

Technical Bulletin # 43

To: Coulometrics Support Personnel

From: Engineering Dept.

Date: 4-21-03

Subject: CM5240 – Low Analytical Results

Purpose: This bulletin describes causes for and corrections to be made to the CM5240 in the event low analytical results are obtained for samples or standards.

Problem: Low analytical results – samples or standards.

Possible Sources:

1. Degraded, or pin-holed 1/8" x 1/4" silicone adapters. Replace all 1/8" x 1/4" adapters.
2. Blocked or partially blocked acid and/or rinse reservoir bottle filters.
3. Acid dispensing volume.
4. SV-4 – three-way flow diverter solenoid valve.
5. Pre-scrubber – solid Ascarite II plugged by moisture absorbed from the atmosphere.
6. Post-scrubber – frit may be plugged or in process of being plugged.

The following procedure will assist the CM5240 user in determining the reason for low analytical results and the corrective measure(s) necessary to restore analytical precision and accuracy.

Accessing the Interior of the CM5240

Turn off the CM5240 with the switch at the front of the instrument and unplug the power cord from the 120V receptacle.

1. Carefully remove the condenser from the CM5240.
2. Remove the carousel from the CM5240.
3. Disconnect the reservoir bottles from their respective pick-up lines. Remove the acid and rinse water reservoirs from the bottle rack. Leave the bottle rack with the waste bottle receiver in the rack compartment.
4. Remove the alignment/shipping screw from the CM5240 top panel then remove the panel by working the fingertips between the back panel and top.
5. Remove the side panel on the bottle rack side of the instrument in the same manner as described in 4, above.

Reservoir Bottle Filters

1. Remove the filters from the pick-up lines in the acid and water rinse reservoir bottles and note which of the two holes the pickup line is inserted.
2. Back flush each filter with distilled water using vacuum or with a 50 mL syringe.
3. After back flushing, re-install the filters on the pickup lines of the reservoir bottle caps.
4. Alternatively, replace the two (2) filters with two (2) new filters (CM250-017).

Pump Dispensing Volume Check

1. Disconnect the white male electrical connector (top of pump - 4 wires) from the rinse pump (closest to the front of the CM5240). Pull the connector straight up - it is non-locking.
2. Make sure carrier gas delivered to the instrument is set at 20 psig.
3. Carefully re-install the condenser onto the reaction chamber.
4. Plug the power cord into the 120V receptacle and turn on the CM5240 power switch. (The 10-mL acid dispense and 'Load Sample' green LED's will be illuminated and remain steady.)
5. Press the 'Clear/Init.' button one time. (This will discharge acid remaining in the acid pump lines.) At this time, make sure the carrier flow rate is set to 100 mLs' per minute.
6. When the acid pump has stopped (acid LED stops blinking), press the 'Clear/Init.' button again. At this time, make sure the dump flow pressure is set to 250 mLs' or slightly higher. The slider returns to the 'home' position after the cleaning cycle.
7. Inspect the acid reservoir bottle luer lock fitting (CM129-111). If the fitting is bent or cracked replace.
8. Insert the acid pickup line (marked with red tubing and/or has a luer lock connector) into a 600-mL beaker of distilled water and secure the line to the beaker to prevent the line from coming out of the distilled water.
9. Press the 'Clear/Init.' button. The slider valve will move to the reaction chamber, and the acid pump will turn on.
10. When the pump has stopped, press the 'Clear/Init.' button. This initiates a cleaning cycle and discharges the liquid from the reaction chamber.
11. When the slider has returned to the 'home' position (see 9, above) remove the distilled water beaker, waste discharge bottle and bottle rack from the bottle rack drawer.
12. Re-insert the acid pick-up line in the distilled water beaker securing the pick-up line to prevent it from coming out of the distilled water in the beaker and place in the bottle rack drawer.
13. Press the 'Clear/Init.' button to move the slider to the reaction chamber to dispense 10-mLs' of distilled water into the reaction chamber and to clear the line of air bubbles.
14. Press 'Clear/Init.' a second time to initiate a cleaning cycle and return the slider to the 'Load Sample' or ready position.

