Varian 3300 Gas Chromatograph
Abbreviated Operating Instructions

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1. Introduction and Startup

[Brackets] denote a button on the instrument
CAPS denote an instrument message on the display

Turn on gas flows: He 80 psi
H₂ 40 psi
Air 60 psi

Turn on instrument with the master switch on the back left-hand side of the instrument.

[Shift] [Instr Test]
This command will run a series of tests including a display test as well as some internal tests. The display will show any error messages if any anomalies are discovered. If an error is discovered, it will be displayed as FAULT ## and the number can be looked up in Volume 2 of the manual in the trouble shooting section for further information. FAULT 12 refers to a low battery. The instrument’s battery is dead and thus this message will always be displayed. After viewing the fault messages, clear them by pressing [Reset].

Pneumatics Panel inside the front door:
Carrier Pressure on Column A: ~25 psi
Injector (make-up?) flow: ~30 mL/min
Detector (make-up?) flow: ?

Ignition of the Detector:
(Make sure that the H₂ and Air are on)
[Shift] [Ignite] and hold for about 5 seconds.
If the status light flashes after this period, the error message is probably “flameout” which indicates that the detector did not ignite. To confirm this, remove the cover over the detector. You can check if the detector is lit in two ways:
1. Place a small vial of cool water over the detector chimney and see if condensation appears on the bottom. If it does, then the detector is lit.
2. Carefully place your finger over the top of the chimney. If the detector is lit you will not be able to hold your finger over it for more than a couple of seconds.

It is important to confirm that the detector is lit before trying to run any samples. Do not depend on the error light to indicate whether or not it is lit. Instead, use one of the previous methods. Once the “flameout” error appears, press [Reset] and try to ignite again. Repeat these steps until you have verified that the FID is lit.

To enter commands and parameters, you will use the entry keypad. Often a yes or no answer will be required. The display will show the currently selected answer. This can be changed by pressing either [yes] or [no] and then [Enter]. Numerical values are also entered via this keypad. [CE] clears your entry. [Prompt/Help] will display allowed values if you are unsure of what to enter. [Enter] enters the selected value.
2. Basic configurations for the GC

[GC Configure]
SET TIMES AND DATE? No.
SET TEMP LIMITS? Yes.

Enter the temperature limits for the column and injector. This varies according to what kind of column and injector is being used but 300°C is probably about right. These values limit how high the temperature can be set while building a program.

There is no auxiliary device, so that temperature can be set at anything (35°C).

Enter the temperature limit for the detector. Again this varies, but 350°C is probably about right.

SET COLUMN STANDBY TEMP
ENABLE COLUMN STANDBY TEMP?

If the standby temp is enabled, then the instrument will return to the specified temperature between each run. I found this to be inconvenient as otherwise the instrument returns to the initial temperature of the program and is immediately ready for the next run, so I always chose “No.”

SET CHECKS FOR GC READY? Yes.
WAIT FOR INJ TEMP READY? Yes.
WAIT FOR AUX TEMP READY? No.
WAIT FOR DET TEMP READY? Yes.
WAIT FOR EXT DEVICE READY? No.
WAIT IF FAULTS PRESENT? Yes.

INITIAL RELAYS AT END RUN? Yes.

This tells the instrument to return to the initial conditions of the program after a run has been completed.

SET LOCK CODE? No.
TURN HARDWARE ON/OFF? No.
OTHER CONFIGURATIONS? No.

GC CONFIGURE TABLE COMPLETE

3. Sample Prep

Prepare the sample: Create a very dilute solution of the sample to be analyzed in the proper solvent. The sample should be fairly clean to prevent heavy, stickier compounds from contaminating the injector and column.

4. Building a Method

[Build/Modify]
PRESS SECTION OR TABLE
[Column]
ENTER STARTING COLUMN TEMP

Enter the initial column temperature.

INITIAL COL HOLD TIME

Enter how long to remain at the initial temperature in minutes after beginning a run. Enter 0 if no hold time is desired.
**TEMP PROGRAM COLUMN?**

A “no” answer will give you a program that remains at the initial temperature for as long as you have set the hold time. A “yes” answer allows you to program up to four temperature changes into the program.

If “yes” is selected:

**PRGM 1 FINAL COL TEMP**

Enter the final temperature of the first ramp.

**PRGM 1 COL RATE IN °/MIN**

Enter the rate at which the instrument will go to the final temperature in °/min.

**PRGM 1 COL HOLD TIME**

Enter how long to remain at the final temperature.

**ADD NEXT COLUMN PROGRAM?**

“Yes” will allow you to program another temperature ramp following the same steps. This can be repeated four times. A sample program follows:

```
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Enter starting column temp. 100
initial col hold time 0
Temp program column? yes
prgm 1 final col temp 150
prgm 1 col rate in °/min 25
prgm 1 col hold time 0
prgm 2 final col temp 210
prgm 2 col rate in °/min 10
prgm 2 col hold time 0
prgm 3 final col temp 220
prgm 3 col rate in °/min 1
prgm 3 col hold time 0
prgm 4 final col temp 250
prgm 4 col rate in °/min 10
prgm 4 col hold time 5
-------------------------------
```

**INJECTOR TEMP**

Enter the temperature for the injector. Make sure this temperature is high enough to make the vaporization of the sample fast and complete.

