Optimization Routine For Large Volume Injection

This LVI routine is based on OPTIC and fast (not speed controlled) injections. A packed liner should be used to hold the solvent during evaporation. (check for the LVI training manual on www.glsciences.eu)

1. OBTAIN A REFERENCE CHROMATOGRAM

Either from previous work, or by using an on-column injection of a 1µl standard solution, obtain a reference chromatogram for comparison purposes. (if no on-column injector available use a 1µl splitless injection).

2. CHECK THE LINER PACKING IS INERT WITH RESPECT TO THE SAMPLE

Fit a packed liner and inject 1µl of the standard in the cold splitless mode. Compare the results with the reference chromatogram. Differences between the two chromatograms can indicate non-optimised splitless transfer conditions. For example: If the peak areas tend to be smaller than the reference chromatogram, the Optic temperature could be too low, or, the splitless time is too short: If some of the peaks are smaller or absent, when compared to the reference chromatogram, the liner packing may not be inert to all of the analytes of interest.

3. DETERMINE THE LIQUID CAPACITY OF THE LINER (Vmax)

Without turning off the carrier gas, remove the column from the injector and inject 150µl of solvent. Look for solvent droplets at the injector outlet. Progressively reduce the amount injected until no droplets are observed. This volume is Vmax.

4. DETERMINE THE SOLVENT ELIMINATION TIME (Vent time)

Reconnect the column to the injector. Set the column temperature to the injector initial temperature. Set the detector to minimum sensitivity. Set the vent flow to 100 ml/min. Rapidly inject a volume of pure solvent equal to the volume to be used in large volume injection (do not exceed 90% VMAX). Measure the peak width of the solvent in seconds.

5. LARGE VOLUME INJECTION OF STANDARD

Dilute the standard by a factor equal to the number of µl to be used in the large volume injection, using clean solvent. Set the vent time to the solvent elimination time. Set the detector sensitivity to that used to obtain the reference chromatogram. Inject the selected volume of dilute standard (do not exceed 90% Vmax).

Compare the chromatogram obtained with the reference chromatogram.

6. FINE TUNING

If the chromatogram from the large volume injection looks like the reference chromatogram, no further optimisation is required. If volatile components are lost, reduce the vent time in steps of 3-5%.

7. HINTS

- Do not be tempted to take short-cuts.
- Set the vent flow to 100 ml/min.
- Set the split flow to 50 ml/min.
- Set the initial injector temperature in the range 30-40°C below the solvent boiling point.
- Set the final temperature to the liner packing maximum temperature or 50°C above the elution temperature of the latest running peak.
- Set the ramp rate to 4°C/min.
- Vmax is usually 60-125µl.
- Vent time is usually 20-50 seconds. (Or use the solvent sensor to determine the vent time)
- Polar solvents have larger elimination times.
- When using GC-MS, the rise in source pressure can be used to determine the solvent elimination time.