
Operating Manual

Fast Total Olefin Analyzer

Manual part number 665.95.201 version 3.0 1998

Software part number 665.95.251 version 05.01

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Warnings in the manual or on the instrument must be observed during all phases of operation, service, and repair of this instrument. Failure to comply with these precautions violates safety standards of design and the intended use of the instrument. Analytical Controls assumes no liability for the customer's failure to comply with these requirements.

WARNING

A warning calls attention to a condition or possible situation that could cause injury to the user.

CAUTION

A caution calls attention to a condition or possible situation that could damage or destroy that product or the user's work.

Safety Information

Throughout this manual, the reader and/or system operator will be required to utilize hazardous chemicals. During the performance or routine maintenance, the reader and/or system operator may be exposed to potentially dangerous electrical voltages and/or other hazards. To reduce the

personal risk involved, the following guidelines are established. These requirements are within accepted standards for general analytical laboratory operation. Please refer to established safety requirements and government safety and disposal for additional information.

Chemical Hazards

WARNING

LIGHT HYDROCARBONS ARE TOXIC. UTILIZE THE CONTENTS OF ALL AC STANDARDS ONLY IN A FUME HOOD. AVOID INHALING FUMES AND CONTACT WITH ANY PART OF THE BODY.

WARNING

LIGHT HYDROCARBONS ARE EXTREMELY FLAMMABLE. THE FUMES AND VAPORS FROM LIGHT HYDROCARBONS ARE EASILY IGNITED. KEEP THE CONTENTS OF ALL AC VIALS AWAY FROM FLAMES, SPARKS, OR SOURCES OF HEAT.

WARNING

HYDROGEN IS A FLAMMABLE GAS. IF HYDROGEN OR ANY OTHER FLAMMABLE GAS IS USED, PERIODIC LEAK TESTS SHOULD BE PERFORMED. BE SURE THAT THE HYDROGEN SUPPLY IS OFF UNTIL ALL CONNECTIONS ARE MADE AND ENSURE THAT THE INLET FITTINGS ARE EITHER CONNECTED TO A COLUMN OR CAPPED AT ALL TIMES HYDROGEN GAS IS PRESENT IN THE INSTRUMENT. DO NOT USE PLASTIC OR RUBBER GAS LINES FOR HYDROGEN.

CAUTION

All gases are dangerous when compressed. Do not store cylinders where they might be dropped or exposed to excess heat. Always direct the flow of compressed gases away from people.

Electrical Hazards

WARNING

DANGEROUS ELECTRICAL VOLTAGES ARE PRESENT IN MANY PARTS OF THE SYSTEM, EVEN WITH THE POWER SWITCHES TURNED OFF. UNPLUG ALL THE MAIN POWER CORDS FROM THE POWER SOURCE FOR ALL THE SYSTEM COMPONENTS PRIOR TO SERVICING ANY OF THE SYSTEM COMPONENTS.

Other Hazards

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Pre installation

Pre-installation Requirements

This chapter contains pre-installation procedures for the Analytical Controls Fast Total Olefin Analyzer. Because the system is based upon the HP 6890 Series gas chromatograph (GC), make sure your laboratory meets the environmental, weight, power, and gas requirements as described in the *Site Preparation and Installation Manual* for the HP GC. This section contains additional requirements, a pre-installation checklist for the HP/AC Gas Analyzers, and a record form.

Pre installation
Gas Requirements

Gas Requirements

Table 1-1. Gas Purity Recommendations

	Carrier Gas	Flame Ionization Detector Support Gases		Switching Air
Gas type	Helium	Hydrogen	Air	Air
Pre-pressure	5 bar 75 psi	3 bar 45 psi	5 bar 75 psi	3 bar 45 psi
Percent purity	99.995%	99.995%	Zero-grade or better	(water- and oil- free) from a compressor

The columns are operated at high temperatures. Oxygen in the carrier gas will result in rapid oxidation of the stationary phase of the columns, causing short lifetime. The installation of a high quality filter is suggested to reduce the oxygen content of the carrier gas. Oxygen will diffuse through certain types of diaphragms, valve seals, etc. We recommend that the filter should be located near the gas chromatograph.

NOTE

The FID is very sensitive to hydrocarbons. If these compounds are present in any of the gases in quantities greater than trace amounts, baseline noise will result. Charcoal filters will eliminate this source of noise.

Pre installation
Configuration

Configuration

- HP 6890 gas chromatograph
- Packed column inlet
- Flame Ionization Detector
- Auto injector HP G1512A or G1513 (optional with 100 sample tray)
- AC FTO hardware modification

Computer system

HP Vectra PC—The HP Vectra system bundle configuration changes on a regular basis (typically every 6 months or so) to be faster, have a faster computer chip, and have a larger hard disk drive (HDD). The following are system guidelines:

- For one or two instruments, HP 6890 Series GC only:

Supported Configuration	Recommended Configuration
Pentium 75 MHz	Pentium 133 MHz
32 MB of memory	40 MB of memory
- For three or four instruments, either an HP 5890 Series II or HP 6890 Series GC:

Supported Configuration	Recommended Configuration
Pentium 133 MHz	Pentium 166 MHz
48 MB of memory	64 MB of memory
- For each computer listed above, the following is required:
 - Hard disk drive 500 MB or more, larger disk allows more data storage
 - Floppy disk drive 3.5-inch for software loading
 - CD-ROM For software loading
 - Mouse Supported by Microsoft Windows
 - Printer Hewlett-Packard LaserJet or DeskJet
- - Video monitor SVGA or USVGA

Pre installation
Software

Software

The following software is required for the operation of the AC FTO Analyzer

- Windows 95 or Windows NT
- HP ChemStation G2070AA rev. A.04.02 or A.05.02 software
- AC FTO software rev. 05.01 or higher

NOTE ChemStation rev 04.02 can only be used with Windows 95. ChemStation 05.02 can be used with Windows 95 or Windows NT.

NOTE When using Windows NT, the computer's HP-IB card must be of type 82341C and the HP-IO library software must be installed.

NOTE The HP G2070AA ChemStation software consists of "core" software plus one HP GC instrument driver (either for the HP 5890 Series II or HP 6890 Series GC). Each additional HP GC requires the HP G2071AA additional instrument software, up to a maximum of three add-ons for four instruments per system.

Pre installation
Materials

Materials

The following materials are required to operate the **FTO** analyzer :

- Glass auto sampler vials, caps and a cap crimper. (Available from Hewlett Packard)
- Glass funnel to transfer solutions
- Disposable glass Pasteur pipettes and bulbs (Available from Fisher Scientific)
- Volumetric glass pipettes, 1, 3, 5, 10 and 20 ml capacity.
- Rubber bulb for pipettes.

Reagents

- Iso-octane (2,2,4 tri methyl pentane), olefin free as a solvent

Training for Operation and Maintenance

The person operating the system must be at the level of a Windows basics training course. There are numerous books, tutorial CD-ROM's and training classes to teach basic Windows skills. Windows basics will not be dealt with in this manual.

The person operating the system must have a working knowledge of the ChemStation software.

The person performing maintenance of the chromatographic system must have at least a chemical laboratory education have the level of a ChemStation Operation course.

The person performing maintenance of the computer system must have the level of a Windows Core Technologies or Windows Administering course.

Pre installation
Recording Important Numbers

Recording Important Numbers

In any verbal or written correspondence with Analytical Controls concerning your FTO analyzer, you may be asked the following information :

FTO software Revision Code

FTO software License No.

HP G2070AA Revision Code Example A.04.0X

HP G2070AA License No.

Microsoft Windows Type and Revision Code

Microsoft Windows License No.

HP Gas Chromatograph Serial No.

HP Sampler Serial No.

HP Computer Serial No.

Take a few minutes now to fill in the information. The revision code is found on the diskettes. The license numbers are found on the documentation shipped with the software.

Introduction

This chapter contains installation procedures for the Analytical Controls Fast Total Olefin Analyzer for the HP 6890 Series gas chromatograph (GC). Refer to the *Site Preparation and Installation Manual* of the HP GC for installation information.

Installing the hardware

Before starting with the installation, make sure that the requirements as set in the pre-installation requirements are met. Details on the installation of the GC and sampler can be found in the *Site Preparation and Installation Manual* from Hewlett Packard. Details on the PC can be found in the PC documentation.

- 1 Position the GC on the bench
- 2 Position the sampler and sampler controller
- 3 Connect the sampler to the GC
- 4 Connect the carrier, FID and cooling air gases and check for leaks
- 5 Position the PC and printer on the bench
- 6 Connect keyboard, mouse and display
- 7 Connect the PC to the GC with an HP-IB cable
- 8 Connect power cords and turn on the sampler, GC and PC

Installation

Electrical Installation

Electrical Installation

The electrical configuration is illustrated below. The electrical connections to make are described in the following sections.

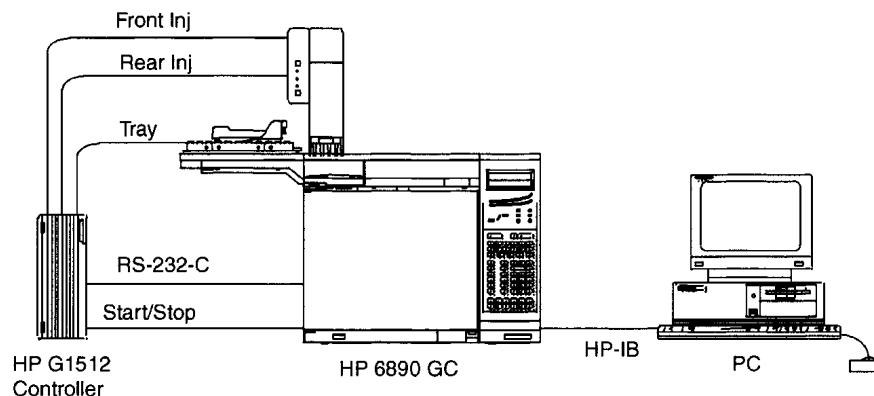


Figure 2- 1

HP-IB loop

Connect the HP-IB port of the computer and of the GC with a HP-IB cable.

NOTE

If you have additional HP-IB devices (GCs and samplers) connected to the computer, link the HP-IB cables in sequential order. **Do not** connect all HP-IB cables onto each other. See the HP getting started manual.

Sampler

Refer to the G1512 auto sampler manual. The connections described here are valid for HP G1512 samplers. Older sampler versions require a different injector mounting plate and a remote start/stop cable. Refer to the documentation of the sampler or contact AC or HP for information.

