

## Standard Operating Procedure

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# Agilent 7890 Gas Chromatograph (Thermal Conductivity Detector)

## Introduction

Gas chromatography (GC) is a separation process used to isolate the individual components of various mixture. Gas chromatography (GC) is an analytical technique used to separate and detect and quantify small molecular weight compounds (<1000 Da) in the gas phase. Unlike other forms of chromatography - the mobile phase is a gas, and the components are separated as vapors. The Thermal Conductivity Detector (TCD) is the most universal detector used with a GC. The major benefit is it detects all molecules, and it commonly used for permanent gas analysis (O<sub>2</sub>, N<sub>2</sub>, CO, CO<sub>2</sub>, H<sub>2</sub>S, NO, etc.). The TCD can also detect concentrations from 100% down to around 100 ppm).

## Purpose

The purpose of this document is to introduce the user to the routine operation of Agilent 7890 Gas Chromatograph with a Thermal Conductivity Detector (7890 GC-TCD). This document also outlines the special safety concerns introduced when using the instrument.

## Scope

This procedure applies to established procedures of analysis using the standard configuration (shown below) for the 7890 GC-TCD. Individuals must complete a hands-on one-on-one training session with a TRACES staff member.

## Referenced Documents

- Agilent GC ChemStation: Familiarization Guide ed. 1<sup>st</sup> (p/n 5973-1771)
- Agilent 7890A: GC Advanced User Guide ed.11<sup>th</sup> (p/n G3430-90015)
- Agilent 7890A: Operating Guide ed.3<sup>rd</sup> (p/n G3430-90011)
- Agilent 7890A: Quick Reference ed. 3<sup>rd</sup> (p/n G3430-90009)
- Agilent 7890A: Fundamentals of Gas Chromatography ed. 1<sup>st</sup> (p/n G1176-90000)
- Agilent 7890A: Troubleshooting ed. 6<sup>th</sup> (p/n G3430-90017)

## Responsibilities

### 1. TRACES Users

- 1.1. All Users must obtain training with TRACES Staff prior to system operation. It is the responsibility of the User to ensure they have a good understanding of the instrument and all operation protocols.
- 1.2. If additional training sessions are needed it is the responsibility of the User to schedule these with TRACES Staff.
- 1.3. User must ensure that the helium gas (mobile phase), GC column, and TCD are required for impending analysis are available in TRACES\*.

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1.4. Instrument time must be booked by the User via the online booking system prior to system operation.

### 2. TRACES Staff

2.1. It is the responsibility of TRACES Staff to provide introductory and, if required, subsequent training to all users.

### Equipment (Standard Configuration)

- Agilent 7890A GC TCD\* (Go to **Initial Setup** for more details)
  - .1. Mobile Phase: Helium Gas
  - .2. Detector: TCD
  - .3. GC Column:
    - .3.1. Agilent GS-CarbonPLOT 30m x 0.32mm (3.0um)
    - .3.2. Agilent VF-WAXms 30m x 0.25mm (1.0um)
    - .3.3. Agilent HP5 30m x 0.32mm (0.25um)

### Safety

**IMPORTANT:**

- All the organic solvents used for GC analysis are flammable
- Many permanent gases are flammable
  - Take the appropriate precautions
- Seek assistance from TRACES Staff if you have any concerns

### Initial Setup

1. Ensure the carrier gas (Helium) contains MORE than 500 psi in the cylinder. If you expect a long run time contact the TRACES Lab Manager
2. Turn the regulators to 'OPEN'.
3. Check the gas pressures entering the GC are set to the values below (they are set by the TRACES Staff, please DO NOT Touch)
  - a. Helium :60 psi
4. Power on the GC
  - a. Once a diagnostics test is run, "Power Successful" should appear
5. Start the computer
  - a. Username/Password is given verbally by TRACES Staff
  - b. Click on Instrument 1 Online
6. Confirm that all the Instrument Actuals (components within the GC) \* are not greyed out.

### GC Setup Section

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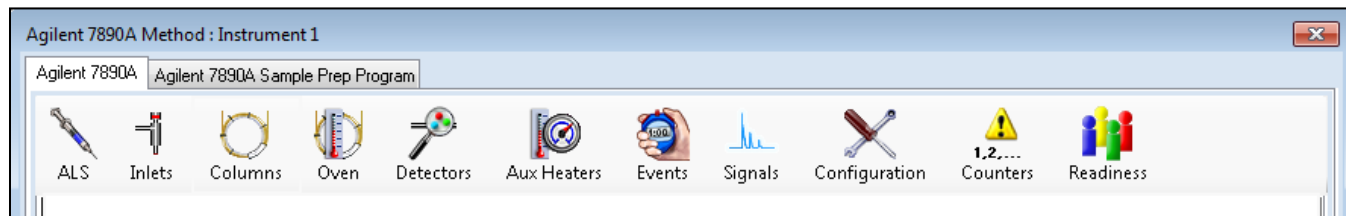


Figure 1

### 7. Load a Method

- a. Click on Method and Load a method from the list provided
  - i. Should include 'TCD' in the method name
  - ii. If you wish to use this loaded method (skip to #10)
  - iii. To create a new method, use the loaded method as a template (go to #8)

