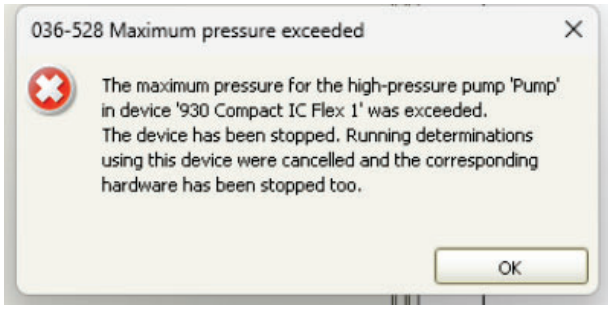
IC Maximum Pressure Exceeded

Troubleshooting IC High Pressure

Technology: Ion Chromatography

High Pressure Error Message 036-528

The below error message will appear, indicating that the maximum allowed pressure was measured, and will turn the hardware off to prevent further damage to the system. Below are some troubleshooting items to help determine the source of the back pressure.



Confirm column, flow rate, temperature, & method parameters

1. Confirm the analytical and guard column types. Check the column manual for typical pressure at standard conditions (temperature, flow rate). Column manuals can be found at: United States | Metrohm
2. Confirm the maximum pressure and flow rate in the method.
    a. Confirm that the flow rate listed in the method does not exceed the column's maximum flow rate. Check the column properties in Configuration for the max flow rate. See Figure 1.
    b. Ensure the maximum pressure matches the column type max pressure. For example—ensure that the max pressure is not listed as 0 in the method.
        i. *Method tab > File > Open. Select Pump on the IC > Check and update Pmax value. Don't forget to save the method.* See Figure 2.
    c. If the max pressure is listed as 0 in the method, update per the column requirements found in Configuration. See Figure 1.
    d. **Contact Tech Support if column properties shows 0 for a Metrohm column with an intelligent chip.**
3. If using an oven for column heating, ensure the temperature has stabilized.
    a. For an **A Supp 7 column**, increased pressure is observed at lower temperatures.
    b. Ensure the oven is on and that the column has reached the recommended oven temperature, which is 45C.

c. Confirm that the pump start-up time in the method is at least 10 minutes in MagIC Net. Go to the *Method tab > File > Open. Select Pump on the IC > Adjust start-up time. Don't forget to save the method.* See Figure 2.