- 1 Connect the GC remote connector with the Sampler APG remote connector
- 2 Connect the GC sampler connector with the Sampler HP6890 series connector
- 3 Connect the auto sampler injector to the auto sampler controller.
- 4 Connect the auto sampler tray to the auto sampler controller.

Installation
Gas connections

Gas connections

Make gas connections as in the picture below. The connections are located on the top-rear of the 6890 GC. The connections are made on the EPC modules for all gases except the air for the valves and the cooling air, which are made to connections on the rear-right of the GC.

Verify that the gases are of correct purity, as specified in the pre-installation requirements.

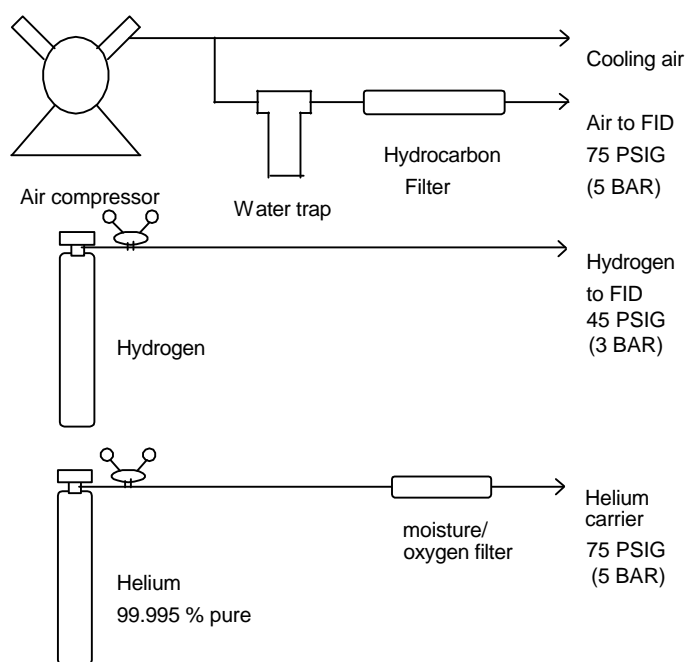


Figure 2- 2 Gas Connections

Installing and configuring the software

Make sure before doing any installation if the computer meets the specified requirements as described in chapter 1. Note that when more than one instrument is installed, additional memory is required. Please refer to the Hewlett Packard documentation for specifications.

All software has been installed at AC prior to shipping so normally no software installation is required. If (re-)installation is required, refer to the appropriate manuals for the installation of the Operating system (Windows 95 or Windows NT) ChemStation (04.02 or 05.02) and if necessary the HP-IO library software.

Installation of the Operating System

The system is shipped with the operating system already installed and in general installation is not required. There are several options for installation of the operating systems which will not be described here. Please refer to the appropriate Microsoft documentation. For Windows NT, make sure that service pack 2 or 3 is available (at least service pack 2 is required for ChemStation).

If for some reason re-installation of the operating system (Windows 95 or Windows NT) software is required, it is often advisable to format the hard disk first. Before doing so, make a backup of the information on the system (datafiles, sequences, methods and so on). Write down the configuration of the installed PC cards with IRQ and address specifications.

Installation of the ChemStation software

The system is shipped with the ChemStation software already installed and in general installation is not required.

NOTE

For HP-IB cards of type HP82341 the HP-IO library software must be installed. This card is required when running Windows NT. Make sure that the "SICL interface name" is set to hp82341 (no capitals) and that the "base address" is set to 30 in the IO config program. The default settings for this card are incorrect and the computer will not communicate with the GC.

NOTE

When using a hp82335 HP-IB card, the memory address range from DC00-DFFF must be excluded. The following lines must be present in the CONFIG.SYS file in the root directory of the boot disk :

```
DEVICE=C:\WINDOWS\HIMEM.SYS  
DEVICE=C:\WINDOWS\EMM386.EXE NOEMS X=DC00-DFFF
```

Installation

Installing and configuring the software

Advanced reinstallation

When re-installation is required, make sure that the current ChemStation is removed. It can be removed and later restored with the following procedure

- 1 Rename the ChemStation directory
- 2 Make a backup copy of the WIN.INI file in the Windows directory
- 3 Remove the [PCS], [PCS,1], [PCS,2], [PCS,3], and [PCS,4] sections from the WIN.INI file.
- 4 Make a backup copy of the AC.INI file in the windows directory
- 5 Delete the AC.INI file
- 6 Reinstall ChemStation
- 7 Configure ChemStation
- 8 Reinstall the FTO software

You can revert to the original situation by

- 1 Deleting the HPCHEM directory
- 2 Renaming the renamed HPCHEM directory back to HPCHEM
- 3 Copying the backup of the WIN.INI and AC.INI file over the modified files.

Installation
Installing and configuring the software

Configuring the ChemStation instrument

Follow the guidelines for configuring the GC and sampler as described in the appropriate Hewlett Packard documentation. Set the HP-IB address of the GC on the 6890 keyboard. Configure the instrument on the PC with the “Configuration Editor” program. On 6890 systems, the sampler is recognized by the GC and does not have to be configured with the configuration editor.

Setting the HP-IB Addresses for the HP 6890 Series GC

Each of the devices connected via HP-IB must have a unique address between 0 and 29. Table 2-1 can be used as a guideline.

Table 2-1. Standard HP-IB Addresses for HP/AC ChemStation Systems

Instrument	1	2	3	4
GC Address	1	3	5	7
Sampler Address (only when connected to an HP 5890 Series II GC)	2	4	6	8

Setting the HP-IB Address on the HP 6890 Series GC

- On the GC front panel, press **[Option]**.
- Scroll to communication, and press **[Enter]**.
- Set the HP-IB address according to the table, and press **[Enter]**.

Power cycle the GC for the address to take effect.

Installation
Installation of the FTO software

Installation of the FTO software

The system is configured and tested before shipping so in general installation of the software is not required.

For the installation of the FTO software, the license number and instrument number must be known. The operating system and Chemstation software must be installed and the ChemStation instrument must be configured before installing the FTO software.

The setup procedure consists of the following steps :

- 1 Turn on the PC
- 2 Make sure that no ChemStation instruments are running
- 3 Insert disk with FTO software in floppy drive
- 4 Select "Start" and "Run"
- 5 Type A:\SETUP

The FTO installation program will start

- 6 Type the laboratory name and license number in the appropriate fields and select the instrument to install the FTO software on
- 7 Press OK

The FTO software will be installed on the computer. When the installation is complete, the instrument can be started.

Initial configuration of GC methods

Since at least the serial number is different in the installed methods from the GC system, the GC methods supplied with the system must be resolved. This procedure is performed at AC when the computer is tested at AC.

Resolving a GC method consists of the following steps :

- 1 Load GC method

A dialog box will be displayed that the method needs to be resolved

- 2 Press OK

ChemStation will try to resolve the settings of the different zones from the saved method to the GC

- 3 When a zone is not configured (the icon was not moved from method to GC and remains grayed), try to drag the zone from method to GC

- 4 Repeat step 3 for each zone that is not configured

- 5 Press OK

A screen comes up with the various zones displayed. Each zone can be displayed by selecting the tab of the zone.

- 6 Review the GC method against the method settings as printed in the manual

- 7 Press OK

- 8 Save the method

— Operation

Introduction

The operation of the instrument requires operation of the ChemStation software in the Windows environment. See the information in the chapter pre-installation for training requirements.

The operation of the instrument is subdivided into a startup and configuration phase and a routine operation phase.

The startup and configuration phase determines and optimizes the analytical conditions and checks the instrument performance.

The routine operation phase consists of calibration of the instrument, performing quality control analyses and sample analyses.

Instrument startup

Adjusting flows

Note

Do not adjust flow or pressure controllers upon receiving the instrument. They are adjusted by AC during testing of the instrument.

Recommended gas flow settings are :

He (flow A) : 20 ± 2 ml/min

He (flow B) : 20 ± 2 ml/min.

H₂ (FID) : 30 ml/min.

AIR (FID) : 300 ml/min.

Flows are controlled through the EPC modules on the 6890. Settings can be made through the 6890 keypad or the ChemStation software.

In order to set the flows, first turn on the gases and check for leaks. Then turn on the GC. Turn on the computer and start ChemStation if you want to make changes through ChemStation.

On 5890 systems the flows are mostly regulated by manual flow regulators. The flow must be measured with a flow meter and adjusted at the regulator.

Flows can be measured manually using a bubble flow meter or an electronic flow meter at the FID outlet or the vent outlet.

Flow A is the main carrier gas flow. The outlet of the flow is at the FID.

Flow B is used to back flush components from the ether/alcohol column and the OV275 to vent. The setting is not critical but should approximately equal the A flow. The outlet of the flow is at the vent.

Hydrogen and air are used as fuel gases for the FID. These flows add up to the flow A carrier gas flow. Set the Hydrogen and air flow at the 6890 keyboard or through the ChemStation control options.

Operation
Instrument startup

Optimizing the analytical conditions

The system needs to be tuned for correct separation of the different columns. The procedure determines the following four parameters

- 1 OV-275 cut-time
- 2 Ether/alcohol trap separation temperature
- 3 Ether/alcohol trap cut-time
- 4 Olefin trap separation temperature

The startup procedure consists of the following 5 analyses. Which parameter is determined is described with the method. The methods can be performed in a sequence. Methods START3 and START3A may need to be optimized and rerun.

START1

This method is used to determine the retention times of selected alkanes on the capillary column. This information is used in later startup runs to determine retention times when components leave a column.

START2

This method is used to determine the cut-time on the OV275 column. The cut-time is found by correcting the retention time of decene for the retention on the OV101 column.

START3

This method is used to determine the cut-time and separation temperature on the Ether/Alcohol column.

START3A

This method is used to verify the cut-time and separation temperature of the Ether/Alcohol column for ether analysis.

FTO1

This is the analysis method. During startup phase, the separation temperature of the olefin column is verified.

Operation
Instrument startup

Determine capillary column retention times

Analyze sample 50.17.001 or 50.17.011 in the START1 GC method.

Purpose

This method is used to determine the retention times of selected alkanes on the capillary column. This information is used in later startup runs to determine retention times when components leave a column.

Checkpoints

The criteria for this analysis are chromatographic in nature.

- Decene must elute at 1 minute +/- 0.15 minute (correct average linear gas velocity).
- The peaks must be symmetrical and without tails.
- The peak width must be around 0.04 minute (correct injection and column connections).

