

Experimental Guidelines for Collecting Data on the Agilent GC/MS for MassWorks

EXPERIMENTAL SETUP – GENERAL GUIDELINES

GC/MS data must be acquired in what is known as “Raw Scan” or “Profile mode”. MassWorks requires a spectral calibration standard to be acquired to calibrate the instrument for accurate mass and high spectral accuracy, usually by turning on the built-in calibration gas during an otherwise conventional GC/MS run.

MS Acquisition and Calibration (REQUIRED!)

- The instrument must be set to acquire “Raw Scan” data (also known as “Profile Mode”)
- The instrument “Threshold (counts)” should always be set to 0 to ensure all raw data be acquired
- The instrument scan rate should be fast enough to assure 8 to 10 scans across the typical chromatographic peak, but not overly fast. On Agilent GC/MS systems, “Sampling Rate” = 3 is usually recommended.
- The scan m/z range should be set as short as possible (Start at Mass, End at Mass) to cover only the mass range necessary for analyzing the compounds of interest
- The PFTBA calibration gas should be turned on briefly at the end of the run AFTER THE OVEN COOLS DOWN to acquire data for the accurate mass spectral calibration
- Calibrations can be internal or external to the GC/MS sample run and it’s best to have a new calibration acquired at least once per day, even though published work has shown that the calibration on Agilent GC/MSD will hold for as long as 3 weeks, as long as the MS tune and MS acquisition parameters remain the same.
- To take advantage of MassWorks “AutoCal” feature, the calibration has to be “internal” to the sample run and the calibration or tune gas has to be **first turned on and then off** before concluding the data acquisition. Turning PFTBA on without turning it off would not trigger AutoCal.

Always use the same instrument scan parameters for the sample and calibration acquisition!

Screen shots showing scan parameter setup for Agilent GC/MS is shown below in Figure 1-3. For a detailed description and explanation of acquisition parameters and setups, please refer to the [Agilent Application Note 5990-4966EN](#).

Acquisition setup for Agilent Chemstation

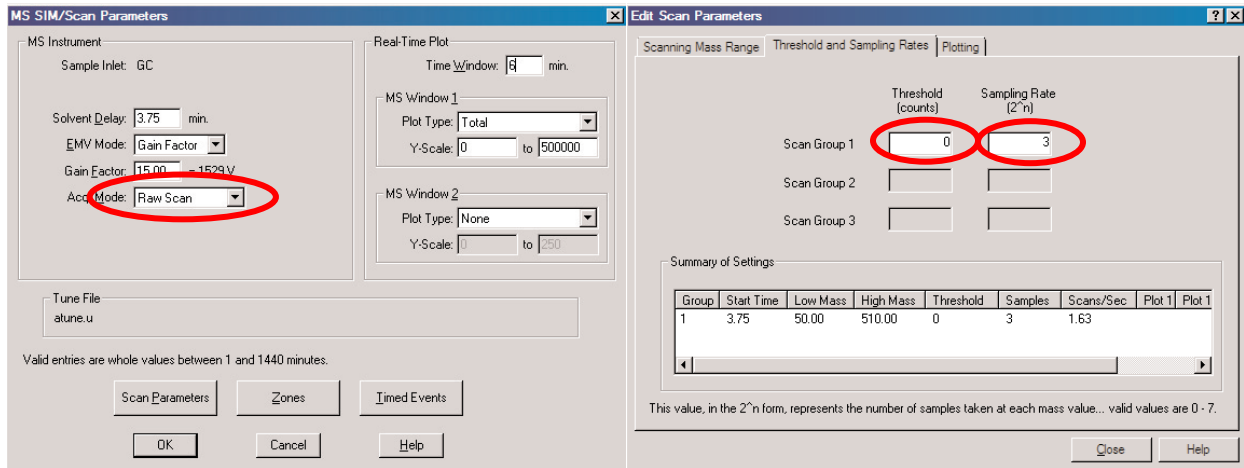


Figure 1. Typical parameter settings for Agilent GC/MSD Scan parameters in ChemStation. Note Acq. Mode = Raw Scan, Threshold = 0, and Sampling Rate = 3.

