

Plasma Diagnostics and Simulation

Task: Gas Chromatography

Exercises

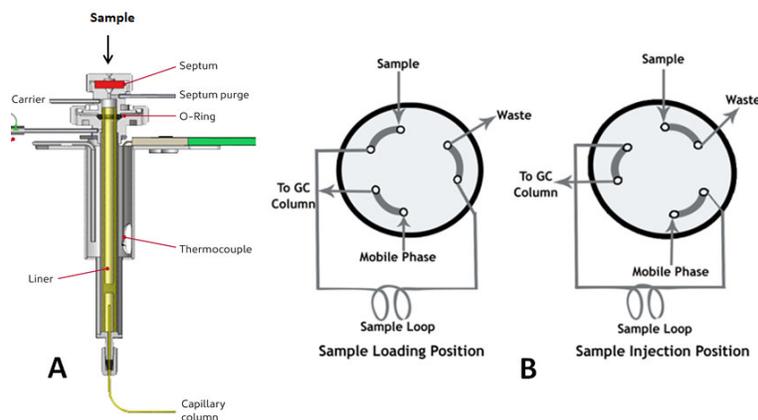
1. Identification of organic vapors.
2. Identification of organic components in the sample solution.

Introduction

Gas chromatography is a powerful method for identification and quantification of organic compounds in gas and liquid mixtures and their solutions in organic solvents. The analyzed compounds should be sufficiently volatile (high vapor pressure, low boiling point) and thermally stable. Various components of a mixture are separated according to their distribution coefficients between stationary phase and mobile phase (helium) in the capillary chromatographic column (compound with larger distribution coefficient value remains in the stationary phase for longer time and thus has longer retention time in the column). After separation on the column, separated components are detected in a detector: flame ionization detector (FID) or quadrupole mass spectrometer (MS) are commonly used. Parallel usage of both detectors (MS and FID) improves selectivity (some compounds like permanent gases, carbon monoxide and dioxide, water, formaldehyde, etc., are not active in the FID detector) and sensitivity of the system.

Gas chromatography device *Agilent Technologies 7890B* enables two different methods of sample input:

- A. Injection of a liquid sample to the chromatograph inlet using microsyringe (via autosampler or manually).
- B. Loading of a mixture of gases or organic vapors using six port injection valve (via high-volume syringe or online interconnection with a reactor).



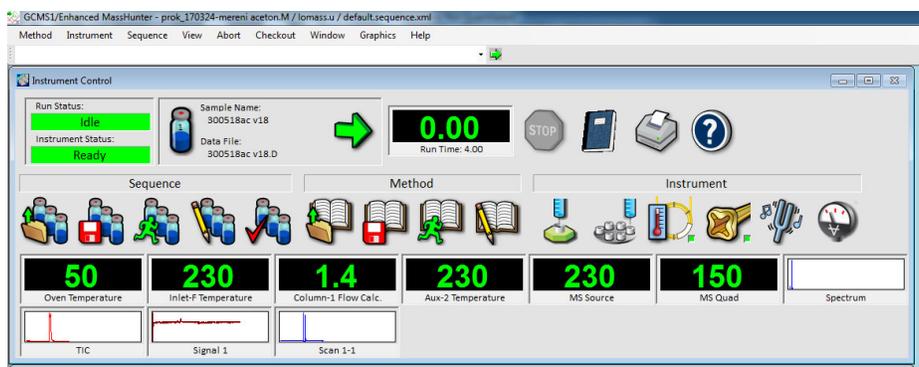


Non-polar chromatographic capillary column HP-5 (5% phenyl 95% dimethylpolysiloxane; 30 m length, 0.32 mm diameter, 0.25 μm thickness of the stationary phase film), installed in the chromatograph, give good performance in a wide range of applications. The column for is splitted parallelly to two detectors: flame ionization detector and *Agilent Technologies 5977A* quadrupole mass spectrometer. The results will be shown as chromatograms: detector signal vs. time. Individual components are visible in the chromatograms as peaks occurred in particular time (time between injection of the sample and time of maximum for a peak is called “retention time”).