Typical result

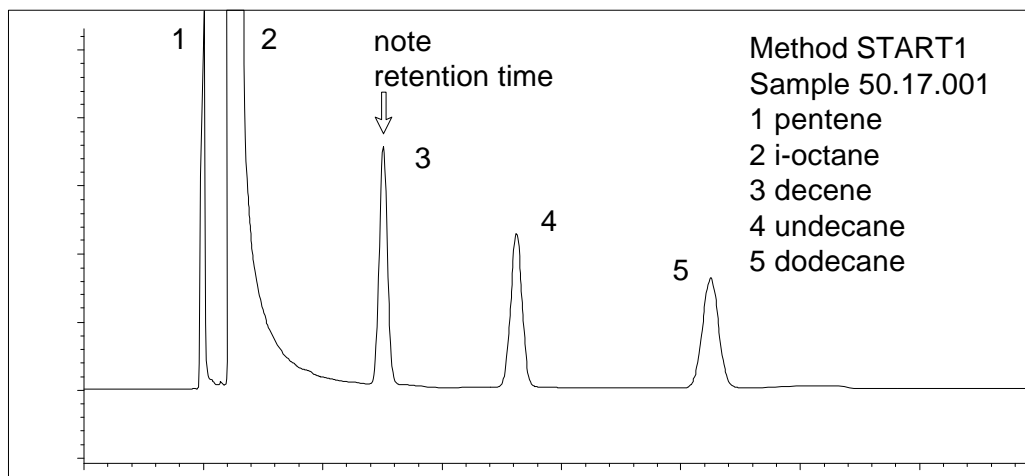


Figure 3- 1 Start1

Operation
Instrument startup

Determining the OV275 column cut-time

Analyze sample 50.17.001 or sample 50.17.011 with GC method START2.

Purpose

This method is used to determine the cut-time on the OV275 column. The end-time of the decene peak minus the retention time of decene in the START1 method yields the cut-time on the OV275 column.

Checkpoints

The criteria for this analysis focus on the retention behavior on the OV275 column. The cut-time on the OV275 column must be between 1.7 and 2.2 minutes.

Typical result

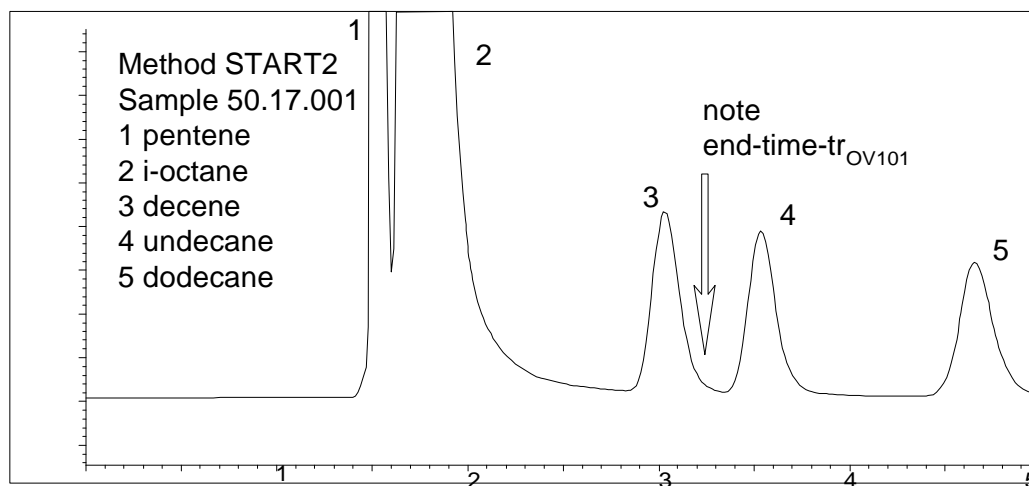


Figure 3- 2 Start2

Operation
Instrument startup

Determining the ether/alcohol column separation temperature

Analyze sample 50.17.001 or sample 50.17.011 with GC method START3.

Purpose

This method is used to determine the cut-time and separation temperature on the Ether/Alcohol column and to verify the cut-time on the OV275.

The end-time of the decene/undecane peak minus the retention of decene in the START1 method yields the cut-time on the Ether/Alcohol column.

Checkpoints

This cut-time must be less than 4.8 minute. When the cut-time is too long, rerun the analysis with a 5°C increased alcohol/ether trap separation temperature.

Typical result

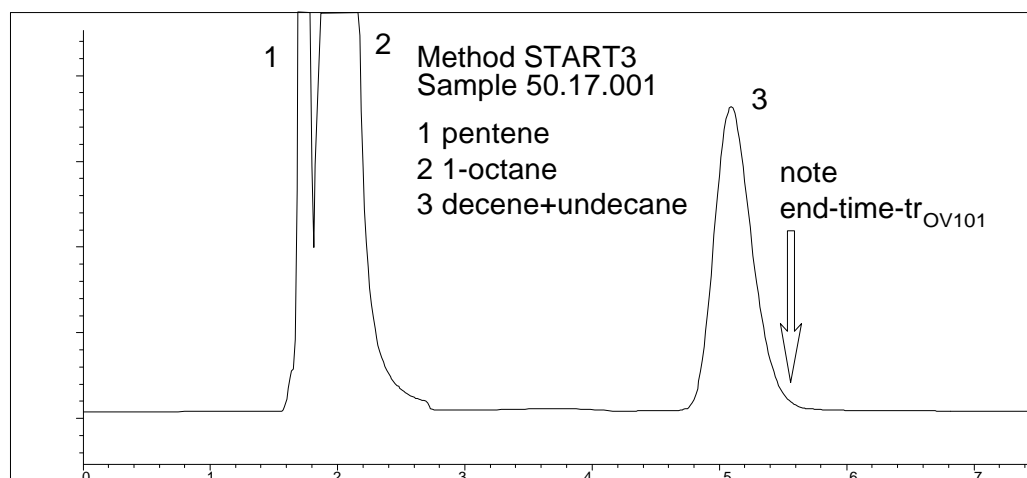


Figure 3- 3 Start3

Note:

When water has been adsorbed on the Ether/Alcohol column, the retention decreases. If the system has not been used for some time, condition the column by heating it to 280°C for 10-15 minutes with the OV275 column in foreflush and the ether/alcohol column to vent (see valve positions for 5890 and 6890).

Operation
Instrument startup

Verifying the ether/alcohol column separation temperature

Analyze sample 50.17.021 or sample 50.17.031 with GC method START3A respectively START3B.

Purpose

This method is used to verify the cut-time and separation temperature of the Ether/Alcohol column for ether analysis.

Checkpoints

The start of the MTBE peak must be later than 5.5 minutes.

Typical result

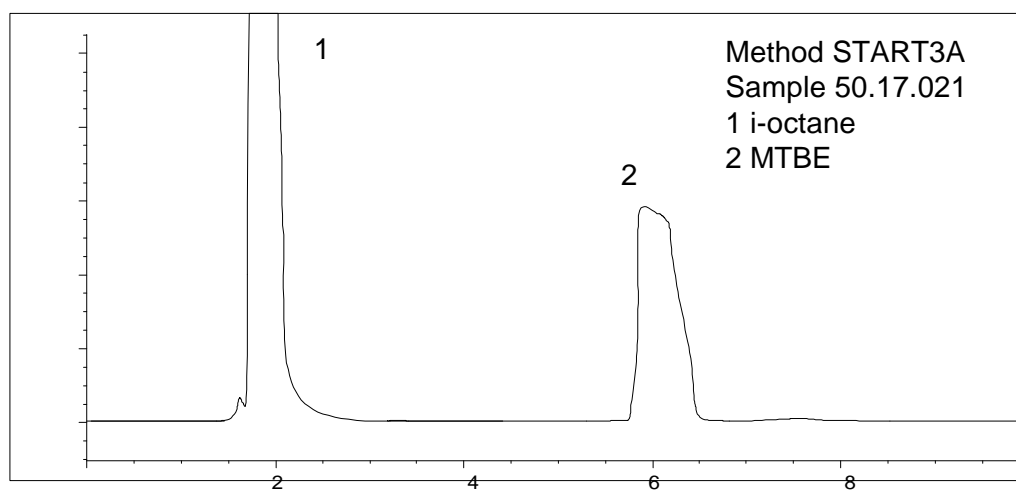


Figure 3- 4 Start3A

Note

If the MTBE peak starts at an earlier retention time, there is not sufficient separation between decene and MTBE and part of the MTBE peak will elute to the olefin trap and affect the analytical results of sample analyses.

Operation Instrument startup

Verifying the Olefin trap separation temperature

Analyze sample 50.17.041 or sample 50.17.051 with GC method FTO1 respectively FTO2.

Purpose

This method is used for several purposes. Initially, it is used with the calibrant to check the elution of different components and makes sure that all components elute on the right location. It is also used for calibration of the system and sample analysis.

Checkpoints

The first three checks are chromatographic checks. The last two checks apply when the sample is run as a calibration analysis.

- The retention time of undecane must be between 7.0 - 7.5 minute and the peak must have completely eluted before 9.5 minute
- There should not be a peak ahead of pentene (break through of MTBE on ether/alcohol column) of more than 0.1%
- The front of the pentene peak must be sharp
- The recovery of pentene must be equal or higher than 90%
- The recovery of decene must be equal or higher than 80%

Typical result

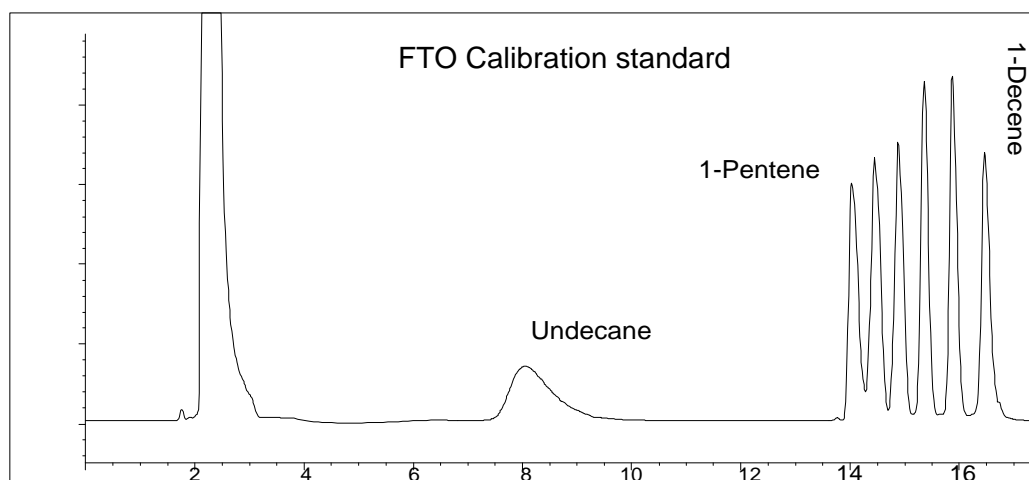


Figure 3- 5 FTO1

Routine operation

Routine operation requires periodical calibration of the instrument, running quality control and sample analyses and verifying the correct

Instrument Calibration

The instrument calibration consists of two steps : performing a blank run to compensate the signal and a calibration run that determines the response factor of the system and the retention times of the olefins.

