

TSQ GC/MS Performance Monitoring and Maintenance

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TSQ GC/MS Performance Monitoring and Maintenance

1 INTRODUCTION

This document addresses the performance monitoring and maintenance of the TSQ GC/MS (EI/CI) System. The Thermo TSQ GC/MS consists of a Trace Gas Chromatograph (GC) and a Triple Stage Quadrupole (TSQ) Mass Spectrometer (MS). These two instruments work in tandem and are referred to as the TSQ. The instrument is configured with a combination electron impact (EI) ionization and chemical impact (CI) ionization source using an interchangeable ion volume system.

When the instrument is in EI mode, it is implied that an EI volume is being used and that the reagent gas is off. Alternatively, when in CI mode, it is implied that a CI volume is being used and the reagent gas is on. The instrument can also be used with a Solids Probe. Solids Probe is a sample introduction technique utilizing the mass spectrometer for analysis and can be in either EI or CI mode. Definitions and guidelines are outlined in IOSS-701.

2 SCOPE

This document applies to personnel using the associated instrument(s)/equipment in the Explosives Chemistry subdiscipline.

3 EQUIPMENT

- Instrumentation
 - Thermo TSQ 8000 EVO, Thermo Trace GC, and data system with XCalibur software (or equivalent)
 - Thermo TriPlus Autosampler, accessories, and software (or equivalent)
- Materials
 - Agilent DB-5MS GC Column, 30 m, 0.25 mm i.d., 0.25 μ m film (or equivalent)
 - Helium, 99.99% (high purity)
 - Methane, 99.99% (high purity)
 - Perfluorotributylamine (PFTBA, FC-43) (Agilent or equivalent)
 - Chloroform, GC grade
 - Lidocaine HCl (Sigma or equivalent)
 - Tributyoxyethyl Phosphate (TBEP)(Chem Service or equivalent)
 - 2 mL GC autosampler vials, crimp or screw top, with or without 100-500 μ L inserts (Agilent or equivalent)
 - 4 mm split-splitless, tapered GC inlet liners, with or without glass wool (Restek or equivalent)
 - Low-bleed 11 mm injection port septa (Restek or equivalent)
 - 10 μ L gas tight autosampler syringes (SGE or equivalent)
 - General laboratory supplies

4 STANDARDS AND CONTROLS

- Testmix (0.05 mg/mL each of Lidocaine and TBEP)

The testmix is used to assess daily operating performance, mass assignment, and continued integrity of the system. To prepare, weigh 5.8 mg Lidocaine HCl and 5 mg TBEP into a 100-mL volumetric flask. Bring to the mark with chloroform and mix well. This solution does not expire, but should be re-evaluated for continued use after three years.

- PFTBA Tuning Solution

The PFTBA tuning solution is used for tuning the mass spectrometer and verifying mass calibration. It is supplied by the instrument manufacturer and does not expire. It is stored in a glass container attached to the TSQ. Under normal conditions, this should not need to be refilled.

5 PROCEDURE

5.1 Daily Checks

The following steps will be performed daily. Enter the information in the appropriate instrument log to indicate completion.

- A. Check to ensure that the GC wash vials are filled, the waste vials are empty, and all are in the appropriate positions.
- B. Record the remaining disk space on the hard drive. Verify that the hard disk has at least 1GB of free disk space. Do not use if less than 1GB remain.
- C. Record the line pressure of the building helium supply (carrier gas). The regulator should read 50 p.s.i. or above. If it cannot maintain this pressure, contact appropriate instrument support personnel. If the helium is supplied by a gas cylinder, record the tank pressure. Change the tank if less than 100 p.s.i. remaining.
- D. If using CI mode, record the tank pressure of the methane tank (reagent gas). Change the tank if less than 100 p.s.i. remaining.
- E. Check the ion gauge to ensure that there are no significant vacuum leaks in the system. Do not use if the pressure is higher than 1×10^{-4} torr with the reagent gas off.
- F. Prepare instrument for EI mode or CI mode. In the TSQ 8000 EVO Dashboard, select the ionization mode under 'Setup.' Open both the corresponding tune and calibration files (such as EI_TUNE or PICI_TUNE). Insert the correct volume. Check that the reagent gas is ON with a value of 2.0 for CI mode and OFF for EI mode.
- G. Perform an analysis of the testmix. Open the appropriate testmix instrument method (such as 'TestmixEI' or 'TestmixCI'), and verify the parameters as listed in the 'Instrumental Conditions' section of this procedure. Set up a sequence, load the autosampler with a vial containing the testmix, and start the analysis. Evaluate the results using the 'Acceptance Criteria' section. If the results are acceptable, print the following information for the testmix:

- For EI: RIC of m/z 86, RIC of m/z 299, and TIC. Label the peaks with scan number and/or retention time.
 - For CI: RIC of m/z 235, RIC of m/z 399, and TIC. Label the peaks with scan number and/or retention time.
 - Complete mass spectrum of both Lidocaine and TBEP.
- H. If all requirements are within specification, prepare the documentation as outlined in IOSS-701. If any requirements fail, contact appropriate instrument support personnel.

