SYSTEM SETUP FOR PCI SENSITIVITY

1. Ensure that the CI ion volume has been installed and that the **CI + Autotune** has been performed.
2. Open **Chromeleon** and verify that the mass spectrometer is **Ready** in the status window.

8.8.2 PERFORMING THE PCI SENSITIVITY TESTS

1. Open the CI Sensitivity sequence in Chromeleon (i.e., **OQ for GCMS PCI Sensitivity**
2. Verify that the sequence has been set up for one 1-µL blank injection and two 1-µL sensitivity injections of the Test Mix.
3. Verify that the vial position is correct for each injection and save the sequence.
4. Save the sequence and then start the sequence.
5. The sequence will begin, and the autosampler will perform the injections. If no autosampler is installed, the system will await the manual injection process.
    a. Manual injection process:
        i. Inject 1 µL (plus needle volume) of Blank and press **Start**.
        ii. Inject 1 µL (plus needle volume) of PCI sensitivity standard and press **Start**.
        iii. Allow acquisition to complete (~18 min).

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iv. Repeat for a total of two injections.

6. When the acquisition has completed, double-click on the samples to view the data in the Chromeleon Studio.
7. If necessary, edit the integration parameters in the processing method to optimize for the PCI peak detection.
8. From the Chromeleon studio, select each sample, right-click and choose, **Export**, select **Raw file format (*.raw)** and click **OK**.
9. Open the Signal-to-Noise Test application (Start > All Programs > Thermo Xcalibur > Signal to Noise Test).
10. Disregard the first raw file of the batch, which is for the blank injection; using the Signal-to Noise-Test application, open the second raw file of the batch **PCI_SN**, which is for the first of the two actual sample injections.

NOTE: If the application does not recognize the peak, change the detection drop-down from Automatic to the appropriate setting.

11. Transcribe the Signal-to-Noise (RMS) value into the corresponding field of the RPG Reports UI.
12. Using the **Evaluate / Run Test** function, run the RPG report.
13. Repeat this process for the second sample injection in the batch.

9 EVALUATING THE REPEATABILITY TEST SEQUENCES

9.1 Review

1. Review each chromatogram to ensure that the standard peak has been properly identified and integrated. Processing parameters may need to be updated to ensure proper standard integration.
2. Some reports may need editing. To edit any report, open the report, remove the protection (if any), make the appropriate edits, or record entries, and then save the report to incorporate customer specific information.
3. If any samples were re-run for any reason and should not be used in the final OQ analysis, they must be marked with an X in the **Do Not Use** column of the sample sequence. This may occur if a test needed to be re-run. The **Do Not Use** column of the sequence can be displayed by moving the **Do Not Use** column to the visible sequence columns. By placing an X in the **Do Not Use** column, the data will not be exported to the RPG Reports UI for analysis of the final OQ results.

9.2 Exporting the Standard Injection Data

1. Open the OQ sequence view.
2. Select the sequence in the **Data** column.
3. Right-click and select **Export**.
4. Use the following export settings:
   a. Use report template: **smaRT Export**
   b. With selected channel: **(Specific Detector)**
   c. Parent Folder: Select OQ data folder
   d. Export Formats and Filenames: **Text format (Results)(*.txt)**
5. Click **OK**.

9.3 Importing the Peak of Interest Injection Data into RPG Reports UI

9.3.1.1 RPG Reports 2.x

1. In section 4 of the RPG Reports UI, click the **Repeatability** section.
2. Complete any necessary system/detector information fields.
3. Using the smaRT export file created, load the peak data, and set the filters for **Sample Name, Injection Volume, Retention Time,** and **Peak Area**.

   The **Data Filter Column Designator** should be set to the **smaRT** column with the **Filter Criteria** using **R** (in order to filter just the Repeatability samples).
4. **Apply** the filter and wait for the values to be confirmed in the lower left-hand portion of the Data Filter screen, before clicking **Continue**.
5. Repeat the process for the remaining Standard Injection Tests (as applicable).