Figure 1. Column Properties in Configuration

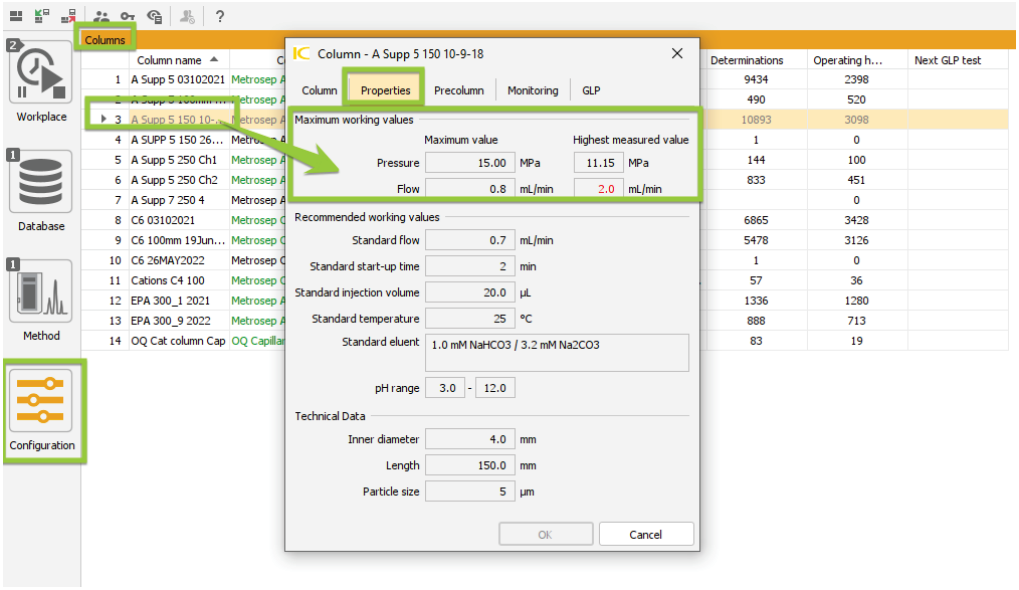
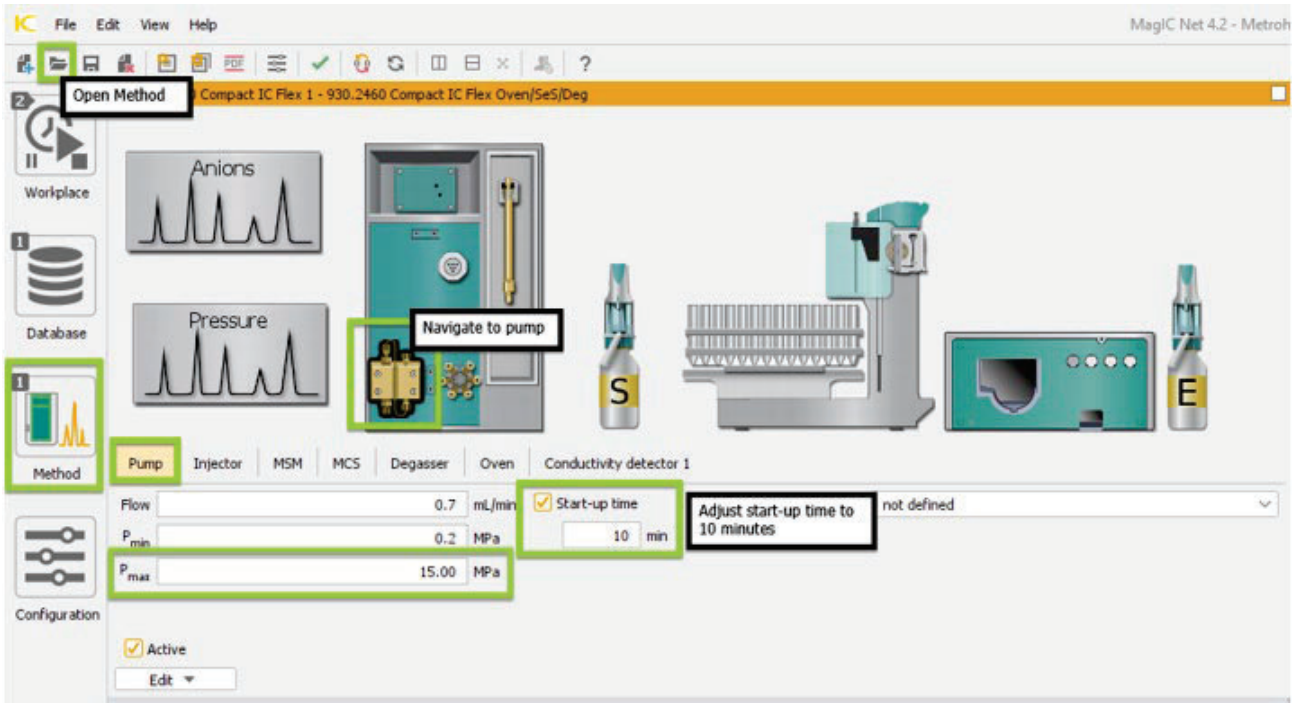


Figure 2. Method Parameters for High-Pressure Pump Pressure and Flow Rate



Troubleshooting High Pressure: At or Before Column

- If the above temperature, Pmax, and flow settings are appropriately defined, and the high-pressure error message is still received – pressure can be measured at each point on the IC to determine the source using Manual Control. See Figure 3.
- Use the below instructions and test points to determine if high pressure originates from at or before the column.
- Most commonly, high pressure is observed at the guard and/or analytical columns. If the pressure measured at the guard or analytical column is high, replace it.
    - If an analytical column needs to be replaced, review this article: How to configure a new IC column in MagIC Net – Metrohm USA

<u>Instructions:</u>

- In Manual Control, choose "All Devices" and set the pump to the normal flow rate.
- Start the pump and disconnect at the numbered points. Record the pressure after stabilizing (~20 seconds). Reconnect the tubing and move to the next step. If the pressure is higher than referenced, you have identified the source of the back pressure.
    1. Column Outlet: Check the column manual
    2. Guard outlet: <1MPa
    3. Guard inlet: <0.5 MPa
    4. Injection Valve, Eluent Out: < 0.2MPa
    5. Injection Valve, Eluent In: <0.2 MPa
    6. Inline filter outlet: <0.1MPa
    7. Inline Filter inlet: <0.1MPa
- Note: Do not check pressure directly at the inlet or outlet of the pulsation absorber, as this may cause it to rupture.
- Note: Pressure limits of capillary tubing
    - PEEK: 20MPa
    - PTFE: 2MPa