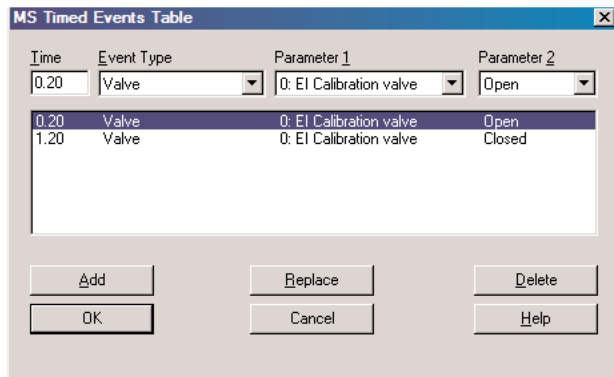


Figure 2. Add a timed event to your MS method to turn on the PFTBA calibration gas at the end of the run after oven cools down. Please return to this interface to adjust the event time to reflect the actual GC run time after programming/editing the GC method.

Acquisition setup for Agilent MassHunter

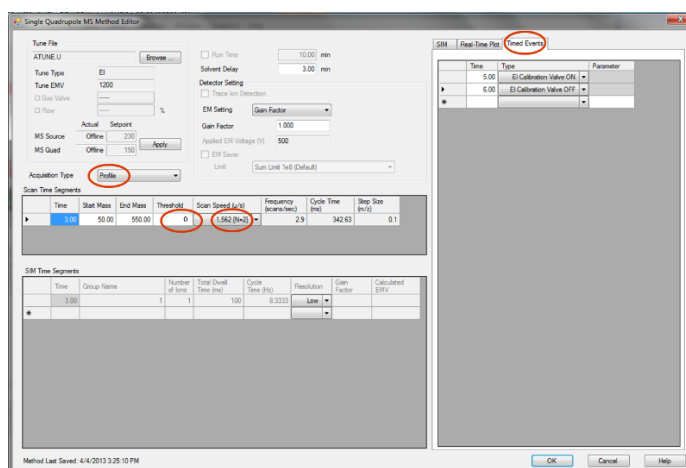


Figure 3. Typical MS Method parameter settings for Agilent GC/MS in MassHunter. Note Acquisition Type = Profile, Threshold = 0, and Scan Speed corresponds to N=3. Also shown is the Timed Events settings for turning on and then off the calibration gas valve near the end of the run (after the GC separation is completed and after oven cools down). Please return to this interface to adjust the event time to reflect the actual GC run time after programming/editing the GC method.

Typical Run and Opening the Data in MassWorks

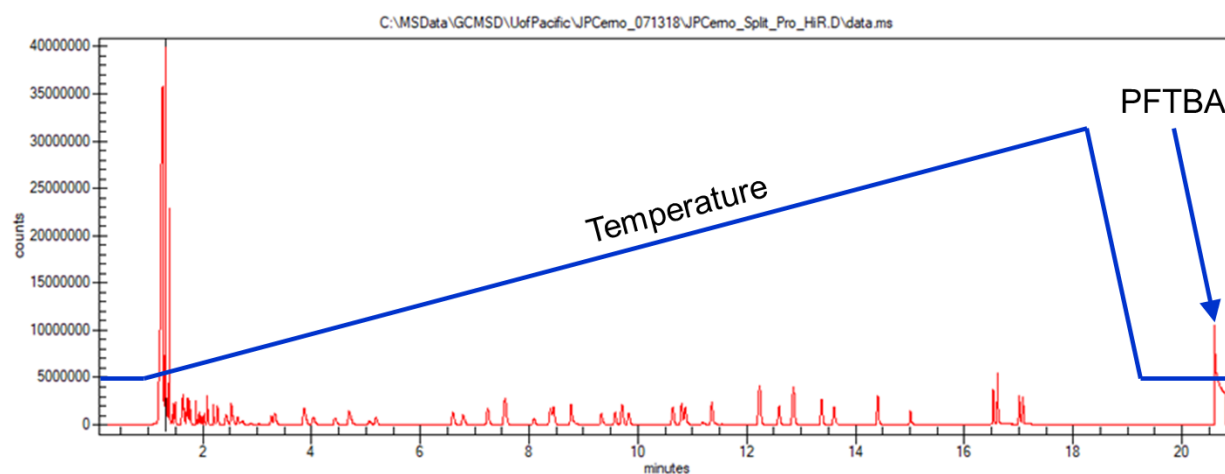


Figure 2. Typical GC/MS run showing the temperature programming including, before the end of run, after GC separation is complete and oven cools down, and during the hold time period, the introduction of PFTBA calibration gas for a short period of time (30 seconds is usually adequate).

Once the data is acquired, it can be directly opened in MassWorks for calibration and processing. Unlike the Agilent software, you must open the binary file located in the folder. For example, in ChemStation, to open the data in the folder "SAMPLE.D", simply navigate to the folder and open the "DATA.MS" file inside the .D folder. This file will also be present and available for GC/MSD data acquired by MassHunter.