Blank Analysis

Analyze iso-octane with the FTO1 or FTO2 method and set the sequence table options as follows:

- Set 'Sample Type' to 'Calibration'
- Set 'Cal Level' to 2
- Set 'Update Rf' to 'No Update'
- Set 'Update Rt' to 'No Update'

Purpose

A blank analysis is performed to correct calibration and sample analysis results for instabilities of the signal due to valve switching and column bleed. This calculation option is included in the FTO1 and FTO2 analysis methods. By setting the analysis type to calibration and the calibration level to 2 in the sequence table, the analysis is treated as a blank run.

The blank signal should be relatively flat. It is intended to correct for small variations in the baseline. If peaks or large disturbances are visible, the performance of the system must be verified.

Operation

Routine operation

Calibration

Analyze the sample 50.17.041 or 50.17.051 with respectively the FTO1 or FTO2 method and set the sequence table options as follows:

- Set 'Sample Type' to 'Calibration'
- Set 'Cal Level' to 1
- Set 'Update Rf' to 'Replace' or 'Average'
- Set 'Update Rt' to 'Replace' or 'Average'

Purpose

External standardization is used as the calibration method. This has been configured in the FTO1 and FTO1 analysis methods. Sample 50.17.041 is used for calibration of method FTO1 and sample 50.17.051 for calibration of method FTO2.

The calibration calculations calculate response factors for volume and weight percent calculations. For method FTO1 and sample 50.17.041, the components hexene through nonene are used to calculate the response factor.

The retention times of the olefins are used to correct the signal for the density of the components.

Checkpoints

The following checkpoints apply to the chromatogram and calculated results of the calibration run.

- The retention time of undecane must be between 6.5 - 8.0 minute and the peak must have completely eluted before 9.5 minute
- There should not be a peak ahead of pentene (see note on MTBE) of more than 0.1%
- The front of the pentene peak must be sharp (see note on pentene)
- All olefin peaks must be found correctly in the calibration report
- The recovery of pentene must be equal or higher than 90%
- The recovery of decene must be equal or higher than 80%

Operation
Routine operation

Typical result

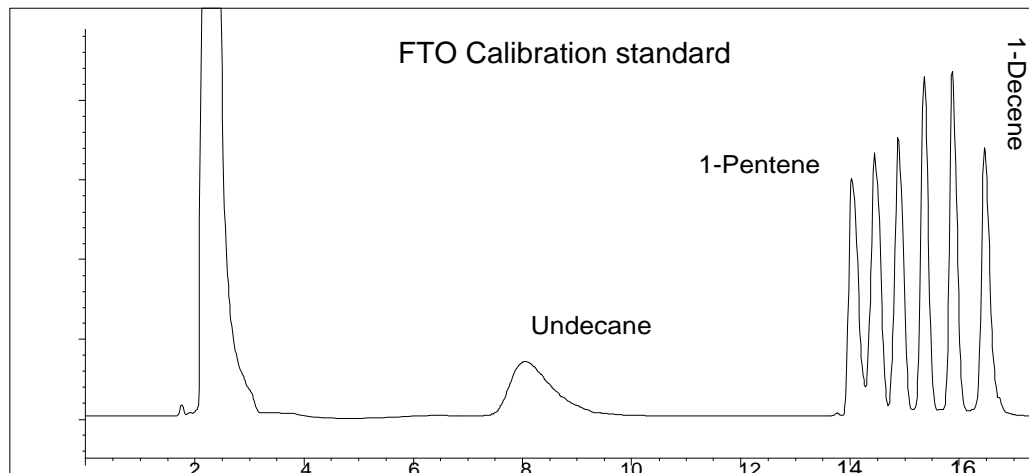


Figure 3- 6 FTO1

NOTE

When the front of the Pentene peak is not sharp, the capacity of the olefin trap may be such that the light olefins in samples are not retained. Decreasing the olefin trap temperature by 5°C may help but the other checkpoints should also be maintained.

NOTE

MTBE is present in sample 50.17.041 and should not be visible in the chromatogram. MTBE may break through the ether/alcohol trap and elute as a peak ahead of pentene. When MTBE is visible, the ether/alcohol trap cut-time should be decreased (verify with START3 and START3A).

Operation

Routine operation

Quality Control

There are two items checked in the quality control procedure. A solvent run is performed to verify detection of low concentrations of olefins. A reference material is analyzed to verify the quantitative performance.

The selection of the reference material depends on the use of the instrument (low olefin samples, gasolines, feedstocks). It should resemble the bulk of samples analyzed in distribution and average concentration of olefins and other matrix components.

For gasoline analysis, the AC sample 65.16.540 can be used as a reference sample. Specifications for the sample must be provided with the sample.

Sample Analysis

Sample analysis consists of a number of steps to prepare the samples, prepare the sequence and run the sequence.

- 1 Prepare the samples by dilution, record the dilution factor
- 2 Set up a sequence - open an existing sequence or create a new sequence
- 3 Fill out the sequence table with the desired samples and the density and dilution information of the samples
- 4 Add calibration runs to the sequence
- 5 Fill out the Sequence Parameters
- 6 Save the sequence with a unique name, so it can later be used for reprocessing when desired
- 7 Run the sequence
- 8 Verify the results - check the calibration runs and inspect the sample chromatograms

Operation

Routine operation

Sample preparation : dilutions

In order to stay within the working range limits of the instrument, samples are diluted with iso-octane. A fixed set of dilutions is described in the chapter 'Analytical Operation'. This is however not a necessity since the dilution factor is entered as a parameter of a sample analysis.

How to prepare dilutions

Dilutions have to be made on a volume basis. When the density is known a mass based dilution can be prepared and a volume based dilution factor can be calculated.

Volumetric preparation

Volumetric glass bulb pipettes provide the most accurate method of preparing dilutions. Do not use adjustable pipettes or glass pipettes for partial outflow since those are less accurate.

The following formula is used to calculate the dilution factor :

$$Dilution = \frac{Volume_{sample}}{Volume_{sample} + Volume_{solvent}}$$

Mass basis preparation

When the density is known from an accepted method the dilution can be made and calculated from the weights of the sample.

The following formula is used to calculate the dilution factor :

$$Dilution = \frac{Mass_{sample}}{Mass_{sample} + \frac{Density_{sample}}{Density_{solvent}} \times Mass_{solvent}}$$

The density of iso-octane at atmospheric pressure and 20°C is 0.691 g/ml.

Operation

Routine operation

Selection of dilution factor

Below is a general dilution selection diagram. On some samples with high amounts of C4 and C5 olefins, the upper limit for the total olefin concentration in the diluted sample has been found to be at maximum 3.5 percent. Guidelines for some specific samples are listed below.

Conventional gasoline (limit 25%)	1:5
RFG phase II gasoline	1:3
Feedstock (FCC naphtha)	1:10
Naphtha / Reformate	undiluted

<< picture not include - error in Acrobat reader >>

Figure 3- 7 Dilution scheme

ChemStation Operation

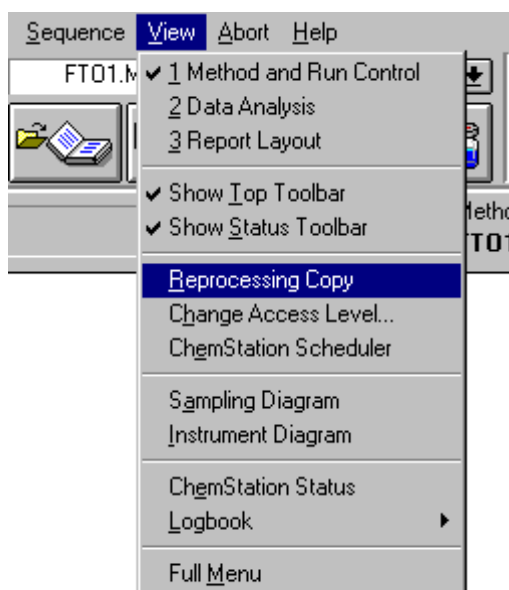
For a detailed description of the ChemStation software, please refer to the manual "Understanding your ChemStation". The following sections describe the basic steps to perform the actions required for FTO operation.

Online and Offline Instrument Control

Usually the Online instrument is started. This instrument is connected to the GC. When loading a method, the method is also loaded to the GC. When changing a setting on the GC, this setting is also transferred to the computer. The Online instrument can be used for all operations of the system. In some cases it is more practical to use an Offline copy.

An Offline instrument can be used to review data, reprocess data, change GC methods without downloading them to the GC, prepare sequences to run with the online instrument.

The picture on the right displays the menu option used to start an Offline instrument.



Method and Runcontrol versus Data Analysis View

ChemStation uses views of the system for specific operations. For operation of the FTO software two views are used : the Method and Runcontrol view and the Data Analysis view.

The Method and Runcontrol view is used for the generation of sequences, control of the instrument settings and performing sample analyses through sequences or single runs.

The Data Analysis view is used to review chromatograms and to set the retention times for the calibration analyses.

Operation
ChemStation Operation

Adjusting the GC method : cut-times and separation temperatures

The cut-times and separation temperatures are the only parameters that need to be changed in the GC method.

Edit cut-times 6890 systems

- 1 Open the 'Instrument' - dialog
- 2 Select the 'Valves' icon
- 3 Select the line with the cut-time to change
- 4 Change the time in the Time field
- 5 Select 'Replace' and make sure that the change is accepted
- 6 Select OK
- 7 Save the method

Edit separation temperatures on 6890 systems

- 1 Open the 'Instrument' - dialog
- 2 Select the 'Aux' icon
- 3 Select the correct temperature zone - Auxiliary 1 for the Ether/Alcohol trap and Auxiliary 2 for the Olefin trap
- 4 Change the initial temperature to the new value
- 5 Select OK
- 6 Save the method

Operation
ChemStation Operation

Creating Sequences

The following procedure assumes that a series of samples has been prepared.

- 1 Create a new sequence or open an existing sequence (template)
- 2 Select 'Sequence' - 'Sequence Parameters...'

