M313 Instrument Configuration Guide

Background

The purpose of this guide is to give new users a starting point for their instrument configuration and to suggest an approach to meet all M313 QC requirements not met during initial setup. The IOM is utilized to evaluate instrument performance for AQMD M313, particularly for discrimination for or against certain molecular weight classes on the volatilized sample's path to the detector. This guide is written for a post-column split to the MS and FID on an Agilent GC-MS, although this configuration is not required to perform M313.

Instrument Parameters and Setup

Set up the GC/MS/FID in liquid analysis mode with a 10 µL autosampler syringe and the appropriate wash solvent. A wide variety of instrument parameters have been used successfully, but the following parameters are recommended as a starting point for instrument configuration.

In a two-detector system, install a capillary column splitter and use a length of fused-silica tubing to connect the splitter to each detector; connect 80 cm of 0.32 mm ID tubing to the FID and at least 50 cm of 0.1 mm tubing to the MS.

Injector Parameters:

Injector Type	Split/Splitless using Splitless Mode (Wide-bore, large volume, double-gooseneck inlet liners containing deactivated glass wool have proven helpful to minimize discrimination)
Injector Temp	255 °C or lower
Injector Pressure	8-14 psi
Total Flow Rate	60-75 mL/min
Purge Flow Rate to Split Vent	>35 mL/min @ 0.1 – 0.5 min
Gas Saver On	20 mL/min after 2 min
Injection volume	1 µL

Column Parameters:

Column	Column DB-624®, 30m X 0.32 mm, 1.8 um film
Flow Mode	Constant Flow
Column Flow Rate	1.5 - 4 mL/min
Total Run Time	35 - 50 min

GC Oven Temperature Ramp:

Initial Temp	35 °C or higher
Init. Hold Time	2 - 5 min
1st Ramp Rate	4 - 8 degrees per minute
1st Ramp Temp	75 °C
1st Hold Time	0 - 4 min
2nd Ramp Rate	10.0 degrees per minute
2nd Ramp Temp	225 °C

2nd Hold Time	6.0 min
3rd Ramp Rate	25.0 degrees per minute
3rd Ramp Temp	255 °C
3rd Hold Time	4 – 10 min
Total Run Time	35 - 50 min

FID Detector Parameters:

Detector Heater	240-255 °C
Hydrogen Flow Rate	40 mL/min
Air Flow Rate	450 mL/min
Makeup Flow Rate	45 mL/min

MSD Parameters:

MS Transfer Line	280 °C
MS Source	230 °C
MS Quad	150 °C
MS Scan Start Time	0.0 min
MS Scan Range	5-505 amu

Turn the MS "off" in the instrument software whenever the solvent (MeOH or THF) peak emerges, and turn it back on just before the peak reaches baseline.

Follow the ***Practical Preparation Guide for Instrument Optimization Mix*** to prepare an IOM. Inject the IOM in replicate with a solvent pre-blank and interstitial blank. Add the IOM results to the ***Prep + Discrimination Template*** and calculate the results. The ***Practical Preparation Guide for Instrument Optimization Mix*** provides information on passing IOM criteria.

It may be the case that the initial instrument parameters meet all IOM QC requirements. In the event that the instrument parameters require modification, it is recommended that analyte sensitivity be addressed first, followed by analyte resolution and then analyte discrimination.

Instrument Sensitivity

To determine detection sensitivity, assess the 0.1 g/L TRIG peak in the IOM analysis by injecting the standard 7 times and inputting the data into the ***Prep + Discrimination Template*** where an LOD will be calculated. If necessary, increase or decrease the injected volume to get the desired detection sensitivity or to avoid sample overload. Be mindful of the EG, EGDE, and PG resolution while adjusting the detection sensitivity. Detection sensitivity must be robust enough that it should not be significantly compromised by slight fluctuations in the amount of sample introduced into the column; it should, however, not be so high that the common VOCs EG and PG are overloaded and co-elute with EGDE. A passing LOD for triglyme is considered to be 0.02 g/L or lower. A common method for improving sensitivity is to increase the injected volume of sample.

Analyte Resolution

Use the following oven temperature ramp profile as a starting point when optimizing analyte resolution:

Oven	°C/Min	Next °C	Hold Time (min)	Run Time (min)
Initial	NA	35 or higher	5	5
Ramp 1	1	50	5	25
Ramp 2	10	255	7	52.5

Adjust the GC oven temperature ramp profile and carrier gas flow rate such that you get the optimum resolution among the components in the IOM. Maximize the resolution of the critical analytes: Ethylene Glycol (EG), Ethylene Glycol Ethyl Ether (EGDE), and Propylene Glycol (PG). In general, better resolution is often obtained at lower flow rates. THF may require a longer splitless hold time than methanol. It is not necessary that the analytes of interest be fully resolved since the following optimization steps may contribute to their separation.

Once analyte sensitivity has been optimized, focus on minimizing analyte discrimination.

Discrimination Profile

Assess the instrument discrimination profile by normalizing the n-hydrocarbon (nHC) peaks in the IOM using each peak's mass (g), then calculating a %D for each peak using the normalized nC10 as indicated in the ***Practical Preparation Guide for Instrument Optimization Mix***. The discrimination profile is within tolerance if the %D for each compound is within ±15%.

If the instrument parameters do not meet the ±15% tolerance, run an instrument sequence comprised entirely of IOM injections. Alter the following instrument parameters, one at a time, for each injection:

(1) hold the inlet split vent closed and open the vent purge at a different time (0.0 to 0.5 minutes after injection)
(2) adjust the inlet vent purge flow rate (40 to 60 mL/min)
(3) change the injection amount (0.2 to 1 µL) and injection speed, dwell time, plunger draw speed and other syringe/injector mechanics.

Through repeated injections, discrimination trends will become apparent and should be used to determine the most appropriate course of action for further instrument adjustments.

After conditions are optimized, run the IOM 7 times with the final chosen GC conditions to check for consistency. If all injections are consistent and pass the discrimination, separation and LOD optimization criteria, the instrument is ready for analysis. If injections are inconsistent, check the integrity of the syringe and check for leaks in the system. To improve precision, use a syringe that has a volume capacity closest to the dispensed volume.