15. Press 'Clear/Init.' a third time to move the slider valve to the reaction chamber and fill the reaction chamber with liquid.
16. Hold a clean, dry, 250-mL beaker against the chassis directly under the waste chute. Access the waste chute trough the side from which the panel was removed.
17. Press the 'Clear/Init.' a fourth time. (The cleaning cycle is initiated and liquid will be discharged into the beaker.) Wait until the slider has moved to the 'Dump Sample' position before removing the 250-mL beaker.
18. Transfer as much as possible of the 250-mL beaker contents to a 25-mL graduated cylinder. The measured volume dispensed by the acid pump should be equal to or greater than 10 mL.
19. If less than 10-mLs replace the valve cartridges in the acid pump.
20. The water rinse pump is checked similarly to the acid pump.
21. Disconnect the electrical connection on the acid pump (back of the instrument) and re-connect the electrical connection to the front pump.
22. Place rinse water pickup line in the distilled water beaker and secure to prevent the line from coming out of the water.
23. Follow steps 13 - 16, above.
24. Press the 'Clear/Init.' button. Keep the beaker in position until the slider has returned to the 'Load Sample' or 'home' position.
25. Transfer as much as possible of the 250-mL beaker into a 25-mL graduated cylinder. If the standard rinse cycle is used the amount of liquid should be equal to about 10 mLs. If a second rinse cycle is active the total amount of liquid should be about 15 mLs.
26. To evaluate SV-4 skip steps 27 and 28.
27. Turn off the CM5240 and remove the power cord from the receptacle. Carefully remove the condenser from the reaction chamber. Re-assemble the CM5240 by re-connecting the acid pump, distilled water pump, installing the side panel, top panel and shipping/alignment screw (finger tight), and condenser. Install the bottle rack and connect the pick-up lines to their respective reservoir bottles.
28. Plug in the power cord and turn on the instrument.

SV-4

If the acid delivery volume is correct check SV-4. (See attached diagram for location.)

1. Place an empty 1-liter beaker in the bottle rack drawer under the waste chute of the slider valve.
2. Insert the acid pick-up line in the distilled water beaker securing the pick-up line to prevent it from coming out of the distilled water in the beaker and place in the bottle rack drawer.
3. Press the 'Clear/Init.' button to move the slider to the reaction chamber and to dispense 10-mLs' of distilled water into the reaction chamber.
4. Examine the waste discharge line of SV-4 (line on SV-4 toward the slider valve) for liquid movement. If liquid movement in this line is observed SV-4 is malfunctioning and must be replaced.