Sequence Parameters: FTO

Operator Name:

Data File

Auto **Prefix/Counter**

Prefix: **Counter:**

Signal 1: **Signal 2:** **Counter:**

Subdirectory:

Path:

Part of methods to run

Use Sequence Table Information

Shutdown

Post-Sequence Cmd / Macro

Bar Code Header

Use In Sequence

On a bar code mismatch

Inject anyway

Don't inject

Sequence Comment:

OK **Cancel** **Help**

Figure 3- 8 Sequence Parameters

- 3 Specify a data file subdirectory name and operator name
- 4 Press OK

Operation
ChemStation Operation

- 5 Select 'Sequence' - 'Sequence Table...'

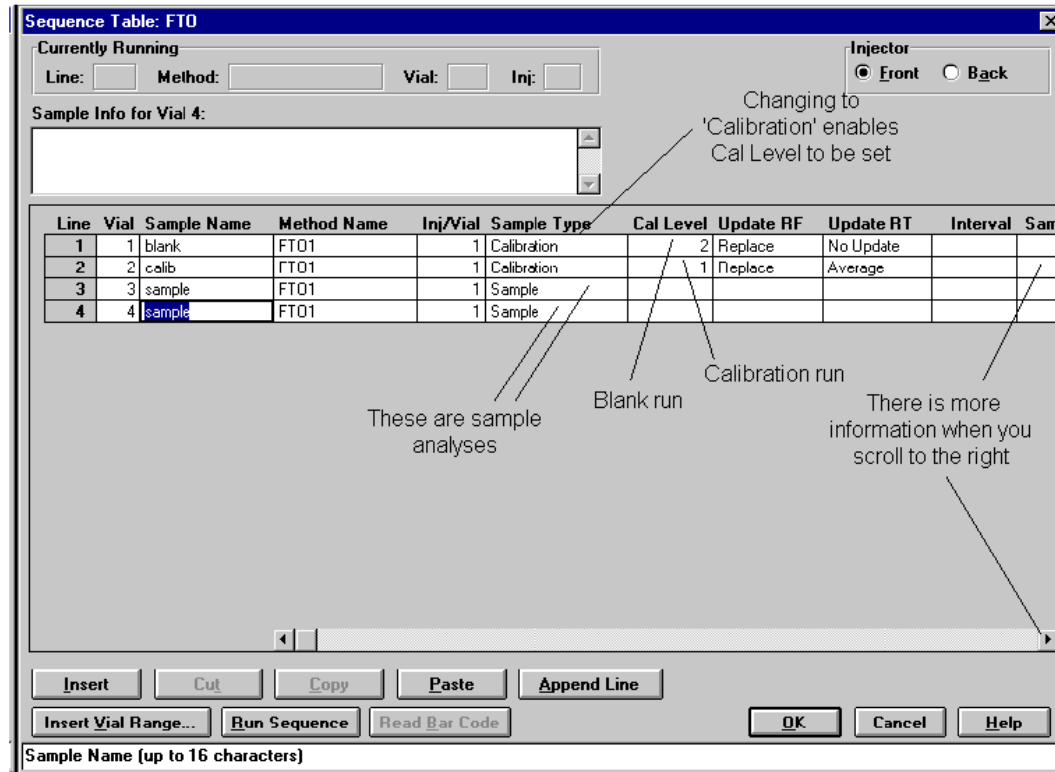


Figure 3- 9 Sequence Table - Samples

- 6 Fill out the samples in the table

Operation ChemStation Operation

- 7 Scroll the table to the right to fill out the density and dilution factor

Sequence Table: FTO

Currently Running
Line: Method: Vial: Inj:

Injector
 Front Back

Sample Info for Vial 6:

Line	Vial	Sample Name	Sample Amount	ISTD Amount	Multiplier	Dilution	Datafile	Inj Volume
1	3	blank						
2	4	calib						
3	6	sample			0.726	0.25		
4	7	sample			0.742	0.25		

Fill out the density here Fill out the dilution here

Insert Cut Copy Paste Append Line
Insert Vial Range... Run Sequence Read Bar Code OK Cancel Help

Each compound result is multiplied with Multiplier in the report.

Figure 3- 10 Sequence Table - density and dilution

- 7 Insert calibration runs with appropriate parameters
- 8 Press OK
- 9 Save the sequence

Operation
ChemStation Operation

Calibration selection in sequence table

By setting the analysis type to calibration and the calibration level to 1 in the sequence table, the analysis is treated as a calibration run. When the calibration level is set to 2, the analysis is treated as a blank run.

By setting the Update Rf parameter in the sequence table to "Replace", the found response factor is used for new analyses. By setting the parameter to "Average" the average response factor is calculated.

When calibrating the system we suggest to run three calibration analyses in the order Replace - Average - Average. This calibrates the system where the response is based on the average of the three calibration runs.

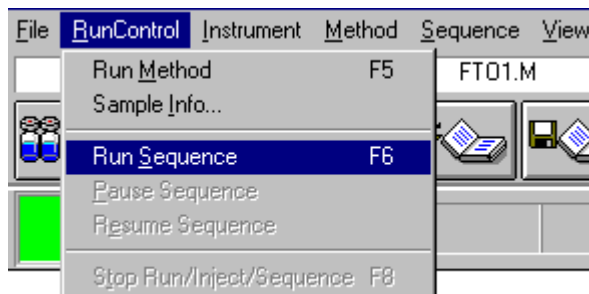
When averaging, each calibration has the same weight. When averaging more than 10 analyses, the new calibration will have a 1/10 weight.

Operation

ChemStation Operation

Running and Controlling sequences

To run a sequence, select 'RunControl' - 'Run Sequence'. While running a sequence, the sequence can be paused after the current run by selecting 'RunControl' - 'Pause Sequence'. Resume resumes a paused sequence. Stop Run/Sequence stops the sequence immediately. Therefore it is often better to Pause the sequence and Stop it when the current run is finished.

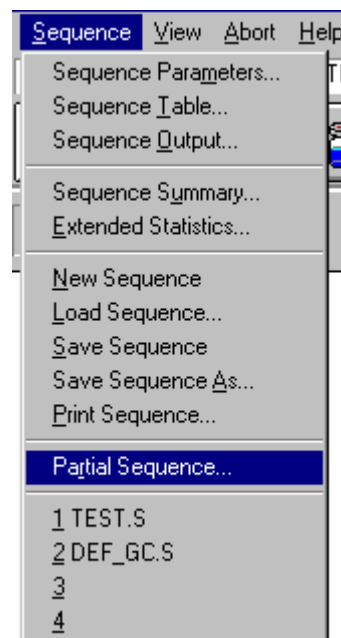


Reprocessing Analyses

Reprocessing of sample analyses can best be performed in an Offline instrument to avoid overwriting existing data files. There are two options. One is to reprocess a complete sequence through 'RunControl' - 'Run Sequence'. The second option is to run a selected number of analyses by running a partial sequence.

After selecting 'Sequence' - 'Partial Sequence' a dialog box is displayed where the desired analyses can be checked. When pressing OK the desired analyses are reprocessed.

Note that first the calibration runs - blank and calibration - must be reprocessed before a sample can be reprocessed.



Note that the dilution and density are not used from the sequence table when reprocessing unless the 'Parts of Method to Run' in the 'Sequence Parameters' dialog is set to 'Reprocessing Only' and the checkbox 'Use Sequence Table Information' is checked.

Verification of Results

In the Data Analysis view, analyses can be loaded and reviewed on screen. The checkpoints for calibrations and sample analysis results can be verified here.

Operation Adjusting Calibration Retention times

Adjusting Calibration Retention times

The retention times of the calibration peaks are updated as selected in the sequence table. When a peak shifts such that it cannot be found, do the following :

- 1 Go to the 'Data Analysis' view
- 2 Load the analysis method (FTO1)
- 3 Load the calibration data file (File - Load Signal)
- 4 Select 'Calibration' - Calibration Table
- 5 Select the arrowglass icon and zoom in on the data file such that the olefin peaks can be seen properly
- 6 Move the selection in the list of retention times of the calibration peaks to the peak that is not recognized correctly (window name 'Calibration Table' - see below)
- 7 Replace the retention time with the retention time of the component (look at the chromatogram) and do this for all components that were not identified correct
- 8 Select the OK button in the Calibration Table window
- 9 Save the method through File - Save - Method

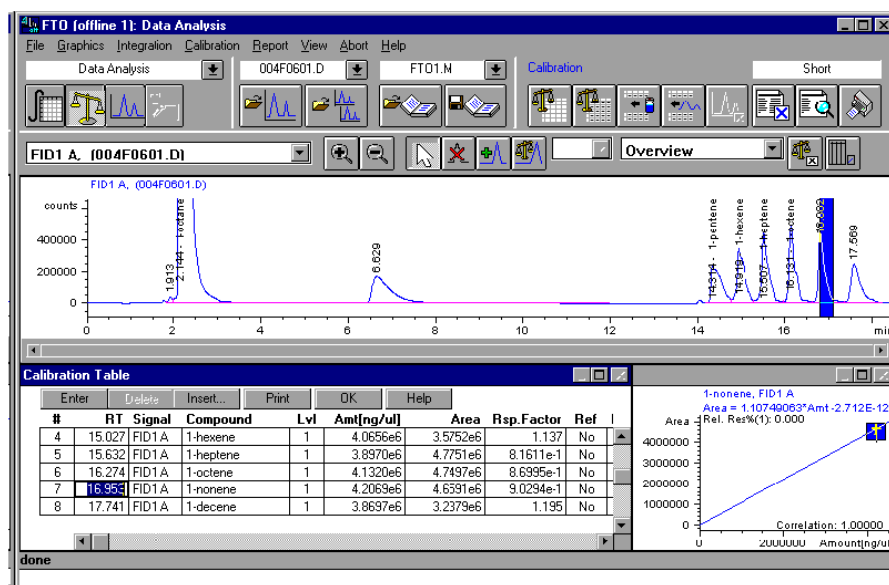


Figure 3- 11 Calibration Table

Preventive Maintenance

Preventive Maintenance
Introduction

Introduction

Preventive maintenance routines are intended to prolong the lifetime of the instrument and to improve the reliability of the results.

Preventive Maintenance
Chromatographic

Chromatographic

The following maintenance schedule is suggested for the chromatographic system. A service contract may provide a routine inspection of the system with a check on the chromatographic performance. The replacement of other parts needs to be performed as needed.

