

Pyrolysis-GC/MS Performance Monitoring and Maintenance

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Pyrolysis-GC/MS Performance Monitoring and Maintenance

1 INTRODUCTION

This document addresses the performance monitoring and maintenance of a GC/MS (EI) with a pyrolysis autosampler. The pyrolysis-GC/MS system consists of a Gas Chromatograph (GC) with a single quadrupole Mass Selective Detector (MSD) and a pyrolysis autosampler. The instrument is configured with a dedicated electron impact ionization (EI) source. It may also be referred to as a 'PyMSD.' Definitions and guidelines are outlined in IOSS-701.

2 SCOPE

This document applies to personnel using the associated instrument(s)/equipment in the Paints and Polymers subdiscipline.

3 EQUIPMENT

- Instrumentation
 - Gas Chromatograph, Mass Selective Detector with EI Source and software (Agilent or equivalent)
 - Pyrolysis Autosampler, accessories and software (Frontier or equivalent)
- Materials
 - Mid-polarity capillary GC column, HP-5, 30 m, 0.25 mm i.d., 0.25 μ m film (or equivalent)
 - Helium, 99.99% (high purity)
 - Alloyed metal sample cups (Frontier or equivalent)
 - Cleaning apparatus for sample holders (small butane torch, sample cup inspector, sample cup holder)
 - Polystyrene pellets (Scientific Polymer Products, Inc. or equivalent)
 - High Density polyethylene pellets (Scientific Polymer Products, Inc. or equivalent)
 - Perfluorotributylamine (PFTBA, FC-43) (Agilent or equivalent)
 - General laboratory supplies

4 STANDARDS AND CONTROLS

- Performance Verification Standards
 - Polystyrene Standard (Daily Performance Verification Standard)
The polystyrene is used to assess daily operating performance, mass assignment, and continued integrity of the system. It can be purchased as a standard in pellet form. Prepare for analysis by cutting a polystyrene pellet to obtain a size that will provide an adequate signal. Place the polystyrene in the bottom of a sample cup.
 - Polyethylene Standard (Monthly Performance Verification Standard)
The polyethylene is used to assess monthly operating performance, column selectivity, and continued integrity of the system. It can be purchased as a standard in pellet form. Prepare for analysis by cutting a polyethylene pellet

to obtain a size that will provide an adequate signal. Place the polyethylene in the bottom of a sample cup.

- **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 MSD. 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 appropriate information in the instrument log.

- A. Perform a tune of the instrument. Select ETUNE and the mass spectrometer will tune itself using PFTBA. Evaluate the results using the 'Acceptance Criteria' section. If the results are acceptable, print the tune report when completed.
- B. Open the appropriate instrument method, and verify the parameters as listed in the 'Instrumental Conditions' section. Set up a sequence, load the autosampler with a blank and a sample vessel containing the polystyrene, and start the analysis. Evaluate the results using the 'Acceptance Criteria' section. If the results are acceptable, print the TIC and mass spectra for the polystyrene as well as for the prior blank.
- C. 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 Monthly Checks

The following steps will be performed monthly. Enter the appropriate information in the instrument log to indicate completion. Refer to IOSS-701, IOSS-702 and IOSS-703 for more information on instrument maintenance and documentation.

- A. Perform an analysis of a blank followed by the polyethylene standard. Open the appropriate instrument method, and verify the parameters as listed in the 'Instrumental Conditions' section. Set up a sequence, load the autosampler with a blank and a sample vessel containing the polyethylene, and start the analysis. Evaluate the results using the 'Acceptance Criteria' section. If the results are acceptable, print the TIC for the polyethylene as well as for the prior blank.
- B. 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

Refer to PP-201 for recommended instrument conditions.

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7 ACCEPTANCE CRITERIA

7.1 Tune

Verify the results of the tune. Compare the results of the tune to previous tune results. Significant voltage increases or changes in the isotope ratios indicate the need to initiate corrective maintenance procedures. The following are typical tune values for the MSD:

- A. PFTBA tune:
 - o $m/z \pm 0.4$ for m/z 69, 219, and 502
- B. Peak width:
 - o 0.45-0.65
- C. Relative abundance:
 - o 69 greater than 50%
 - o 219 greater than 50%
 - o 502 greater than 1%

7.2 Polystyrene and Polyethylene

- A. For the instrument to be considered in good operating condition, all components 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 unrelated peaks in the pyrogram.
- D. Ideally, the blank preceding the performance verification standard should not exhibit any chromatographic peaks greater than the CO₂ response; if extraneous peaks are present but explainable (e.g., siloxanes), this should be noted on the blank printout for technical review.
- E. 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).
- F. For polystyrene, check for the correct mass assignments in the mass spectrum and compare the fragmentation patterns with previous analyses.
 - o Monomer ions - 50, 51, 77, 78, 103, and 104 (base peak).
 - o Dimer ions - 65, 91 (base peak), 104, 115, 117, 130, 193, and 208.
 - o Trimer ions - 91 (base peak), 115, 117, 194, 207, and 312.

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, and areas of the pyrolysis autosampler reach temperatures in excess of 800°C. Precautions should be taken to prevent the contact of skin with heated surfaces and areas.

10 REVISION HISTORY

Revision	Issued	Changes
06	07/01/2022	Revised to match new format requirements. Section 2- Updated scope. Section 5- Updated from ATUNE to ETUNE.
07	11/15/2022	Section 2- Updated scope to remove location. No other substantive changes to content.