

Technical Bulletin #44

To: Coulometrics Support Personnel

From: Applications Department

Date: 12 – 17 - 02

Subject: CM 5200 Erratic Results

Purpose: This document is intended to assist in trouble shooting the CM5200.

Problem: Erratic Blanks and Analytical Results with the CM5200.

Acceptable blanks for performing analyses are:

5 to 10 $\mu\text{g C}$ for 5 readings
18.3 to 36.6 $\mu\text{g CO}_2$ for 5 readings
25.0 to 50.0 $\mu\text{g CO}_3$ for 5 readings

Blanks below the above ranges are routinely achievable provided the user has properly maintained the UIC, Inc., Coulometric instruments and front end units.

Causes and Corrections:

1. Quality of oxygen carrier gas used. UIC, Inc., recommends the using Ultra High Purity Carrier Grade oxygen. The oxygen content must be $\Rightarrow 99.99+$ %.
2. Gas cylinder regulator. Avoid using a regulator with a rubber or elastomer diaphragm. An ultra high purity gas regulator with a stainless steel diaphragm (such as Matheson Gas Products model 3121-540) is recommended for this instrument.
3. Barium chromate insufficiently conditioned. Condition the barium chromate combustion catalyst overnight at 1000°C with a 25 – 50 mL oxygen flow rate and the combustion tube exit vented to atmosphere. After overnight conditioning, lightly coat ball joints with silicone grease then connect all tubing. Reduce the furnace temperature to the combustion temperature normally used.
4. Leak in the CM5200. Make sure all threaded connections are tight. If the ‘O’ ring seals (CM153-024) in the post-scrubbers are suspect – replace.
5. Leak in the gas lines leading to the Coulometer, coulometer fittings (at the rear of the coulometer and on the cell top. The small unions (1/4 x 1/8” – CM191-001) can and do degrade in time. Replace these unions connected to the flow check valve (also, replace the check valve – CM192–003). Replace the male luer connector (CM129-071) connected to the inlet of the coulometer. Replace the male luer connectors (CM129-071) on the inlet and outlet gas lines on the cell top.

6. Ceramic insert may be over filled with swarf (combustion residue). Ceramic inserts should hold about 4 full carousels of swarf. Replace ceramic insert.
7. Sample boats may be hung up on the ceramic insert. Add quartz wool to the inner tube of the combustion tube to raise the insert to the base of the sample valve.
8. Quartz wool plug not inserted in the ceramic insert to the bottom of the first slot. The quartz wool plug assures that the sample is in the hot plateau region of the tube furnace. (See figures 1 and 2, attached.)
9. Loss of sample. If the sample is weighed into the sample boat then encapsulated some sample may be lost during encapsulation. It is best to tare or tare weigh the sample boat. Add the sample to the boat, encapsulate, then weigh the encapsulated sample.
10. Sample encapsulation in the tin boats not properly done. Loss of sample could occur in the carousel to contaminate the carousel base plate and sample valve. Encapsulate the sample in a manner that eliminates sharp edges that can cause hang ups in the carousel, sample valve and on the ceramic insert. Compress the encapsulated sample into as small a size as possible.
11. The percent difference criterion used in 'Run Diagnostics' when 'Coulometer Endpoint' detection mode is selected may be too large for the sample analyte concentration being measured. Due to the automated operation of the CM5200 analyte carryover occurs that affects the following sample. It is strongly recommended to experiment with this criterion to obtain the smallest value that minimizes carryover between samples.
12. Re-fill all post-scrubber components. Caution! Do not tap the post scrubber to compact the components. Always fill using only gravity.
13. Re-pack or replace the vertical combustion tube.

CM5200 Leak Check:

This section describes leak checking the CM5200 after the above causes and solutions have been addressed.

1. Open the gas cylinder valve and set the delivery pressure to the CM5200 at 8 – 10 psig.
2. Set the CM5200 carrier gas flow rate (left flow controller) to 100 mLs/min.
3. Remove the exit line from the rear of the CM5200.
4. Install and tighten the brass plug (CM191-040) on the exit gas bulkhead fitting.
5. Monitor the carrier flow meter ball. It should drop to '0'. (The ball will not drop until the system pressure equals the supply pressure.)
6. Wait several moments after the carrier flow meter ball has dropped to '0': then turn off the gas cylinder valve. Note the pressure on the cylinder pressure gauge. The cylinder pressure should not drop significantly over one-hour. If the pressure does drop check and tighten the regulator fitting to the gas cylinder and, if the regulator is equipped with a regulator shut off valve, make sure the packing nut is tight. Recheck and tighten any fitting that may seem to be slightly loose and could be the source of the leak. Repeat this leak test.