9.3.1.2 RPG Reports 3.x

1. Open the **GCMS Repeatability** report page.
2. For **Test Configuration** select the configuration (**based on injection type),** other to populate the limits.
3. For **Data Entry Mode** select **Import** as the data will be imported from the export file created earlier.
4. Click the **Import Test Data** button and browse to the smaRT export file created earlier (typically .txt file format).
5. Complete the **Sample Name, Injection Volume, Retention Time,** and **Peak Area** filter settings for the imported Data.

   The **Data Filter Column Designator** should be set to the **smaRT** column with the **Filter Criteria** using **R** (in order to filter just the Repeatability samples).

   Note: Filters must be applied ensure the proper data is used from the file. The Reset button can be used to change the filter.
6. **Apply** the filter and confirm the data is properly adjusted, then click **Continue**.
7. Select **Run Test** to process the results.
8. Add comments, as necessary.
9. Repeat the process for the remaining Standard Injection Tests (as applicable).

If an imported test fails, a report for that individual test should be created for the qualification packet, and a note made to the Exceptions and Comments. After remediation, additional data may be imported into RPG Reports.

10 EXCEPTIONS AND COMMENTS

If an exception is made during the qualification, the exception must be recorded in the appropriate **Comments** section of the Qualification Checklist or RPG Reports UI.

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10.1 What to do if a Test Fails

If at any point the instrument results do not pass within the specified limits, the instrument must be serviced and repaired. It is necessary to notify the customer that the system has failed a test and discuss the next steps, as the customer may have internal documentation to be completed detailing the repair. The appropriate hardware or service guide for the instrument may need to be referenced to return the instrument to operation. Once the repair has been completed, all applicable tests we need to be carried out. The failing and passing data or result report must be kept as part of the Qualification record.

All actions for failures and repairs should be recorded on the Comments section of the Qualification Checklist or in the RPG Reports UI.

11 QUALIFICATION REVIEW

The Service Representative must review all data and results and confirm whether each Qualification Test, and the overall qualification, has **Passed** or **Failed**. These results must be documented on generated Operational Qualification Reports or Checklists.

The Service Representative must indicate on the **OQ Review and Completion** and/or **Service Review and Completion** section of the RPG Reports UI that the Overall Operational Qualification is complete.

12 PRINTING AND SAVING QUALIFICATION DATA

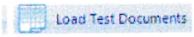
Ensure that all supporting data, chromatograms, and reports have been printed from the CDS. Ensure that all qualification test reports have been printed. If reporting is electronic, ensure all materials have been generated in a suitable file format (e.g., PDF).

Save to a storage device or other specified media or location all qualification related data and files that may have been created during the qualification process; this is an optional convenience for the customer.

12.1 Generating the Qualification Report in RPG Reports 2

1. Evaluate and review all applicable reports in the RPG Reports UI (Section 5: FINISH).
2. In the **Print/Publish** screen, move and arrange the desired reports from the **Available** list-box to the **Selected** list-box; click **Create PDF**.
3. Be sure to attach all CDS Reports to the Qualification Packet.

12.2 Generating the Qualification Report in RPG Reports 3

1. Attach the certificates in the **Test Equipment and Standards** section. PDF copies can be attached directly to the appropriate sections in the RPG Reports UI.
   a. FSE Certificate
   b. Tool Certificates
   c. Reagents Certificates
2. Procedures can be attached using the **Protocols** section (Note: this can drastically increase the size of the final report).
3. External CDS Reports should be attached to the applicable test page using the **Load Test Documents** button. 

