TRACE 1310 Auxiliary Oven: Methanizer Assembly User Guide

This note provides instruction for installing, maintaining, and troubleshooting the methanizer assembly for the TRACE 1310 Auxiliary Oven.

Note This guide updates the sections related to the Methanizer Assembly described in the Chapter 3 and Chapter 5 of the *TRACE 1310 Auxiliary Oven Instruction Manual* PN 31705011.

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Installing the Methanizer Assembly

This section provides instruction for installing the methanizer assembly into the TRACE 1310 Auxiliary Oven. See Figure 1.

Figure 1. Methanizer Assembly



To install the methanizer assembly

1. Remove the protective grid from the back panel by unscrewing the four fixing screws. See Figure 2.



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2. Look for the four slots and for the hole on the back wall of the main oven, and for the four hooks on the back of the methanizer assembly. See Figure 3.



Figure 3. Methanizer Installation (2)

- 3. Insert the methanizer assembly into the back of the instrument, paying attention to guide the OUT tubing into the passing hole provided on the back wall of the main oven.
- 4. Push the assembly until the four hooks enter into the relevant slot, then hook the assembly pushing it downwards. See Figure 4.





5. Connect the ground wire on the bottom of the assembly using the proper fixing screw, then connect the other end of the wire to the common ground point on the chassis of the instrument. See Figure 5.

Figure 5. Methanizer Installation (4)



6. Mount and fix the protective grid provided with the kit. See Figure 6.





 Connect the cables of the heater element and of the thermocouple PT100 respectively to the connectors marked J3 and J9 on the Aux Temp VO/Power Supply board. See Figure 7.

Figure 7. Methanizer Installation (6)



- 8. Fix the mixer on the left wall of the methanizer assembly.
 - a. If not already done, screw the dowel provided into the central hole of the mixer using a T8 Torxhead wrench. Stop the screwing of the dowel when it starts to grip the mixer; the dowel must protrude few millimeters from the central hole of the mixer, do not overtighten. See Figure 8.



b. Screw the mixer into the hole provided on the left wall of the methanizer assembly. See Figure 9.



Figure 9. Mount the Mixer (2)

Methanizer Pneumatic Circuit

The methanizer device is used to determine low concentrations of carbon monoxide (CO) and carbon dioxide (CO_2) obtained by converting these gases into methane, making them pass on a catalytic bed kept at a temperature of 350 °C. Determination of the total quantity of methane developed is carried out using the Flame Ionization Detector (FID).

The methanizer consists of an aluminium block that includes a metal tube filled with a nickel-base catalyst kept in place by a quartz wool. The block is heated to the reaction temperature. The catalyst must be swept by a constant flow of hydrogen.

The hydrogen flow passing through the catalyst allows the reduction of carbon monoxide and carbon dioxide into methane according to the following reaction.

$$CO_2 + H_2 \Rightarrow 350^{\circ}C/Cat \Rightarrow CO + H_2O$$
$$CO + H_2 \Rightarrow 350^{\circ}C/Cat \Rightarrow CH_4 + H_2O$$

WARNING Hydrogen is a dangerous gas that when mixed with air may generate an explosive mixture. The use of hydrogen as a carrier gas or as fuel for certain detectors requires the operator's strict attention and compliance with special precautions due to the hazard involved.



The methanizer device pneumatic circuit is schematically represented in Figure 10.









Note The **mixer** has the purpose of mixing the hydrogen with the sample. Figure 10 shows the mixer anchored to the methanizer assembly. This is the best positioning to facilitate the connection of the fittings.



The **restrictor** is placed on the line of the hydrogen. When installing it, try to minimize the length of the tubing out the main oven. A metal label indicates the column ID value that must be set in the **Auxiliary Column** page.



The hydrogen comes from a line of the **Auxiliary Gas** module connected to the restrictor through an union.

WARNING No fitting where hydrogen flows may be installed into the main oven.





The outlet of the methanizer is connected to the **FID** detector (installed on the TRACE 1310 Auxiliary Oven or on the GC), using the adapter for packed column. Figure 10 shows a FID installed on the TRACE 1310 Auxiliary Oven.



Maintaining the Methanizer

This section provides instructions for replacing the methanizer when the catalyst results exhausted.