Replace the septum every 100 injections

Replace the liner every 500 analyses

Daily inspect the head pressure

Weekly inspect the chromatographic operation of the instrument

Monthly inspect the flow of the system

Yearly or semi yearly inspect the general condition of the instrument : fan, actuators, gas lines, cabling

Computer System

The most important reason for preventive maintenance on the computer system is to prevent the loss of data and to minimize the effects of a malfunction of the computer system due to hardware failure or software corruption.

With a new computer (part), the installation disks of the software, the license and configuration information and backup of the GC methods, a system can be up and running in two hours. Without either of these items it may take days.

Keep the installation disks of the operating system, ChemStation and FTO software in a safe place. Make notes of the license information of the various software packages. Make notes of the configuration of the instrument.

The information to be stored is the following :

- 1 GC methods
- 2 Data files
- 3 Sequences
- 4 Information you create on the system

There are various methods for backups. Changed GC methods can best be stored on a floppy disk and at least one copy kept with the installation disks.

For storage of the data files, sequences and your own information a tape streamer, ZIP, Jaz or other mass storage backup system is a more practical approach. Backups can also be performed through a network connection.

The frequency of the backup schedule depends on the importance of the loss of data and the time required to create the backup.

Introduction

This chapter describes known chromatographic problems that may occur during operation of the instrument.

Symptoms

The following symptoms are handled in this troubleshooting guide

- The baseline is flat
- There are missing peaks
- Retention times are unstable
- Baseline is unstable
- Higher olefin concentration than expected
- Lower olefin concentration than expected
- Problems setting up decene and MTBE on START3 and START3A
- Problem during calibration with undecane and pentene in FTO1
- Peak ahead of pentene in calibration standard
- Low pentene recovery in calibration standard
- Low decene recovery in calibration standard
- Valves do not switch
- Actuators are leaking air

Flat Baseline

The signal can be zero when the FID is not lit (FID turned off in method) or when the flame goes out because of a valve switch. When there is a non-zero signal it is possible that no sample is injected or the sample does not reach the detector.

- 1 Check if the FID flame is lit. On 6890 systems press the Front Detector button on the GC and verify that the gases and flame are on and that a signal is present
- 2 Check if the syringe is installed and that the plunger is drawn out when the sampler injects the sample.
- 3 Check that the syringe is not clogged. Remove the syringe, manually fill the needle with solvent and empty it on a piece of paper. There must be some solvent on the paper.
- 4 Check the flow through the system at the FID outlet.
- 5 Run the Start1 method with the hydrocarbon mixture 50.17.001 and verify that peaks are present.

Missing Peaks

- 1 If one of the olefin peaks is missing from the calibration run, first verify the correct analytical performance with the START methods.
- 2 Check the head pressure of the A-channel with all columns in flow. A reduced pressure indicates a leak.
- 3 When there is no signal in a section of the chromatogram, verify that the FID flame remains lit. If the flame goes out, check the FID hydrogen, air and temperature settings.
- 4 Check the flow through the system at the FID outlet and verify against the set value when using an EPC controlled system. A difference can be caused by the calibration of the EPC board or a leak.

Retention times are unstable

- 1 Check the head pressure of the A-channel with all columns in flow. A reduced pressure indicates a leak
- 2 Check the flow through the system at the FID outlet and verify against the set value when using an EPC controlled system. A difference can be caused by the calibration of the EPC board or a leak.
- 3 Set the oven temperature to 100°C and let the system stabilize for at least one minute. Set the flow of the front inlet to zero and monitor the pressure. When the Tot. Flow does not return to zero, verify the “Auto Flow Zero” option - see below. Otherwise verify that there is no leak in the system.

Unstable retention times may be caused by the flow-zero option of the EPC module. While the system switches valves and thereby causes pressure pulses in the system, this mechanism may not function properly. To check/turn off the flow-zero option of the EPC, do the following on the 6890 GC front panel :

- 1 Select the “Options” button
- 2 Select the “Calibration” option on the display and press “Enter”
- 3 Select the “Front Inlet” option on the display and press “Enter”
- 4 Make sure that the “Auto Flow Zero” option is set to “Off”

Troubleshooting
Baseline signal is unstable

Baseline signal is unstable

- 1 Verify that the carrier gas is of correct purity.
- 2 Verify that carrier gas filters are not saturated.
- 3 Verify the purity of the FID air.
- 4 Verify that there are no leaks in the carrier gas lines before the GC.
- 5 Run two blank analyses and verify the stability of the two consecutive runs. A more stable baseline indicates the presence of heavy material in the system. Run a number of solvent runs and check if the baseline stabilizes.
- 6 Replace the liner and run a solvent analysis.
- 7 Place carrier gas filters ahead of the GC (active resin filter like Supelco OM2).

Lower Olefin concentration than expected

- 1 Verify that the dilution factor is correct.
- 2 Verify that the correct injection volume has been used.
- 3 Verify that the dilution factor is not too low. When the olefin level in the diluted sample is above 4%, inspect the chromatogram and look for a step decrease at 10 minutes and step increase at 13.5 minutes. Prepare a higher dilution and rerun the sample.
- 4 Verify that no olefins have eluted to the saturated section. This can be inspected visually in the chromatogram. Rerun the sample with a decreased olefin trap temperature or rerun the sample at a higher dilution.

Troubleshooting
Higher Olefin concentration than expected

Higher Olefin concentration than expected

- 1 Verify that the dilution factor is correct.
- 2 Verify that the correct injection volume has been used.
- 3 Verify that the dilution factor is not too high. If the olefin level in the diluted sample is below 0.5%, the blank may introduce an increased olefin concentration to be reported. Use a lower dilution factor when possible for the level of ethers.

Problems setting up decene and MTBE on START3 and START3A

- 1 Make sure that there is no water in the carrier gas. Water affects the separation on the ether/alcohol trap. When some analyses are performed, the water may be driven off and the separation changes.
- 2 Make sure that the ether/alcohol trap is conditioned. When the trap has not been used for some time, it may collect traces of material from the carrier gas stream. Setting the trap 10 minutes to 280°C with Valve 1 set to OFF and Valve 2 set to ON.
- 3 Determine the end of the decene/undecane peak in method START3 and subtract the decene retention time from method START1. If this time is less than 4 minutes, lower the ether/alcohol trap temperature by 5°C and repeat. If this time is more than 4.8 minutes, increase the temperature by 5°C and repeat.
- 4 Determine the start of the MTBE peak in method START3A. If this time is less than 5.5 minute, part of the MTBE peak may elute to the Olefin trap and influences the analytical result. Lower the ether/alcohol trap temperature by 5°C and repeat START3 and START3A.

If the requirements of START3 and START3A cannot be achieved at the same temperature, the selectivity of the trap is insufficient and the trap must be replaced.

Problem during calibration with undecane and pentene in FTO1

This procedure deals with cases where the undecane peak retention time is too long or the pentene recovery is too low.

- 1 If the undecane retention time is too long and/or a peak is visible after decene, increase the olefin trap separation temperature by 5°C and rerun the calibration.
- 2 If the pentene recovery is too low and a fresh calibration vial was used, first verify the correct integration of the pentene peak. If this appears to be OK, lower the olefin trap separation temperature by 5°C and rerun the calibration.
- 3 If after step 2 the pentene recovery increases, some of the pentene peak may have eluted to the paraffin fraction. Verify if the undecane peak retention time is still OK.

If the undecane peak does not completely elute before 10 minutes and the pentene recovery is low, the olefin trap needs to be replaced. When the response factor is still OK (calculated from hexene to nonene, use earlier calibration reports) the system can be used for some time be it that the maximum olefin concentration in the diluted sample needs to be lower (2.5 - 3% maximum).

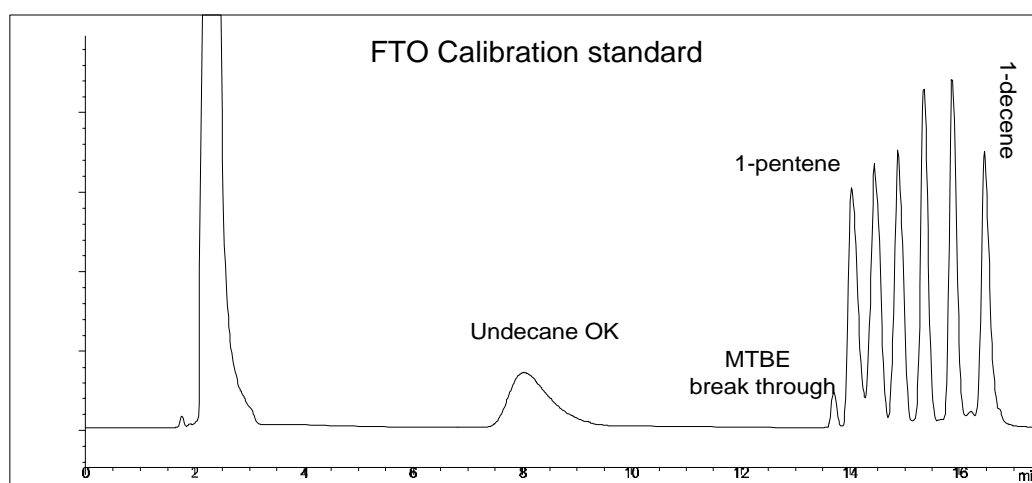
Troubleshooting

Peak ahead of pentene in calibration analysis

Peak ahead of pentene in calibration analysis

A peak ahead of pentene in the calibration standard indicates break through of MTBE, see picture below. Samples may contain C4 olefins and these will also elute ahead of pentene. This can not be distinguished from MTBE.

When MTBE breaks through, the separation on the ether/alcohol trap must be adjusted. Use methods START3 and START3A to determine and check the separation.



Low Pentene recovery in calibration

When the pentene recovery is low in the calibration run, some of the component may have been lost from the vial or may have broke through to the paraffin section.

- 1 Verify that a fresh calibration vial was used for the analysis. Pentene evaporates from a crimp cap vial when the vial is punctured or when it is stored for a long period of time.
- 2 Verify that the front of the pentene peak is relatively sharp. When the front is curved and a step decrease is present at 10 minutes and increase at around 13.5 minutes, some of the pentene may have eluted in the paraffin section. Decrease the olefin trap separation temperature and rerun the calibration standard. Verify that the pentene recovery is OK and that the undecane peak is within the correct retention window.

Low decene recovery in calibration

A low decene recovery in the calibration analysis is often caused by too low Ether/Alcohol column separation temperature or a too short OV275 cut-time. One cause is when the Ether/Alcohol column is optimised when it is not conditioned.