4. Ensure the pages required are all selected, and that the content and order of the pages is correct.
5. The PDF Viewer button in the tool bar will allow you to see the final PDF report.
6. Select the number of signatures and signoff preference for the report:

a. Use Digital Signature (allows to attach a digital signature to the final PDF through Acrobat, signatures can be applied to electronic report after it is Submitted)

b. Use Pad Signature (allows to sign the report in the RPG Reports application using a signature pad, this must be done before Submitting the Report)

c. Manual Signature (spaces for handwritten signatures will be added to the final report, this requires printing)

7. Select **Finalize Report** to complete the report and remove the watermark (**Open Report** option will allow you to go back and make changes).

   You may then go to **File > Print** and either print the report to paper or save to PDF for electronic distribution.

13 BUILDING THE QUALIFICATION PACKAGE

1. Place all documentation in the appropriate section of the printed qualification binder/folder (paper documentation) or organize accordingly in the appropriate electronic storage location (electronic eBinder documentation).

   NOTE: Alternative / customer-oriented document storage is allowed.

2. Documentation required as follows (as applicable):
   - Hardware Installation Qualification Procedure
   - Software Installation Qualification Procedure
   - Hardware / Software Installation Qualification Reports/Checklists
   - Hardware Installation Qualification Data System Generated Test Reports
   - Hardware / Software Operational Qualification Procedure
   - Hardware / Software Operational Qualification Reports/Checklists
   - Hardware Operational Qualification Data System Generated Test Reports
   - Qualification Service Representative's Training Certificates
   - Test Equipment Certificates and Chemical standards Certificate(s) of Analysis
   - Any Additional Supplementary Documentation (e.g., Deviation Memo)
3. Complete the information on the qualification stickers, seal, and place a sticker on each of the following:
   - Qualification binder spine or folder (if paper is used, for component serial number, write Qual Package).
   - Each main system component that was qualified.
4. Complete the information on the qualification stickers, seal, and place a sticker on each of the following:
   - Qualification binder spine or folder (if paper is used, for component serial number, write Qual Packet).
   - Each main system component that was qualified.

14 QUALIFICATION COMPLETION AND REVIEW WITH CUSTOMER

The Service Representative must sign and date the qualification results (electronically or pen and ink). It is important that all qualification attachments have been properly labeled, initialed, and dated (as applicable).

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It is critical the final Qualification Package be reviewed with the customer as the final step in completing the Qualification. The customer can perform any reviewer signoffs at this time. This will help educate the customer on the work performed, allow any questions to be answered, and help prevent return visits.

After qualification, ensure the system is left in an appropriate state for the customer. This includes setting applicable modules into a standby (or off) status, removing any test columns, replacing any solvents, and disposing of any standard vials accordingly.

15 APPENDIX

A. Qualification of a TriPlus RSH SMART w/ VOC Sample Prep Station

A. Qualification of a TriPlus RSH SMART w/ VOC Sample Prep Station

If the system consists of a VOC sample prep workstation, Autosampler Repeatability with MS is performed per this procedure instead of the testing defined earlier in Section 6.3. Standalone MS, and GC testing is still performed under their respective protocols.

The Workflow Repeatability test is performed injecting 6 standards prepared with the script that runs the system. See **VOC_Workflow** sheet of gcms_method_reference.mht for operating parameters.

Workflow Repeatability with MS			
The repeatability test demonstrates that system injections are precise and consistent over a series. The relative standard deviation (%RSD) of sample peak area characterizes the repeatability of the injection volume and standard preparation.			
How Test is Performed			
Six standard samples are prepared diluting a solution in water and analyzing it in headspace mode.			
Module	**Test Name**	**Test Point / Standard**	**OQ Limits**
TriPlus RSH SMART LS/HS Sampler w/ VOC Sample Prep Station	Sample Preparation and Injection Repeatability	1,2 Dichlorobenzene	≤ 10.0% RSD

SAMPLE PREPARATION

1. Prepare 6 empty 20 ml vials,
2. Dispense 5mL of water into each vial.
3. Close the vials with a suitable magnetic cap, and place them in positions 55-60 on the first tray holder (in the 1st vt 15 tray).
4. Place a 2 mL vial with the standard in position 1 (in the 1st VT 54 tray.)

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