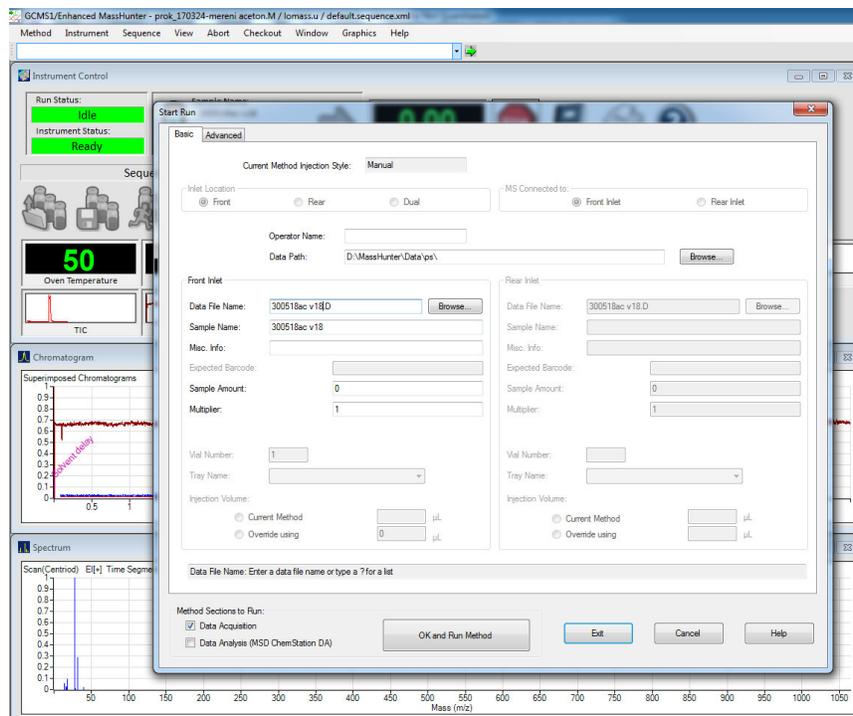
Measurement

Exercise 1: Identification of organic vapors

1. On the upper menu bar of the *MassHunter* software click to **Method > Load Method** and choose method *Exercise1.m*. Wait, till **Status** icons turn to green.



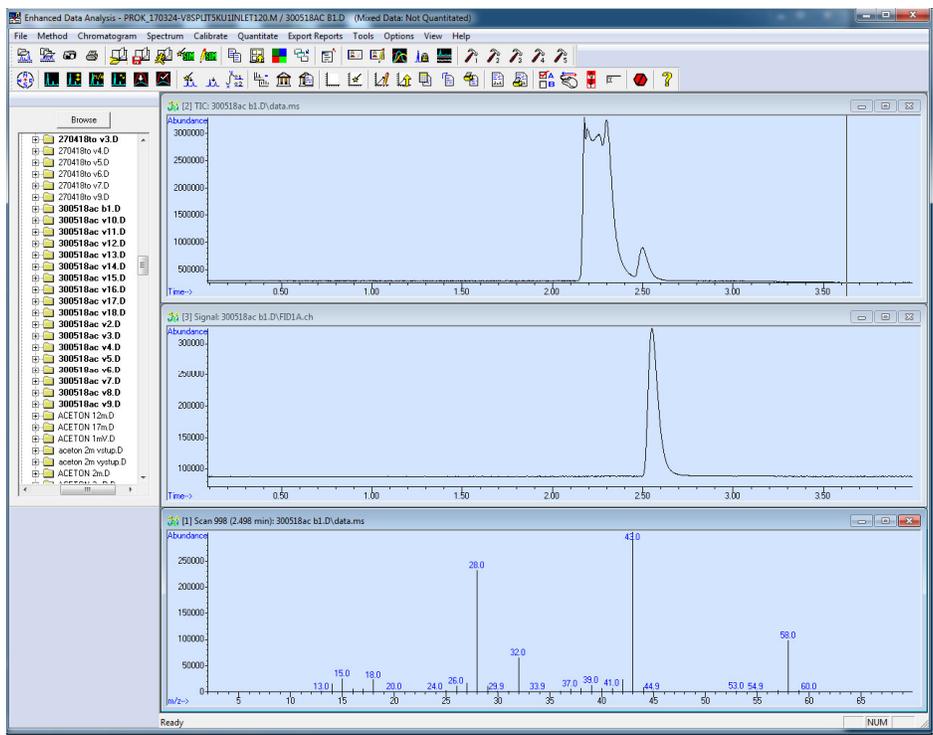
2. Click to **Method > Run Method**, fill the sample name and click to **OK and Run Method** button. Wait till window **GC Acquisition** for valve switching will occur.