### 8. Click on the Instrument Actuals window (Figure 1)

#### a. Manual Injection (using 250uL gas-tight syringe)

##### i. Remove the ALS

1. **This procedure will be done by TRACE Staff members**
2. **ALS will be greyed out (skip to #8c)**

##### b. ALS

- i. Trays: Set by program, do not change
- ii. Sample Overlay: Used particularly when runs are long
  1. Apply when using isothermal runs
- iii. Injector
  1. Set to Back Injector
  2. Set the injector volume between 1-5 uL
  3. Set the number of washes and pumps per injection

##### c. MM Inlet

1. Set Points for:
  - a. Temperature
  - b. Pressure
2. Septum Purge flow
  - a. Rate: 3 mL/min (MINIMUM)
  - b. Mode: Standard
3. Mode
  - a. Split (standard)
  - b. Splitless (low level)
  - c. Pulsed (maximize transfer)
  - d. PTV (Large Volume Injections)

##### d. Column

##### i. Control Mode: **On**

1. Set the control mode. The remaining parameters will be defaulted once this is identified.
  - a. Constant pressure or flow
  - b. Programmable pressure or flow

##### ii. GC Column will be loaded by the TRACES staff

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- e. Oven
    - i. Temperature set
      - 1. Ramped
      - 2. Isothermally
    - ii. Check with the TRACES Manager for the Maximum Oven Temperature
  - f. Detector
    - i. TCD-Back
      - 1. Set (DEFAULT: Do not change without consulting TRACES Staff)
        - a. Heater temperature
        - b. Reference flow (He): 20 mL/min
        - c. Makeup flow (He): 2 mL/min
        - d. Column + Makeup: computer generated
  - g. Events
    - i. Setting time events during the analysis run
      - 1. AO signal range
      - 2. Inlet septum purge
  - h. Signals
    - i. Back Signal (TCD) in the #1 Slot
      - 1. Data Rate :50 Hz/0.004 min (default)
      - 2. Zero is checked (AO at injection)
      - 3. Save is checked (save the signal)
9. Method>Save
- a. Use an exclusive method name
  - b. Loading a previous method and making changes
    - i. Save 'new' method using an exclusive method name

## GC Analysis Section

10. Single Sample/**Manual Injection**
- a. Select Run Control from the Task Bar
    - i. Set the subdirectory (if necessary, a new one must be created)
    - ii. Select the file name using the format; Course/PI/Student\_DDMMYY
      - 1. Counter set to value between 0001 and 9999
    - iii. Enter the vial location
    - iv. Enter the sample name and comment if required
  - b. Run the sample from the Task Bar or the Run Method icon in ChemStation
11. Sequence
- a. Sequence Parameter
    - i. Set a new subdirectory for EVERY new sequence
      - 1. The data may be overwritten
      - 2. Change to prefix/counter and set a base name to id samples
      - 3. Sequence output: As specified by method
  - b. Sequence Table
    - ii. Populate the sections below for each sample:

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1. Vial number
2. Sample name
3. Method\*
4. Injections/Vial
5. Sample Type: Set to sample
- iii. Fill-down Wizard can be used
- iv. Press **Okay**
- c. Press Run Sequence (i.e. Analyze samples)
  - i. Under the Run Control menu
  - ii. On the Sampling Diagram

## Data Evaluation Section

12. Click **Instrument Offline**
13. Select **Data Analysis** tab
14. Click on **File>Load Signal**
  - a. This will load the data file of interest
15. Reports
  - a. Print Report
    - i. Press **Okay** to print to the default printer
    - ii. If the need is to auto print the Report go to #15(c)(i)
    - iii. If the Report is unsatisfactory go to #15(b) and alter accordingly
  - b. Specify Report
    - i. Determine Qualitative Results Info
      1. Calculate: **Percent**
      2. Based On: **Area**
      3. Sorted by: **Signal**
    - ii. Report Style: **Short**
      1. **Add Chromatogram Output** box checked
    - iii. Report Layout for Uncalibrated Peaks
      1. **With Calibrated Peaks** box checked
  - c. Destination
    - i. **Printer** box checked
      1. Reports are auto printed at the end of a run
        - a. Ensure a default printer is available
    - ii. **Screen** box checked
      1. Reports are shown on the screen at the end of run where they can be printed
      2. The subsequent run report will remove the preceding run report from the screen
    - iii. **File** box checked
      1. **File Setting**
        - a. Check the Unique pdf file box

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- b. Check the desired file format
  - i. Files are saved in the \*.D files
- 16. Integration\*
  - a. Using the CS Integrator
    - i. Integrate Manually
      - 1. Using Integration Events
    - ii. Auto Integrate
- 17. Calibration\*
  - a. Calibration Table
    - i. Set the Analyte Name, Level and Concentration
    - ii. Calibrate
- 18. **Method>Save**
  - a. This will transform the Report/Integration/Calibration Results, lock it to the method for successive analysis that use this method will contain the default values saved

\*The TRACES Manager will provide full details during hands-on training.