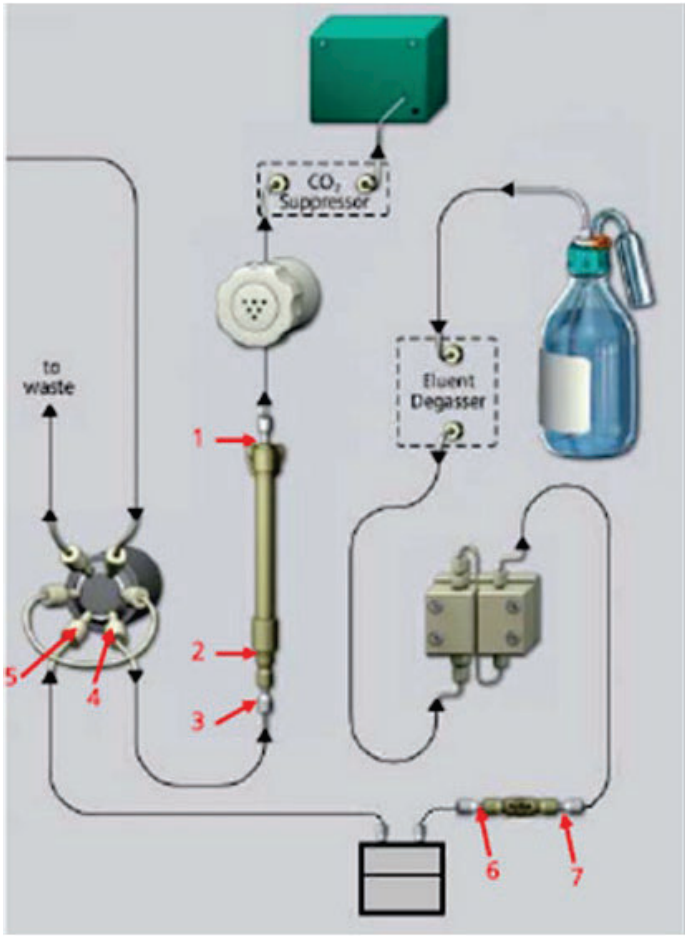
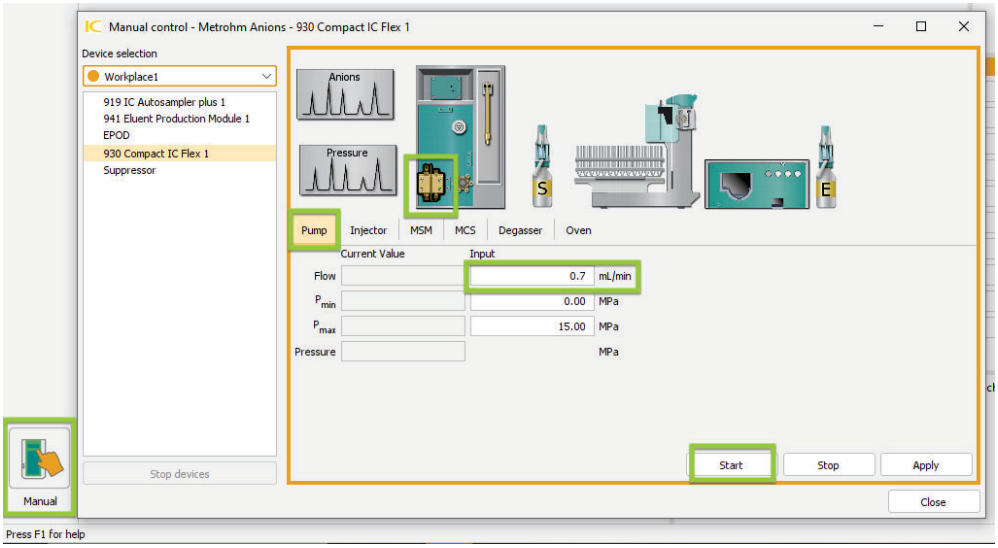
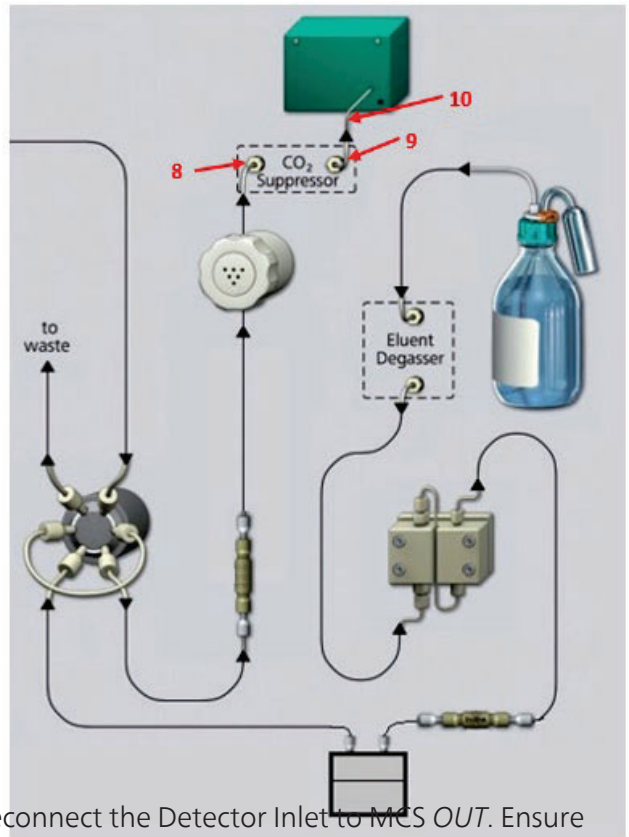