- 1 Run the Start1 through Start3A methods to verify the cut-times and separation temperatures. Adjust the FTO1 or FTO2 method and rerun the calibration standard if adjustments were made.
- 2 Run the calibration standard with a 5°C increased ether/alcohol trap separation temperature. Check if the recovery of decene increases and no MTBE breaks through to the olefin trap (visible as a small peak ahead of pentene).
- 3 Run the calibration standard with a 0.1 minute increased OV275 cut-time and check if the decene recovery increases.

Troubleshooting
Valves do not switch

Valves do not switch

- 1 Verify that the valve air is opened and the correct pressure is applied (3 bar - 45 psi).
- 2 When the actuator has been removed, check if the air lines to the actuator have been mounted in the correct position. Remove the valve air pressure, switch air lines at actuator, reapply pressure and test valve.
- 3 When the actuator has been removed, check if it has been placed correctly. Remove the valve air pressure, remove the air lines to the actuator and manually try to switch the valve. If it cannot be switched, remove the actuator and remount it with the actuator switched to the other position.

NOTE

The valve box is heated. Please turn off the heating to the valve box and let the valves cool to a temperature such that they can be handled.

Actuators are leaking air

The actuators on top of the valve bracket may leak some air. A small air leak is generally not a problem (unless compressed air from a cylinder is used) as long as the pressure and operation of the valve is not affected.

- 1 Verify if all valves operate properly by manually switching the valves. Perform this test as well with all valves set to ON and then manually switching a valve.
- 2 Verify if the air leak is located on the flexible tubing or comes from the rotating pin on the actuator.
- 3 When the air leak is located on the flexible tubing, turn off the air pressure, release the flexible tubing from the actuator, cut off a small section and remount.
- 4 When the air leak is located on the pin on the top of the actuator there may be a leak on the O-ring of the piston inside the actuator. This may occur in one valve position only. The valve needs to be replaced.

Reference

Introduction

This chapter contains reference material for the operation of the Analytical Controls Fast Total Olefin Analyzer.

Precision of Instrument

The following precision data has been obtained during a Round Robin study.

Table 6- 1 Precision

Concentration level (lv%)	repeatability	reproducibility
1	0.1	0.3
3	0.2	0.6
5	0.3	0.8
10	0.4	1.4
15	0.5	1.8
20	0.6	2.2
25	0.8	2.6
30	0.9	3.0
35	1.0	3.4
40	1.1	3.7
45	1.2	4.0
50	1.3	4.4

Reference
Flow Diagram

Flow Diagram

The systems contains four columns. The flow through these columns is directed by the use of three six-port rotary valves. Below is a flow scheme of the instrument and a description of the columns and valves with their purpose.

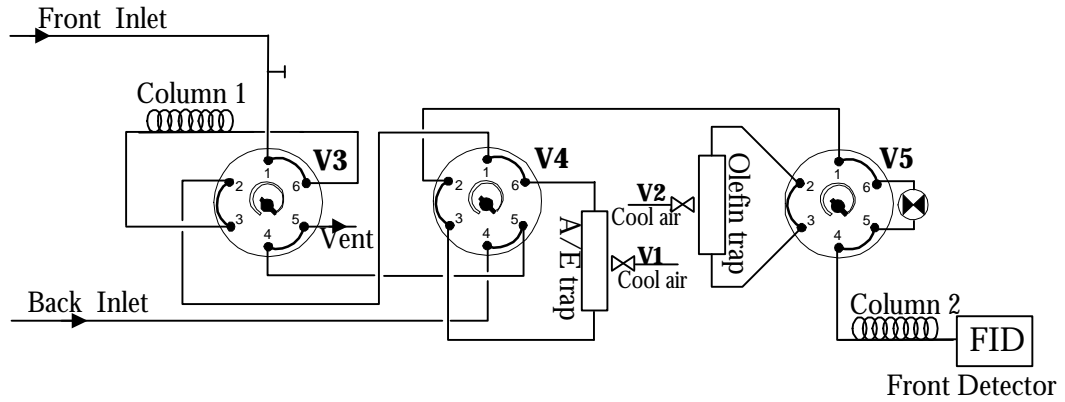


Figure 6-1 Flow Diagram of 6890 GC

Reference
Zone Configuration

Zone Configuration

The following tables list the configuration of the heated zones and valves on a 6890 GC.

Table 6-2 Heated Zones

Heated zone	Location	Control	Operating temperature (°C)	Maximum temperature (°C)
Oven	Oven	OV275 + OV101	60 - 160	160
Inj A	Inj A	Injector	220	N/A
Aux 1	Det B	Alcohol/ether column	110 - 140	280
Aux 2	Inj B	Olefin column	140 - 170	280
Det A	Det A	FID	180	N/A.
Det B	Top middle GC	Valve block	100	180

Table 6-3 Valve Configuration

Valve	Type	Control	OFF	ON
1	solenoid three port	Alcohol / ether column	No action	Cooling of column with compressed air
2	solenoid three port	Olefin column	No action	Cooling of column with compressed air
3	rotary six port	OV275	In flow behind injection port	Backflush to vent
4	rotary six port	Alcohol/ether column	In flow behind OV275	Backflush to vent
5	rotary six port	Olefin column	Stop flow	In flow behind alcohol/ether column

Reference
Column Information

Column Information

The application uses four columns for the separation of the components. A description of the purpose of each column is given below.

Table 6-4 Columns

Name	Stationary phase	Phase loading	Length (m)	Internal diameter (mm)	Operating Temp (°C)	Maximum Temp (°C)
OV275	OV275	30%	3.0	2	160	275
Ether / Alcohol			0.30	3	110 - 130	280
Olefin			0.30	3	140 - 170	280
methyl silicone	cross-linked methyl silicone	5 μ m	25	0.530	50 - 160	325

Reference

Column Information

The polar OV-275 column

The OV275 column separates hydrocarbons into aromatic and non-aromatic hydrocarbons. This column is used as a pre-separation column and is mounted in the GC oven. It retards the aromatic components : benzene (boiling point 80°C) elutes after dodecane (boiling point 200°C). The part of the sample eluting before benzene contains paraffins and naphthenes up to a boiling point of 200°C, all ethers and some alcohols. The eluate from the OV-275 column from benzene on is backflushed to vent.

The Ether/alcohol column

This column is developed to retain ethers while paraffins, naphthenes and olefins pass through. It is developed to retard MTBE behind undecane (almost coeluting with decene). The oxygenates are desorbed at the desorbition temperature to the vent.

The Olefin column

This column separates olefins from saturated components. Olefins with boiling points below 200°C (C_4 to C_{10}) eluting into the olefin column are adsorbed at the separation temperature while saturated components of the same carbon number range pass through. By heating up the column to its desorbition temperature in a later stage of the analysis sequence the column releases the olefins. The column is temperature controlled with a normal separation temperature of 140-160°C and a desorbition temperature of 280°C.

The OV101 capillary column

The OV101 serves to separate the olefins in boiling point order after they are desorbed from the olefin column. This separation is performed to be able to correct for density differences over the boiling point range.

Reference
Analysis Procedure

Analysis Procedure

At the start of the analysis the OV275 and alcohol/ether column are in flow. The olefin column is in stop flow position. Shortly after the injection the olefin column is put in flow and the flow diagram as in Figure 6-2 is obtained.

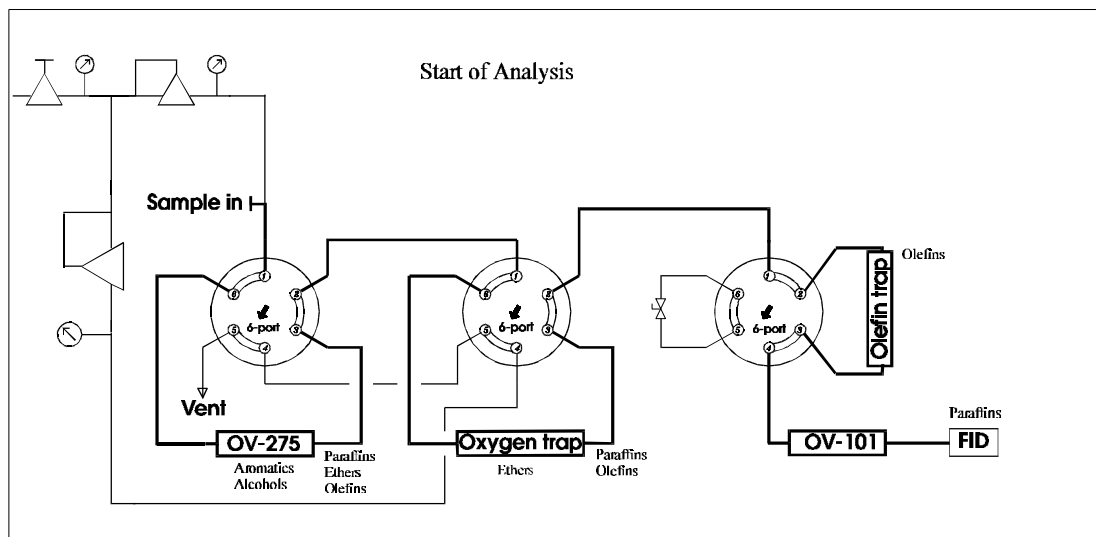


Figure 6-2 Start of analysis

When decene has eluted completely to the ether/alcohol column the OV275 is backflushed and all alcohols and aromatics leave the system through the vent. Separation on ether/alcohol column and olefin column continues (Figure 6-3).

Reference Analysis Procedure

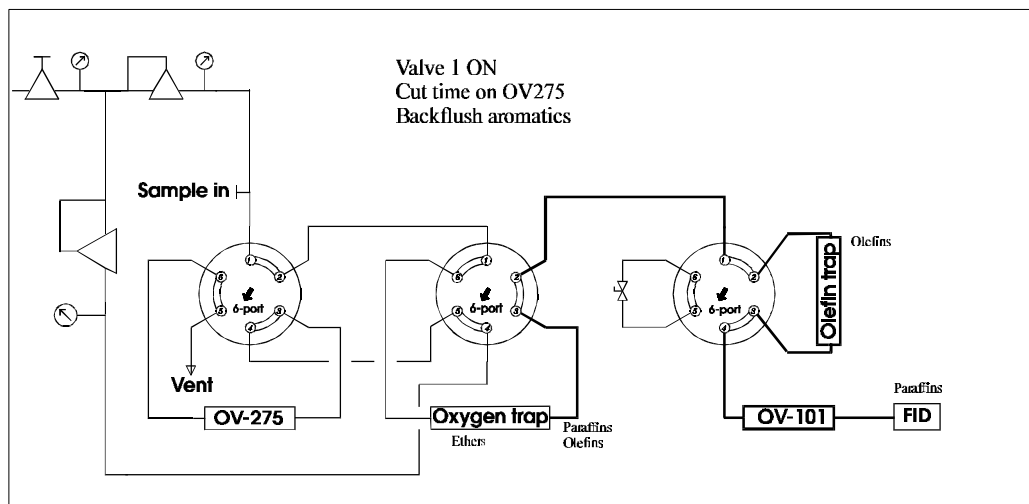


Figure 6-3 Cut OV275

When decene has left the ether/alcohol column this column is also backflushed. Now only paraffins and olefins are present on the olefin column. Paraffins pass through the column over the OV101 to the detector (Figure 6-4). The oxygenated compounds are backflushed to vent.

