

Instrument Optimization Mix (IOM) – Practical Preparation Guide

Background

The Instrument Optimization Mix (referred as IOM henceforth) is a mixture of compounds used to test and optimize peak resolution, injection representativeness and discrimination, mass spectrometer tuning, and to determine and check the endpoint for VOC determination. The purpose of the IOM is to make sure that compounds in samples are correctly represented throughout the whole chromatogram, and it should be run at the beginning and end of each analysis sequence.

Specifically, the results of the IOM runs need to meet the following criteria:

- 1) Chromatographic resolution for ethylene glycol (2-3 g/L), EGDE (5 g/L), and propylene glycol (2-3 g/L) must be at least 90%.
- 2) The analysis system must not discriminate in exceedance of $\pm 15\%$ between hydrocarbons nC6 to nC15 (hexane to pentadecane) normalized to decane as measured at the FID. The hydrocarbon area counts are first normalized to their purity adjusted mass:

$$\text{Hydrocarbon area counts (normalized)} = \frac{\text{Hydrocarbon area counts}}{\text{Mass of hydrocarbon} \times \text{purity of the standard}}$$

The discrimination calculation is then performed as follows:

$$D\% = \frac{\text{Hydrocarbon area counts (normalized)}}{\text{Decane area counts (normalized)}} \times 100$$

This calculation function is automatically performed in the **Prep + Discrimination Template**.

- 3) Triglyme (TRIG) requires a method detection limit (LOD) of 0.02 g/L or lower (defined as the statistically calculated minimum amount that can be measured with 99% confidence). This is verified through the IOM results by a TRIG recovery of 80-120% at 0.1 g/L prepared concentration. Since calibrations will not be performed on TRIG during phase one of the pilot test, detection of TRIG is sufficient for a successful IOM analysis.
- 4) 4-bromofluorobenzene (BFB) must pass the EPA TO-15 tuning criteria for 0.1 g/L prepared concentration. This is achieved by measuring the relative intensity of key mass fragments of BFB in the IOM mix to demonstrate appropriate ionization. Mass spectrometer tuning will not be reported during phase one of the pilot test, but will be evaluated in phase two.

- 5) Methyl palmitate (MeP) is the first peak not to be integrated in the VOC sample analysis, thus the retention time should not substantially drift from injection to injection. The drift in MeP retention time between IOM runs should not exceed 0.1 minutes.

Preparation

The following compounds are required for the IOM preparation:

Compound	CAS	Concentration (g/L)
THF or Methanol	109-99-9 or 67-56-1	NA (Solvent)
Hexane	110-54-3	3
Heptane	142-82-5	3
Octane (can be excluded if co-eludes with EGDE)	111-65-9	3
Nonane	111-84-2	3
Decane	124-18-5	3
Undecane	1120-21-4	3
Dodecane	112-40-3	3
Tridecane	629-50-5	3
Tetradecane	629-59-4	3
Pentadecane	629-62-9	3
Ethylene glycol diethyl ether (EGDE)	629-14-1	5
Triethylene glycol dimethyl ether (TRIG)	112-49-2	0.1
Ethylene glycol (EG)	107-21-1	3
Propylene glycol (PG)	57-55-6	3
p-Bromofluorobenzene (BFB)	460-00-4	0.1
Methyl palmitate (MeP)	112-39-0	3

Note: When using methanol as solvent, more rigorous mixing is required. Alternatively, 2 g/L hydrocarbon concentrations may be used. This guide refers to 3 g/L level preparation in a 25 mL volumetric flask.

Required lab equipment:

- Analytical balance (0.1 mg sensitivity)
- Clean 25 mL Class A volumetric flasks
- Gas-tight syringes with needles (500/250/100 µL)
- Weighing boats or weighing paper
- 1 mL Class A Volumetric pipets
- Vials, 2 mL, screw cap with Teflon® faced septa

Procedure:

To prevent evaporative losses of the lighter hydrocarbons, it is recommended that additions are made starting from the heaviest hydrocarbon. If using the same syringe for multiple additions, the syringe must be properly rinsed between additions: first with whichever solvent (THF or Methanol) is used for the IOM preparation followed by the compound to be added to the mixture. It is also important to keep the flask capped whenever additions are not being made to prevent evaporation.

IOM mix preparation consists of two parts: preparing the 5 g/L dilution stock solution for 0.1 g/L standards (BFB and TRIG) and preparing the IOM mix from 3 g/L standards and the stock solution. See the *Prep + Discrimination Template* for a visualization of how the two standards are related. Record all weights in the Preparation tab of the *Prep + Discrimination Template*.

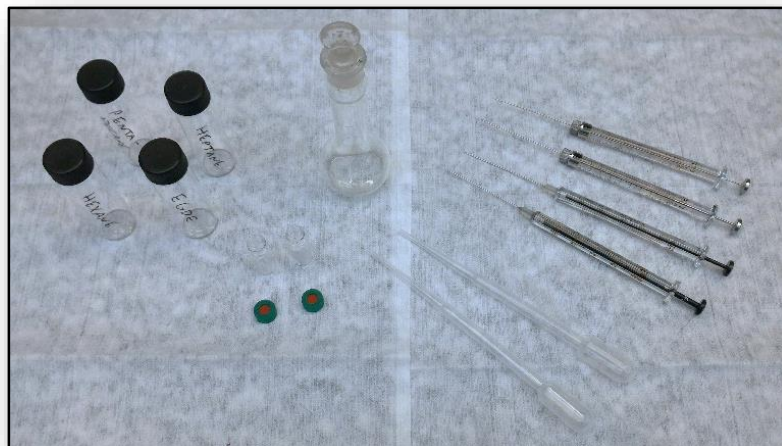
5 g/L dilution stock solution preparation:

1. Weigh a 25 mL Class A volumetric flask. Record weight down to 0.1 mg
2. Add approximately 10 mL of solvent (THF or Methanol). Record weight.
3. Add 80 μ L of BFB. Record weight.
4. Add 130 μ L of TRIG. Record weight.
5. Fill the flask up to its graduation marking with the solvent. Record weight.
6. Mix the solution preferably by vortex mixer or shaking the flask.

IOM solution preparation:

1. Weigh a 25 mL Class A volumetric flask. Record weight down to 0.1 mg
2. Add approximately 10 mL of solvent (THF or Methanol). Record weight.
3. Add 150 μ L of EGDE. Record weight.
4. Add 100 μ L of each hydrocarbon, starting from pentadecane. Record weight after each addition.
5. Add 100 μ L of ethylene glycol. Record weight.
6. Add 100 μ L of propylene glycol. Record weight.
7. Add 500 μ L of previously prepared 5 g/L dilution stock solution. Record weight.
8. Weigh 0.140 g of methyl palmitate using weighing boat or –paper and add to the flask. Record weight.
9. Fill the flask up to its graduation marking with the solvent. Record weight.
10. Mix the solution preferably by vortex mixer, or by shaking the flask.
11. Pipette the solution into 2 mL vials. Store in the freezer for up to two months.

Note: If the IOM is prepared in methanol, more thorough mixing is required (preferably using a vortex mixer) to completely dissolve the heavier hydrocarbons. The IOM needs to be brought to room temperature and then mixed rigorously when vials are removed from the freezer following storage. If THF is used, less intense mixing is sufficient.



Sampling of the required lab ware, including vials for IOM compounds, 25 mL Class A volumetric flask, gas-tight syringes, pipettes, and 2 mL vials.