Figure 3. Manual Control



Troubleshooting High Pressure: After the Column

- If pressure measured at or before the column was found to be in the specification, the pressure after the column must be measured.
- Remove the column from the flow path and replace it with the guard column.
- Start the pump at the same normal flow rate as before. Record pressure after stabilizing (~20 sec). If the pressure is higher than referenced, you have identified the source of the back pressure.



<u>Instructions:</u>

8. MSM *OUT* Line / Detector Line: Pressure from outlet guard + <1MPa
   - 8.1. Step MSM in Manual Control to measure Chamber 2 of MSM Rotor after 20 seconds
   - 8.2. Step MSM again in Manual Control to measure Chamber 3 of the MSM rotor after 20 seconds
9. To evaluate the pressure of the MCS: Reconnect the *OUT* line of the MSM to the MCS *IN*. Disconnect at the Out of the MCS. Pressure from 8.2 + 1Mpa
10. To evaluate the pressure of the detector block: Reconnect the Detector Inlet to MCS *OUT*. Ensure that the detector's waste is disconnected from the MSM Rinse line and located in the waste cup.

The conductivity detector waste outlet is PEEK tubing. Added pressure should be no more than 1Mpa from Step 9.

Prevention Measures

1. Problems in IC systems are usually related to particles. These particles can be introduced from bacterial growth, unfiltered eluents, samples, rinsing solutions, and/or regeneration solutions.
2. Particles clog separation columns over time (column pressure increases). Be especially conscious of ensuring that there are no particles present when producing eluents. The eluent continuously flows through the column at a rate of 500 to 1000 mL per workday compared to about 0.5 mL of the sample solution. Filter or dialyze your sample automatically with one of the Metrohm Inline Sample Preparation techniques (MISP) or benchtop methods to at least 0.45 µm.
3. Monitor the pressure of the analytical and guard columns upon installation. Once the pressure increases +1MPa, the guard column should be replaced.
4. Replace inline filters at least quarterly. Filters may need to be replaced more frequently.
5. All eluents must be microfiltered (0.45 µm) immediately before use.
6. Bacterial growth significantly affects chromatography and ruins separation columns. A vast array of problems in chromatography are caused by the growth of algae, bacteria, and fungi. To prevent bacterial growth, always use fresh eluents, rinsing solutions and regeneration solutions. Do not use any solutions that have not been used for a prolonged period. We recommend cleaning all vessels as follows before filling them:
   1. Thoroughly rinse with ultrapure, UV-treated water (> 18.2 MΩ).
   2. Swirl a methanol-water or acetone-water mixture around the vessel.
   3. Rinse again with ultrapure water.
7. If you notice the growth of bacteria or algae despite these precautionary measures, check the column allowable organic additives for the eluent. For example –
   a. 5% methanol can be used for the A Supp 19 column.
   b. 5% acetonitrile or acetone (no alcohol) for the Metrosep C4/C6 columns.
8. If bacterial growth is observed in standing rinse vials on the rack, methanol can be added.
   a. Again, check the column manual for allowable organic additives.
   b. Acetone should not be used for inline ultrafiltration since it can damage the PMMA material.