7. If the cylinder pressure gauge does drop, repressurize the system, but this time turn off the regulator shutoff valve first then turn off the cylinder valve. Again monitor the cylinder pressure gauge. If the pressure drops, the gas regulator valve is defective and needs to be replaced or repaired. If it does not drop the leak is down stream of the cylinder regulator valve.

The ceramic insert should be prepared as shown in Figure 1. A quartz wool plug should be inserted into the bottom of the slotted, ceramic tube and positioned so that the plug just reaches the bottom of the lowest slot on the tube.

Placing the quartz wool plug in the bottom of the ceramic insert helps assure that all evolved combustion gases are swept from the combustion tube. It also helps assure that the tin sample boats are in the heated zone of the furnace after dropping from the sample valve.

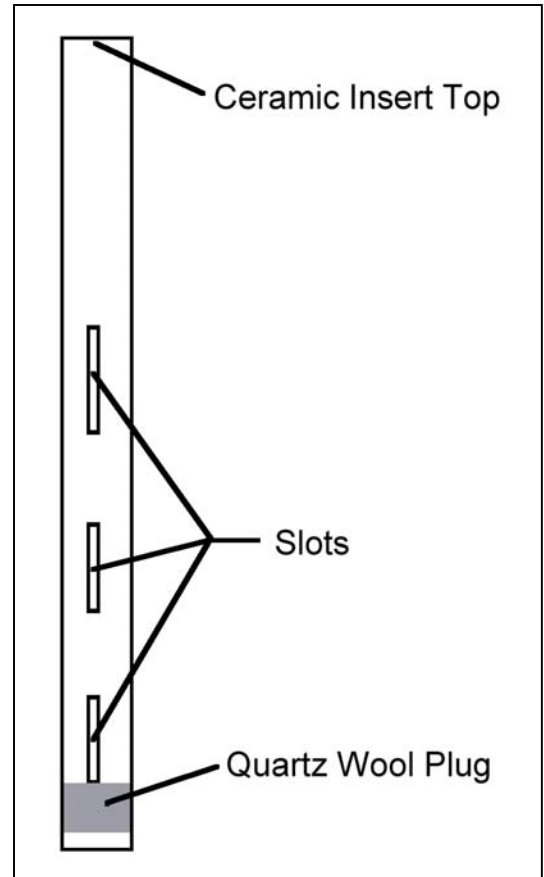


Figure 1

The ceramic insert should be positioned in the combustion tube so that the top of the insert will press firmly against the bottom of the sample valve when the combustion tube is clamped into place. The ceramic insert can be raised or lowered by adding or removing quartz wool to/from the bottom of the combustion tube.

Figure 2 shows how the ceramic insert is positioned slightly above the bottom of the cupped portion of the combustion tube that is mated to the bottom of the sample valve.

The ceramic insert should last for approximately 4 sample carousels worth (90-100) of analyses. Signs that the ceramic insert is full include extended analysis times and or low or high results.

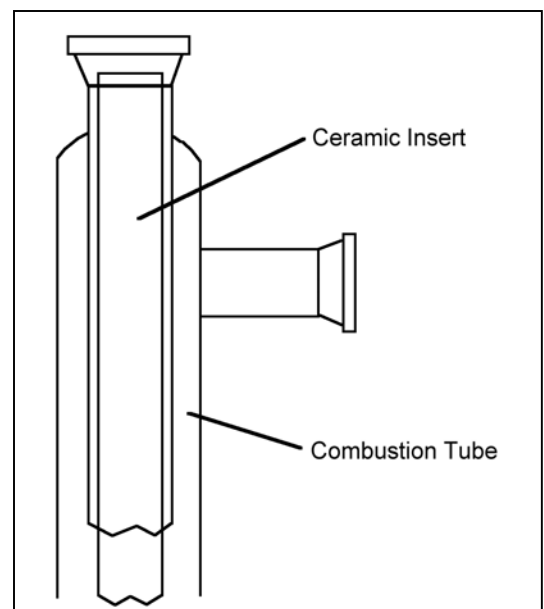


Figure 2