5.2 As Needed Checks

The following steps are to be performed as needed. Enter the information in the appropriate instrument log to indicate completion. Refer to IOSS-701 for more information on instrument maintenance and documentation.

- A. Replace the septum in the GC injection port.
- B. Replace the liner within the GC injection port.
- C. Check the GC syringe in the autosampler. Replace if needed.
- D. Check the internal bungee cords in the autosampler. Replace if needed.
- E. Appropriate instrument support personnel or trained operator: Tune the mass spectrometer. Perform the following procedure for both EI and CI modes:
 1. Save the current tunes as backups with filenames such as 'EI_TUNE_BACKUP' or 'PICI_TUNE_BACKUP.'
 2. Perform a standard tune on both Q1 and Q3.
 3. Save the tune files when completed with filenames such as 'EI_TUNE' or 'PICI_TUNE'.
 4. Manual optimization of parameters may be performed to fine-tune the MS.
 5. Acquire a mass spectrum of the PFTBA for Q1 and Q3 in EI, Positive Ion CI, and Negative Ion CI ranging from 50 to 650 m/z:
 - For EI: Collect approximately 20 scans for each quadrupole under the filename "tunespec1".
 - For Positive Ion CI: Collect approximately 20 scans for each quadrupole under the filename ""tunespec2".
 - For Negative Ion CI: Collect approximately 20 scans for each quadrupole under the filename "tunespec3".
 6. Evaluate the results using the 'Acceptance Criteria' section. If the results are acceptable, print the mass spectrum from each.
- F. If all requirements are within specification, prepare the documentation as outlined in IOSS-701. If any requirements fail, the IOSS Manager or appropriate instrument support personnel will determine the corrective maintenance to be taken.

6 INSTRUMENTAL CONDITIONS

6.1 Gas Chromatograph

Oven	
Initial Temperature:	60°C
Initial Time:	2.0 min
Ramp:	35°C/min
Final Temperature:	250°C
Hold Time:	10.0 min
Inlet/Injector	
Injection Volume:	1.0 µL
Mode:	Splitless
Inlet Temperature:	220°C
Column	
Type:	DB-5(MS)
Length:	30 m
Diameter:	0.25 mm
Film Thickness:	0.25 µm
Flow Mode:	Constant Flow, Vacuum Compensation
Flow Rate:	1 mL/min
Carrier Gas:	Helium

6.2 Mass Spectrometer

Solvent Delay:	5.0 min
Scan Mode:	Full Scan
EI Scan Range:	50-500 m/z (EI)
CI Scan Range:	100-500 m/z (CI)
CI Gas Flow:	2.0 mL/min initial, 1.0 mL/min @ 5.0 min
Transfer Line Temp:	280°C
Source Temp:	185°C

7 ACCEPTANCE CRITERIA

7.1 Testmix

- A. In order for the instrument to be considered in good operating condition, both Lidocaine and TBEP should generate well-resolved, Gaussian-shaped peaks with baseline separation.
- B. A SNR of 3:1 is the minimum response necessary to consider a response a peak.
- C. There should be no significant extraneous peaks in the chromatogram
- D. The retention times of each component should be similar as compared to previous analyses (unless GC maintenance has been performed, such as column clipping or replacement).
- E. Check for the correct mass assignments for the mass spectra:
 - o EI - Lidocaine ions 86 and 234 and TBEP ions 57, 199, and 299.
 - o CI - Lidocaine ion 235 and TBEP ions 299 and 399.

7.2 Tune

Verify the results of the tune. Compare the results of the tune to previous tune results. The following are typical PFTBA values for the TSQ. If the observed PFTBA peaks are outside the values listed below, the IOSS Manager or appropriate instrument support personnel will determine the corrective maintenance to be performed.

A. PFTBA Tune:

- o Mass assignments for m/z 69, 219, 414, 502, and 614

B. Relative Abundance:

- o EI: 69 or 219 base peak, 414 and 502 present
- o CI (POS): 414 base peak, 219, 69, and 614 present
- o CI (NEG): 633 or 452 base peak, 414 present

8 LIMITATIONS

Only properly trained personnel will perform duties involved in the operation, maintenance, or troubleshooting of this instrument.

9 SAFETY

Many instrument components are held at temperatures of 250°C and higher. Precautions should be taken to prevent the contact of skin with heated surfaces and areas.

10 REVISION HISTORY

Revision	Issued	Changes
06	11/15/2022	Section 2- Updated Scope to remove location. No other substantive changes to content.
07	06/01/2023	Section 6- Added CI gas flow to mass spectrometer conditions.