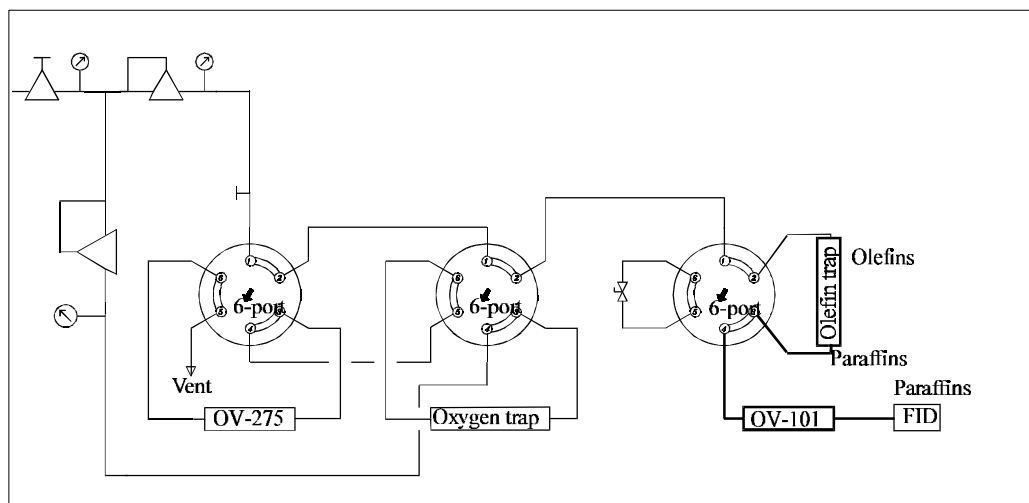


Figure 6-4 Cut Ether/Alcohol column

Finally, when the paraffins have eluted to the FID, the oven is cooled down and the olefins are desorbed from the olefin column by heating it. Then the oven is programmed and the olefins elute by boiling point from the OV101 column to the FID where they are detected.

Reference Calculations

Calculations

The calculation method used is external standardization. From the calibration run the area counts for the olefins are used to determine response factors for volume percent and weight percent calculations (these are listed on the calibration report). These factors are used in sample analysis to calculate the olefin content of the sample. Two results are reported: volume percent and weight percent. For weight percent calculations it is necessary to know the sample density and enter this value as a parameter to the analysis (the multiplier box is used for this purpose).

$$Rf_{\text{volume}} = \frac{\text{Conc}_{\text{ESTD,vol}}}{\text{Area}_{\text{ESTD,vol}}} \quad \text{Equation 6-1}$$

$$\text{Conc}_{\text{volume}} = \frac{Rf_{\text{volume}} \times \text{Area}_{\text{sample,vol}}}{\text{Dilution}_{\text{sample}}} \quad \text{Equation 6-2}$$

$$Rf_{\text{mass}} = \frac{\text{Conc}_{\text{ESTD,weight}}}{\text{Area}_{\text{ESTD,weight}}} \quad \text{Equation 6-3}$$

$$\text{Conc}_{\text{mass}} = \frac{Rf_{\text{mass}} \times \text{Area}_{\text{sample,weight}} \times \text{Density}_{\text{ESTD}}}{\text{Density}_{\text{sample}} \times \text{Dilution}_{\text{sample}}} \quad \text{Equation 6-4}$$

Where :

Rf_{volume}	= Response factor for volume percent calculations, ml/ml per area
Rf_{mass}	= Response factor for mass percent calculations, 1 / area
$\text{Conc}_{\text{ESTD,vol}}$	= Olefin concentration in calibration blend, g/g
$\text{Conc}_{\text{ESTD,weight}}$	= Olefin concentration in calibration blend, ml/ml
$\text{Area}_{\text{ESTD,vol}}$	= Integrated area for the olefin peaks in the calibration blend, g/ml
$\text{Area}_{\text{ESTD,weight}}$	= Sum of density corrected areas (Area peak / density peak)
$\text{Conc}_{\text{mass}}$	= Total olefins, mass%
$\text{Conc}_{\text{volume}}$	= Total olefins, volume%
$\text{Density}_{\text{sample}}$	= Density of sample, g/ml
$\text{Area}_{\text{sample,vol}}$	= Integrated density corrected area of sample, ml/ml
$\text{Area}_{\text{sample,weight}}$	= Integrated area of sample, g/ml
$\text{Dilution}_{\text{sample}}$	= Dilution of sample, ml/ml

Reference
Consumables

Consumables

The content and use of various samples with the methods provided on the instrument are presented.

AC setup and calibration standards

The following table lists the AC part numbers for calibration standards.

Table 6-5 Setup and calibration standards

Part Number	Description	Use method
50.17.001	Setup sample with decene	START1, START2, START3, START3A
50.17.011	Setup sample with nonene	START1, START2, START3, START3B
50.17.021	Setup sample MTBE	START3A
50.17.031	Setup sample ETBE	START3B
50.17.041	Calibration sample MTBE	FTO1
50.17.051	Calibration sample ETBE	FTO2
50.17.510	Setup sample set	Contains 1x 50.17.001, 1x 50.17.011, 1x 50.17.021, 1x 50.17.031
50.17.520	Calibration sample set	Contains 2x 50.17.041, 2x 50.17.051

Reference
Consumables

Setup test mixtures

There are four samples used to set up the instrument parameters for correct operation. These are two hydrocarbon standards and two ether standards, one with ETBE and one with MTBE. The composition of the samples is as follows:

NOTE

Concentration levels listed for setup samples are approximate levels. These samples are for qualitative use only.

Table 6-6 Hydrocarbon test mixture 50.17.001

Component	Concentration
pentene	3.0%
decene	1.0%
undecane	1.0%
dodecane	1.0%
solvent (iso-octane)	94%

Table 6-7 Hydrocarbon test mixture 50.17.011

Component	Concentration
pentene	3.0%
nonene	1.0%
decane	1.0%
undecane	1.0%
solvent (iso-octane)	94%

Table 6-8 Ether test mixture 50.17.021

Component	Concentration
MTBE	5.0%
solvent (iso-octane)	95%

Table 6-9 Ether test mixture 50.17.031

Component	Concentration
ETBE	5.0%
solvent (iso-octane)	95%

Reference
Consumables

Calibration Test Mixtures

There are two calibration mixtures that are used for the calibration of the response and retention times of instrument and as a check for correct operation.

Table 6-10 Calibration Mixture 50.17.041

Sample 50.17.041 lot nr 1						
Component	Density lb/gal	Density g/ml	Weight (g)	Volume (ml)	Wt%	Vol%
pentene	5.384	0.6452	3.12	4.84	0.902%	0.982%
hexene	5.644	0.6763	3.35	4.95	0.969%	1.006%
heptene	5.849	0.7009	3.48	4.97	1.007%	1.008%
octene	5.997	0.7186	3.56	4.95	1.030%	1.006%
nonene	6.116	0.7329	3.64	4.97	1.053%	1.009%
decene	6.209	0.7440	3.68	4.95	1.064%	1.004%
undecane	6.207	0.7438	3.68	4.95	1.064%	1.005%
dodecane	6.276	0.7521	3.72	4.95	1.076%	1.005%
iso-octane	5.829	0.6985	298.97	428.02	86.470%	86.920%
MTBE	6.218	0.7451	18.55	24.90	5.365%	5.056%
Total			345.75	492.43	100.000%	100.000%
lb/gal to g/ml	0.11983					
sample density	0.7021					
Densities from Physical Constants of Hydrocarbons and Non-Hydrocarbon Compounds DS 4B (2nd edition)						
Densities at 60°F in lb/gal (=15.5°C)						

Table 6-11 Calibration Mixture 50.17.051

Sample 50.17.051 lot nr 1						
Component	Density lb/gal	Density g/ml	Weight (g)	Volume (ml)	Wt%	Vol%
pentene	5.384	0.6452	3.09	4.79	0.896%	0.976%
hexene	5.644	0.6763	3.33	4.92	0.966%	1.003%
heptene	5.849	0.7009	3.47	4.95	1.006%	1.009%
octene	5.997	0.7186	3.56	4.95	1.033%	1.009%
nonene	6.116	0.7329	3.64	4.97	1.056%	1.012%
decene	6.209	0.7440	3.66	4.92	1.062%	1.002%
decane	6.121	0.7335	3.62	4.94	1.050%	1.005%
undecane	6.207	0.7438	3.68	4.95	1.067%	1.008%
dodecane	6.276	0.7521	3.72	4.95	1.079%	1.008%
iso-octane	5.829	0.6985	294.58	421.74	85.442%	85.918%
ETBE	6.201	0.7431	18.42	24.79	5.343%	5.050%
Total			344.77	490.86	100.000%	100.000%
lb/gal to g/ml	0.11983					
sample density	0.7024					
Densities from Physical Constants of Hydrocarbons and Non-Hydrocarbon Compounds DS 4B (2nd edition)						
Densities at 60°F in lb/gal (=15.5°C)						

Introduction

Terms

- Calibration level The type of calibration used in the calculation of the results. The normal ChemStation definition is that a sample with different concentration level is used. Here it is used to distinguish between blank and calibration analysis.
- Cut-time The amount of time after injection that a valve is switched in position. Mostly used to change the flow of a column. This is used in routing the components through the system.
- Density The weight of a sample per amount of volume, normally with the unit g/ml or used as specific gravity.
- Dilution The process of mixing an amount of sample with an amount of a non-interfering component (iso-octane is used in the FTO). The amounts have to be measured in volume or weight in order to calculate the dilution factor which is used to calculate the amount of olefins in the original sample.
- Separation temperature The temperature at which a column or trap is operated in order to get a specific separation of components at a certain time.
- Trap A type of column. The word trap is used for columns that are intended to adsorb certain groups of components while other components pass through relatively unretained. Sometimes the words "trap" and "column" are mixed.

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