To replace the methanizer metal tube containing the catalyst

- 1. Power off the GC and the TRACE 1310 Auxiliary Oven. Refer to the section Shutting down the TRACE 1310 Auxiliary Oven Instruction Manual.
- 2. Remove the protective grid from the back panel of the TRACE 1310 Auxiliary Oven undoing the four fixing screws. See Figure 11.

Figure 11. Methanizer Maintenance (1)



3. Remove the front panel and cover of the methanizer assembly undoing the relevant fixing screws. See Figure 12.

Figure 12. Methanizer Maintenance (2)



Methanizer Assembly Front Panel and Cover

4. Open the heating block of the methanizer by removing the four fixing screws to access the methanizer metal tube containing the catalysts. See Figure 13.

Figure 13. Methanizer Maintenance (3)





5. Carefully extract the methanizer until it protrudes about 2-3 cm from the box, as permitted by the length of the inlet and outlet tubings connected to it.

Do not force the extraction of the methanizer more than the suggested distance.

6. Using a 1/4 in. wrench, disconnect the fittings of the inlet and outlet tubings from the methanizer, then remove it. See Figure 14.



Figure 14. Methanizer Maintenance (4)

7. Replace the methanizer with a new one.

Note The connecting ports of the new methanizer are protected by two finger-tightened plugs. Remove the plugs manually before connecting the inlet and outlet tubings to the methanizer. See Figure 15.

Figure 15. Metal Tube Protective Plugs



a. Connect the inlet and outlet tubings to the connecting ports of the methanizer without overtightening the fittings.

Note Because the methanizer is not polarized, it is indifferent to which ports you connect the inlet and outlet tubings.

- 8. Before reinserting the methanizer into the heating block, perform a leak test. See "Performing a Leak Test" on page 8.
- 9. At the end of the leak test, carefully reinsert the methanizer into the heating block and close it by using the four fixing screws.
- 10. Reinstall the front panel and the cover of the methanizer assembly.
- 11. Reinstall and fix the protective grid.
- 12. Restart the GC and the TRACE 1310 Auxiliary Oven.
- 13. Activate the catalyst. See "Catalyst Activation" on page 9.

Performing a Leak Test

To perform a leak test

- 1. Turn on the hydrogen flow.
- 2. Check the metal tube connections with a handheld electronic leak detector to find possible leaks.

Catalyst Activation



IMPORTANT Perform the activation of the catalyst when the methanizer is installed the first time, and when the methanizer temperature, the flow of hydrogen, or both remain off for a period of time.

To activate the catalyst

Before starting this procedure do the following:

- Turn off the flame of the FID.
- Set the required carrier gas flow rate (helium or nitrogen).
- Set the injector temperature to 100 °C.
- Set the column oven temperature to 40 °C.
- Set the detector temperature to 250 °C.



CAUTION During the catalyst activation, hydrogen is discharged to the atmosphere. Therefore the user must take all necessary precautions concerning the use of gases involved danger of explosion or fire, as provided by the Safety Regulations applicable in the country where the instrument is used.

To activate the catalyst proceed as follows:

- 1. Set the hydrogen flow to 30 mL/min.
- 2. Increase the methanizer temperature (Aux 1 Zone) to 150 °C.
- 3. Maintain the methanizer at this temperature for about one hour.
- 4. Increase the methanizer temperature to 380 °C.
- 5. Let hydrogen flow through the catalyst for approximately two hours to ensure the complete reduction.
- 6. At the end of the activation the catalyst can be repeatedly used without further treatment.

Methanizer Analytical Troubleshooting

Table 1 lists the most common problems with the relevant diagnosis and remedy when the methanizer device is used.

Table 1. Methanizer Analytical Troubleshooting

Symptom	Diagnosis	Remedy
No peaks at all.	Column broken or disconnected.	Check the column and connections.
	Defective electrometer or amplifier.	Replace the electrometer or the amplifier.
	FID flame is out.	Light the flame.
	Poor or missing electrical connections.	Check the cables connections.
CO or CO ₂ peaks not present in the chromatogram.	Methanizer temperature not correct.	Set the correct temperature.
	Catalyst exhausted.	Replace the metal tube containing the catalysts.