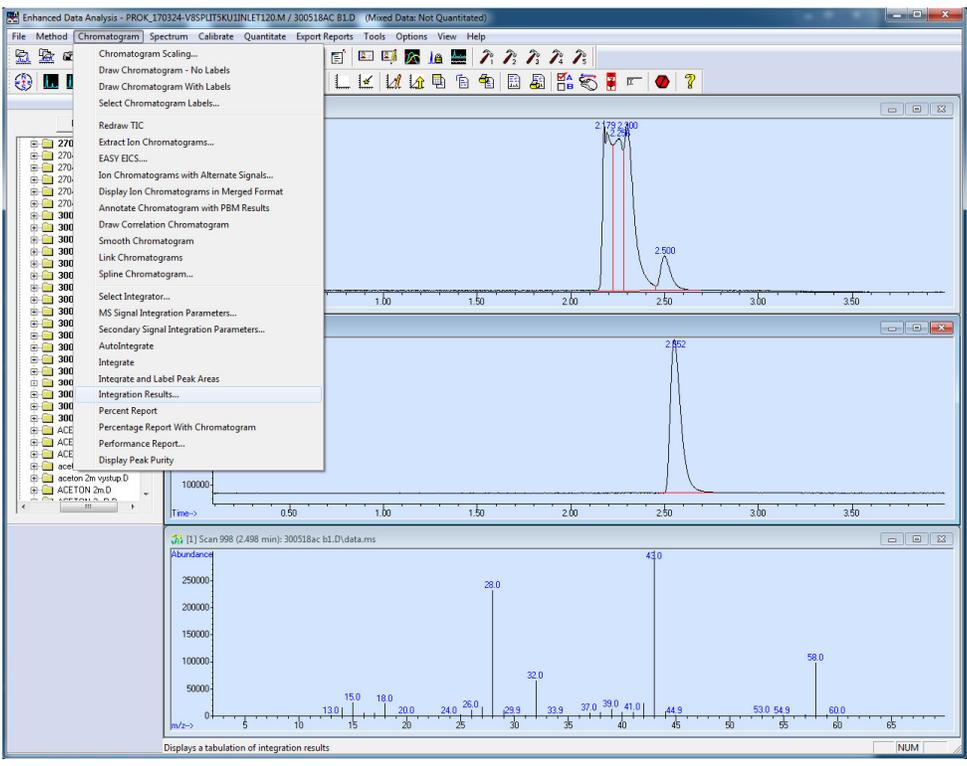
3. Take the organic vapors with 50 ml plastic syringe from bottle containing sample vapors.
4. Inject vapors from the syringe to the plastic tube connected with six port injection valve and then click to **Start run** button on the **GC Acquisition** window for valve switching.