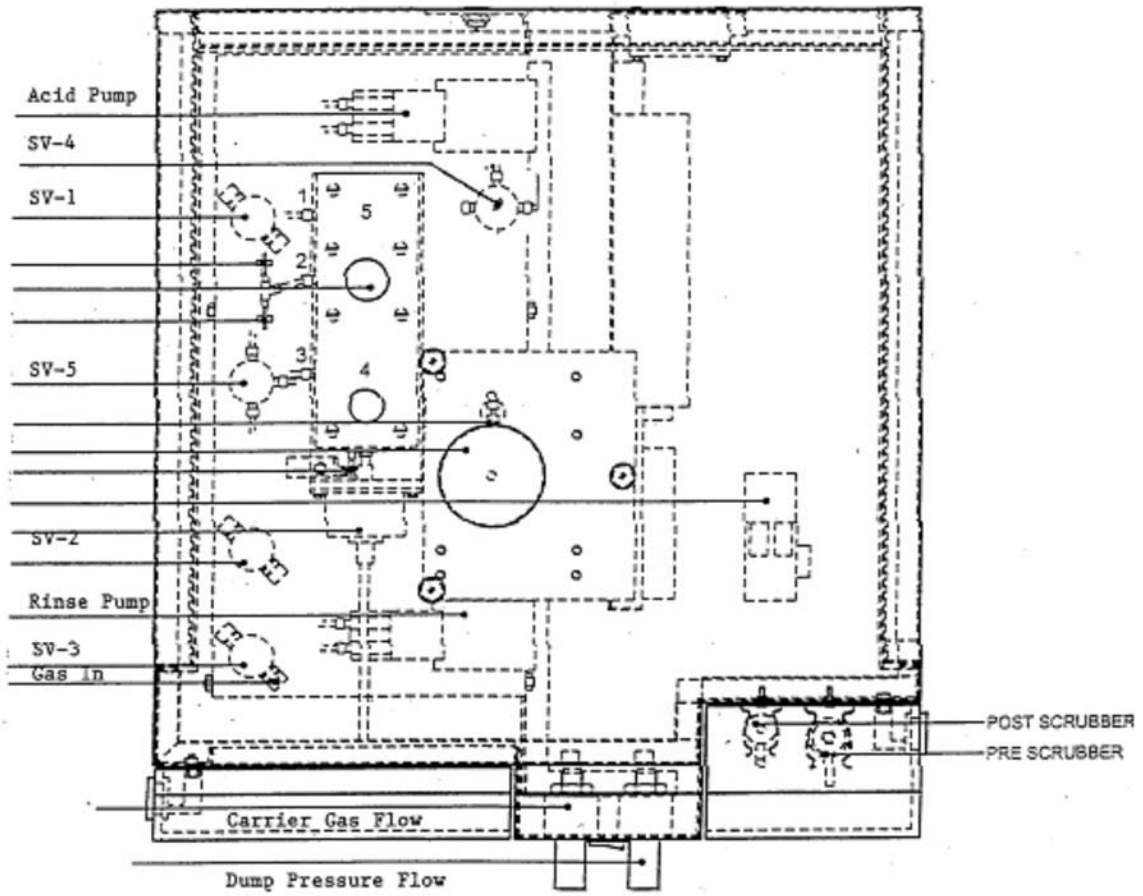
5. Remove the gas line fitting from the connector labeled 'From Condenser'. Place the fitting in a small beaker of water and look for a rapid stream of bubbles. Alternatively, measure the gas flow rate using a soap bubble flow meter and stopwatch. Measured flow should be approximately 100-mLs per minute. If the measured flow does not agree reasonably with the set flow rate SV-4 may be partially open due to particulate preventing the valve from fully closing. The valve channels may also be blocked due to particulate entrainment. Correction: replace SV-4. If flow rate is correct continue testing described below.
6. Re-assemble the CM5240 as described above.

Post-Scrubber

1. Re-insert the male insert on the condenser line into the connector on the rear of the instrument labeled 'From Condenser'.
2. Disconnect the line labeled 'A' (inlet to post-scrubber) from the post-scrubber and measure the gas flow with a soap bubble flow meter and stop watch. Gas flow should be nearly 100-mLs/per minute. Reconnect the 'A' line to the post-scrubber. Bubbles should be observed in the post-scrubber. If not, the post-scrubber frit may be plugged. Replace the post-scrubber body with a clean, unblocked post-scrubber body.
3. Remove the male luer connector from inlet of the Coulometer and insert into a small beaker containing distilled water. A stream of bubbles should be observed. If not, remove the inline check valve and test the line to the check valve for gas flow. Replace the check valve with a new check valve.
4. Reconnect the male luer connector to the Coulometer.

Pre-scrubber

1. Make sure the instrument is in the ready to run position (acid and 'Load Sample' LED on and steady) then turn off the instrument. Make sure 20 psig of carrier gas is supplied to the instrument before proceeding.
2. Disconnect line 'C' (outlet) from the pre-scrubber.
3. Turn on the instrument.
4. If the pre-scrubber is plug free a pressure loss error signal should be indicated (see manual). If no error signal is obtained replace the pre-scrubber.
5. Upon completion of testing re-assemble the CM5240.
6. If this test is not definitive replace the pre-scrubber.



TOP VIEW