**INITIAL AUX TEMP**

**INITIAL AUX HOLD TIME**

There is no auxiliary device so these parameters do not matter.

**TEMP PROGRAM AUXILIARY?** No.

**DETECTOR TEMP**

Enter the temperature for the detector. As a general rule this temperature should always be 50° higher than the highest column temperature.

**FID B INITIAL ATTEN**

This sets the attenuation of the detector. This value must be between 1-1024 in powers of 2.

**FID B INITIAL RANGE**

This sets the range of the detector. This value must be between 8-12.

**AUTOZERO ON?** Yes.

**TEMP PROGRAM FID B?** No.

**INITIAL RELAYS** -1 (off)
I am unsure as to what this parameter does, but it seemed to work well with this setup in my research. (–1 and 1 refer to on and off)

**PRGM 1 RELAY TIME IN MIN**

I believe this is when the split injection mode is being utilized. I think that the time in minutes that is entered is the amount of time the instrument waits to purge the injection chamber during the injection. A setting of 0.1 minutes tells the instrument to wait 6 seconds after injection before purging the chamber. Using this means that a larger and thus more reproducible injection volume (1 µL) can be used without overloading the column.

**PRGM 1 RELAYS** 1 (on)

**ADD NEXT RELAY PROGRAM?**  No.

**METHOD COMPLETE-END TIME ** ##.##

The total running time of the method will be displayed at this point.

To make a new program or modify an existing one:

[Delete Program]

**SELECT SECTION** [Column]

**ENTER PROGRAM NO TO DELETE**

Select which program (1-4) to delete. It may be easier to delete the entire program.

**PROGRAM DELETED**

To delete an entire program:

[Delete Section/Table]

**SELECT SECTION** [Column]

**PROGRAM DELETED**

5. Split/Splitless Injection Valve

The split/splitless injection valve that is installed on the GC is set to run in the split mode. This means that a large percentage of the sample volume that is injected is purged from the chamber. The percentage of sample is controlled by turning the valve which is directly in front of the injection ports. When the valve is turned all the way counter clockwise, this is set for the smallest ratio (the largest amount of sample enters the column). If the valve is turned all the way clockwise (8 turns), it is set at the highest ratio (the smallest amount of sample enters the column). I found that for a dilute sample and a 1 µL injection volume, 7 turns clockwise seemed about right with an attenuation of 256.

6. Making the Injection

Rinse the syringe several times with the solvent you are using. Then rinse the syringe several times with your sample. Fill the syringe with about 3-4 µL of the sample solution. Invert the syringe and expel all but 1 µL of solution. Carefully insert the needle into the center of the injection port. You will feel resistance as you pass through the rubber septum. The injector port is equipped with a starter switch that is activated as the syringe comes in contact with the injector. As soon as the starter switch is activated,
inject the sample quickly and withdraw the syringe. Injecting too slowly can cause broad peaks.

At this point the instrument will begin the programmed run. At any point during the run you can toggle between the elapsed and remaining time and the current temperatures by pressing [Enter]. It is also possible to make adjustments to the program while it is running such as adding or subtracting a hold time to allow something to fully elute. This is done by following the exact same procedures as before. As long as the event you are changing hasn’t happened or isn’t finished happening and the change will not result in a shorter run time than is already elapsed, the change will take effect immediately. Otherwise the program will return to the beginning. A run can be stopped at any point by pressing [Reset] and the conditions will return to the initial settings. Remember if you stop the program in the middle to allow enough time for everything to elute before attempting another run.

Once the program finishes running, the conditions will return to the initial ones and then the instrument will go through a stabilization period. This can be monitored by pressing [Status] during this period.

7. Monitoring Instrument Status

- If the instrument is in RUN, the run time can be displayed by pressing [Status] and [Enter] to display the method number, the instrument state, the elapsed run time and the end time.
- A blinking STATUS light indicates a fault condition. When the light is blinking, press [Status] to display the fault message followed by [Enter] to display any additional messages. Refer to Troubleshooting in Vol. 2 of the manual to determine what the fault numbers are. Clear fault messages by pressing [Reset].
- If the instrument is NOT READY, [Status] may be pressed to display the “not ready” conditions which exist. Setpoint errors indicate that the value of a parameter is out of tolerance.

Instrument states

- READY: The READY light is on and the GC is ready for injection. All parameters are within acceptable limits, the plotter is available and no error conditions exist.
- NOT READY: The NOT READY light is on and there are “not ready” conditions which exist. Press [Status] to display these.
- STABILIZE: The time when the last temperature zone is within its tolerance and the instrument allows 2:00 (or as set during configuration of the table) to check that all parameters are steady. If any value goes beyond tolerance during this time, the stabilization time is reset. Pressing [Reset] during this time will skip it and move directly to READY. Pressing [Status] will allow you to monitor the stabilization time remaining.
- RUN: The state at and after injection until the method end time or [Reset] is pressed.