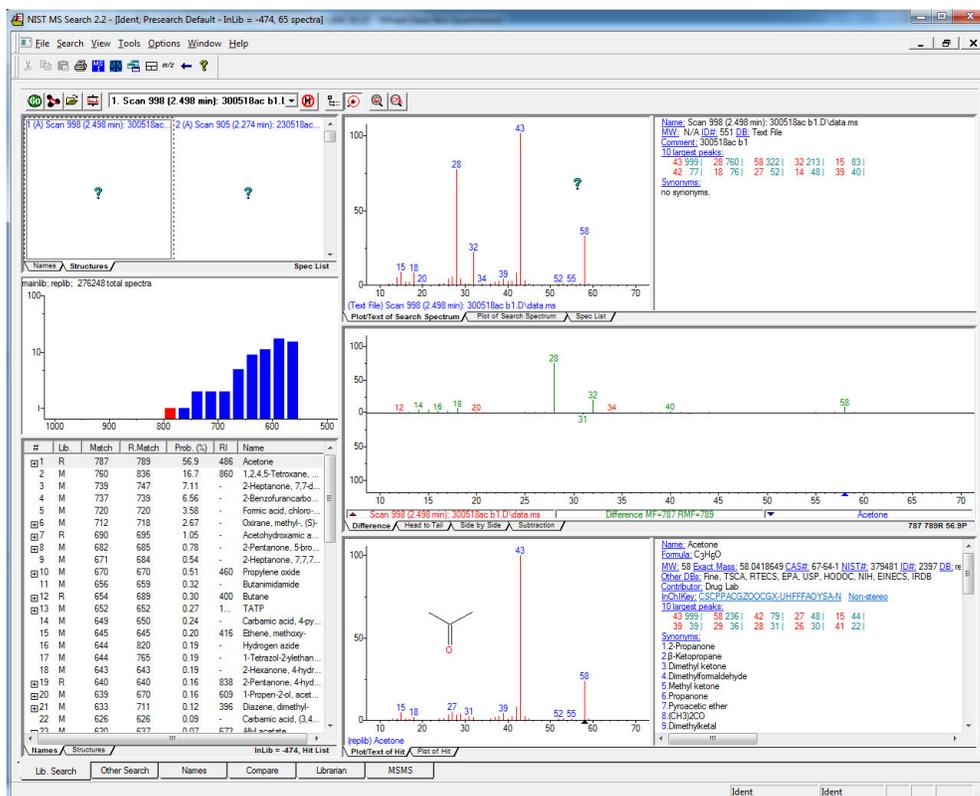
5. When the analysis is finished, click on a green **offline** icon in the computer main desktop.
6. Offline version of the **MassHunter** software window will open and then click to **View > MSD ChemStation Data Analysis** in the main menu bar and data analysis window will be opened.
7. In the data analysis window browse the sample name in the column on the left part of the window. Both chromatograms, from MS and FID detectors, will occur in the window.



8. In the upper bar click to **Chromatogram > AutoIntegrate** and red baseline and retention time values will occur for the both chromatograms.
9. In the upper bar click to **Chromatogram > Integration Results** and tables with integration results (retention times, peak areas, etc.) for MS and FID chromatograms will occur. Using **Copy** button, the tables can be paste to any text or spreadsheet software.



- Click (right-click) to any peak in mass spectrometry (TIC) chromatogram (upper). Mass spectrum of related compound will occur. Then click to **Spectrum > NIST Search** in the upper bar of the Data Analysis window and most probable reference spectrum will be found in a new NIST window.



Exercise 2: Identification of organic components in the sample solution

- On the upper menu bar click to **Method > Load Method** and choose method *Exercise2.m*. Wait, till both **Run Status** and **Instrument Status** indicator bands turn to green.
- Put the sample vial with solution to position 1 in the autosampler.
- Click to **Method > Run Method**, fill the sample name and click to **OK and Run Method** button. The autosampler then automatically inject the sample and start the analysis.
- The other steps, concerning analysis of results, are the same like in the **Exercise 1**.