

AccuTOF DART Performance Monitoring and Maintenance

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AccuTOF DART Performance Monitoring and Maintenance

1 INTRODUCTION

This document addresses the performance monitoring and maintenance of the JEOL AccuTOF DART. The AccuTOF DART system consists of a JEOL AccuTOF Time-of-Flight (TOF) mass spectrometer (MS) and an IonSense Direct Analysis in Real Time Source (DART) ionization source. The TOF has the ability to measure mass accuracy with less than 5 milli-mass units (mmu) error over a range of 1 - 10,000 m/z. The DART source provides the capability of direct ionization with little to no sample preparation needed. The combined system may also be referred to as a 'DART.'

The TOF tube is sensitive to internal and external changes in temperature and humidity. These changes may result in a uniform shift of mass assignments. This shift is easily corrected by analyzing the calibrant in every sample analysis/data file and applying the new calibration algorithm, producing mass-corrected accurate mass data with less than 5 mmu error.

It should be noted that the instrument manufacturer can be referred to as either JEOL or IonSense. DART instrumentation and supplies are sold and supported by JEOL USA, Inc. Definitions and guidelines are outlined in IOSS-701.

2 SCOPE

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

3 EQUIPMENT

- Instrumentation
 - JEOL AccuTOF MS, IonSense DART Source, and data system with IonSense DART Controller and MassCenter Main software (or equivalent)
- Materials
 - Helium, 99.99% (high purity)
 - Nitrogen, 99.99% (high purity)
 - Polyethylene Glycol (PEG), average molecular weight of 550 (Sigma or equivalent)
 - General laboratory supplies

4 STANDARDS AND CONTROLS

Neat Polyethylene Glycol (PEG) is used for performance verification and mass correction.

5 MASS CORRECTION

PEG is analyzed with every sample in the same data file, and used for fine mass correction. This results in the highest mass accuracy possible, taking all instrumental and environmental conditions into account at the time of analysis. This also ensures that mass correction data will be available at any time in the future when data processing is performed. The operator is responsible for verifying that every mass correction graph produced passes based on the

'Acceptance Criteria' section. However, only the first graph is retained for daily performance verification.

6 PROCEDURE

6.1 Start Up

6.1.1 DART Initialization

- A. Open the DART Controller Software.
- B. Select 'Standby'.
- C. Select the appropriate operating temperature and gas type.
- D. Set the polarity required.
- E. Once DART has reached the operating temperature, select 'Run'.

6.1.2 TOF Initialization

- A. Open MassCenter Main.
- B. Open MS Tune Manager.
- C. Put instrument into 'Operate' mode.
- D. Load the appropriate tune file, verifying that the polarity matches the DART source.
- E. Verify that the appropriate analyzer settings.

6.2 Daily Checks

- A. Record the remaining disk space on the hard drive. Use Windows Explorer to verify that the hard disk has at least 20 GB of free disk space. Do not use if less than 20 GB remain. If less than 20 GB of free disk space remain, contact appropriate instrument support personnel.
- B. Record the line pressure of the building nitrogen supply. The regulator should read at least 70 p.s.i. If it cannot maintain this pressure, contact appropriate instrument support personnel. If the nitrogen is supplied by a gas cylinder, record the tank volume pressure. Change the tank if less than 100 p.s.i. remaining.
- C. Record the line pressure of the helium. The regulator should read at least 40 p.s.i. If it cannot maintain this pressure, contact appropriate instrument support personnel. If the helium is supplied by a gas cylinder, record the tank volume pressure. Change the tank if less than 100 p.s.i. remaining.
- D. Check the vacuum pressure readings. The analyzer pressure must be below 9.9×10^{-5} Pa with the DART controller in standby (i.e., nitrogen flowing, not the helium).
- E. Perform a data acquisition for PEG using the procedure in 7.3 below. A PEG analysis from a sample acquisition collected the same day can be used as well.

6.3 Data Acquisition

- A. Open Spectrum Monitor. Ensure that there is a signal.
- B. Start an acquisition by selecting 'Acquire.' Type in the filename and comments and choose a data folder. Set the desired m/z range and verify the length of acquisition is appropriate (typically 2 minutes).

- C. Once the acquisition has started, ensure that there is a signal, and collect several seconds of background data.
- D. Dip the closed end of a glass capillary into the PEG, then place in the DART gas stream until a response is seen in the spectrum viewer for several seconds. Do this at least twice during the acquisition.

6.3.1 Time of Use Performance Verification/Mass Correction

Verify the performance of the instrument in the first data file acquired:

- A. Open the sample data file in the Chromatogram Viewer.
- B. View a background-subtracted PEG spectrum by holding down the 'shift' key and dragging across the PEG area with the right mouse button, then drag across a baseline area while holding the 'ctrl' key.
- C. Once the PEG spectrum appears in the Spectrum Viewer, generate a centroided spectrum.
- D. Right mouse click on the centroided spectrum, and make a calibration file from the spectrum.
- E. Select the appropriate polynomial order to provide the lowest residuals curve (1-R and 1-R* less than 10^{-10}).
- F. Evaluate the results using the 'Acceptance Criteria' section.
- G. If the results are acceptable, retain the PEG mass correction graph.
- H. Save the mass correction using an appropriate file name. The file can now be applied to the sample chromatogram to obtain accurate mass data.

6.3.2 Data Correction

Verify the performance of the instrument in every data file acquired thereafter.

- A. The steps listed under 6.3.1 will be followed for every sample acquisition in order to ensure accurate mass results. However, it is not necessary to record subsequent results in the QA/QC log for the same day.
- B. Verify that the residuals (1-R and 1-R*) are 10^{-10} or lower before applying the mass correction algorithm to the sample.

6.4 Instrument Shutdown

6.4.1 Time of Use Shutdown

- A. Put the MS Tune Manager in 'Analyzer HV' mode.
- B. Put the DART in 'Standby' mode, which will maintain the voltages and switch to nitrogen as the flow gas.
- C. Turn the heater off with nitrogen flowing to allow the heater to cool.

6.4.2 End of Day Shutdown

- A. Put the MS Tune Manager in 'Analyzer HV' mode.
- B. Turn the DART off, which will turn off the voltages and turn off the nitrogen flow gas.

6.4.3 As Needed Maintenance

Refer to IOSS-701 and IOSS-703 for definitions and documentation guidance.

- A. Replace the grid.
- B. Clean the cones, source, and source enclosure.

7 INSTRUMENTAL CONDITIONS

Ionization:	DART
Scan Mode:	Profile
Analyzer:	500 V peaks voltage for 50 - 500 m/z 800 V peaks voltage for 80 - 800 m/z

8 ACCEPTANCE CRITERIA

8.1 PEG Performance Verification/Mass Correction

Verify that the residuals (1-R and 1-R*) are 10^{-10} or lower.

9 LIMITATIONS

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

10 SAFETY

There are several areas of the MS which utilize extremely high voltage and vacuum conditions. For this reason, maintenance should only be performed with the system vented and the main power off. Many MS 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.

11 REVISION HISTORY

Revision	Issued	Changes
03	07/01/2022	Revised to match new format requirements. Section 2- Updated Scope. Section 5- Changed from 'Calibration' to 'Mass Correction' here and throughout document.
04	11/15/2022	Removed 'JEOL' from title. Section 2- Updated Scope to remove location. No other substantive changes to content.