

# Coulometer Quick Start Guide

Coulometer: Quick Start Guide (from Coulometer\_QSG\_371T\_draft)

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## Manual Information

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Audience:	Scientists, Laboratory Technicians		
Origination date:	3/1/2009		
Current version:	V371T	July 2017	
Previous Revisions:	V1.2	2/20/2014, V 1.0	9/08/2010

Domain:	Chemistry
System:	Coulometry

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## Introduction

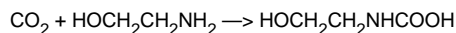
Coulometer analysis determines carbonate concentration in a variety of samples, including pure carbonates, soils, rocks, and liquids. Coulometry quantifies the carbon dioxide evolved from acidified samples and uses this to determine the carbonate content in the original sample. The inorganic carbon value obtained from this method is used in conjunction with TC (total carbon) measurements from the CHNS to arrive at an organic carbon value.

## Theory of Method

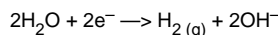
IODP's UIC Coulometrics CM5015 coulometer provides absolute determination of the concentration of carbon dioxide (CO<sub>2</sub>) evolved from an acidification process. The coulometer cell is filled with a proprietary solution containing monoethanolamine and a colorimetric pH indicator. A platinum cathode and silver anode are positioned in the cell, and the assembly is located between a light source and a photodetector. When a gas stream passes through the solution, CO<sub>2</sub> is quantitatively absorbed, reacting with the monoethanolamine to form a titratable acid. This acid causes the color indicator to fade. A spectrophotometer monitors the change in the solution's percent transmittance (%T). As %T increases, the titration current is automatically adjusted to generate a base at a rate proportional to %T. When the solution returns to its original color (original %T), the current stops. The amount of CO<sub>2</sub> evolved is quantitated from the duration and magnitude of the current required to balance the acid by CO<sub>2</sub> evolution. Based on the principle of Faraday's Law of Electrolysis (the quantity of a substance produced by electrolysis is proportional to the quantity of electricity used), each mole of electrons added to the solution is equivalent to 1 mole of CO<sub>2</sub> titrated.

Chemical reactions occurring in the coulometer cell follow:

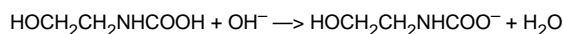
1. Absorption of CO<sub>2</sub> by the cathode solution (cathode reaction):



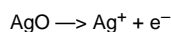
1. Electrochemical generation of OH<sup>-</sup> (cathode reaction):



1. Neutralization of absorbed CO<sub>2</sub> reaction product by electrochemically generated OH<sup>-</sup>:



1. Anode reaction:



## Interferences

A variety of carrier gases can be used for coulometry (O<sub>2</sub>, N<sub>2</sub>, He, and dry air); the JRSO uses N<sub>2</sub> for the carrier gas. Interferences caused by compounds such as SO<sub>2</sub>, SO<sub>3</sub>, H<sub>2</sub>S, HCl, HBr, HI, and Cl<sub>2</sub> are removed with KOH and AgNO<sub>3</sub> scrubbers.

# Apparatus, Reagents, & Materials

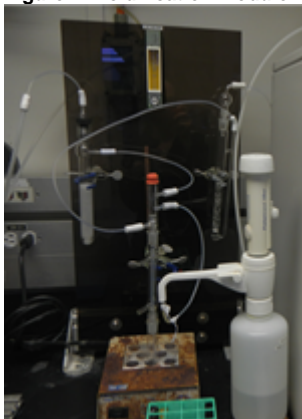
## Hardware

- Coulometer unit (UIC CM5015) with titration cell (*Figure 1*)
- Acidification module (similar to UIC CM5030) (*Figure 2*)
- Dual balance system, motion-compensated, with control software

**Figure 1. Model CM5015 Coulometer.**



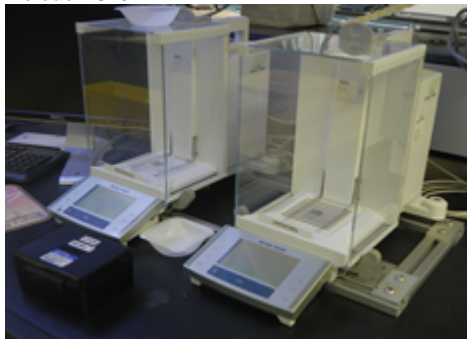
**Figure 2. Acidification Module.**



## Dual Balance System Hardware

A Cahn balance (*Figure 3*) and 2 Mettler Toledo XS204 (*Figure 4*) analytical balances with motion compensation software are used to measure the mass of samples and chemicals. The Cahn balance measures samples for the Coulometer.

**Figure 3. Cahn Electrobalance. Figure 4. Mettler Toledo XS204.**

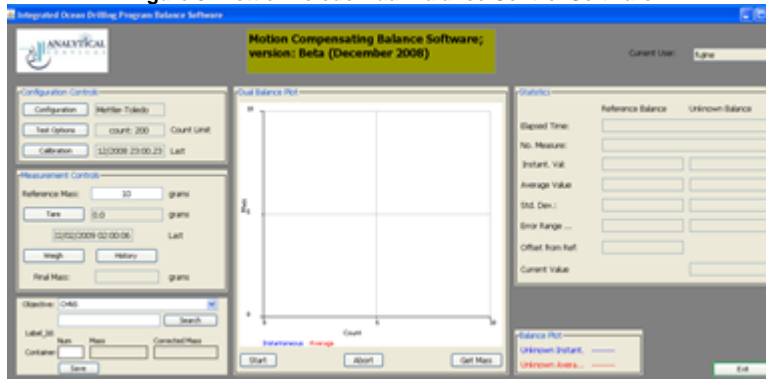


## Software

### Dual Balance System Software

Motion compensation software developed in house allows the user to weigh the mass of chemicals and samples at sea. Reagents and samples >250 mg must be measured on the Mettler-Toledo XS204 balance (*Figure 5*). Reagents and samples < 250 mg must be measured on the Cahn balance (*Figure 6*).

**Figure 5. Mettler-Toledo Dual Balance Control Software.**



**Figure 6. Cahn Balance Control Software.**



## Laboratory Supplies

### Apparatus

- KOH pre-scrubber trap
- AgNO<sub>3</sub> post-scrubber trap
- Reaction flask/reaction vial
- Bottle-top dispenser, 5 mL
- Agate mortar and pestle

### Materials

- Wax paper boats
- Scoop
- Tweezers
- Sample containers

### Reagents

- Potassium hydroxide (KOH)
- Silver nitrate (AgNO<sub>3</sub>)
- Potassium iodide (KI)
- Sulfuric acid (H<sub>2</sub>SO<sub>4</sub>)
- Hydrochloric acid (HCl)
- Anode solution (UIC proprietary)
- Cathode solution (UIC proprietary)

### Gases

- Nitrogen (99.995% or better) is used as carrier gas to minimize the amount of CO<sub>2</sub> the scrubber (KOH) must absorb

### Reagent Solutions

- 45% KOH ([%w/v]): add 90 g KOH pellets to water and make up to 200 mL once fully dissolved.
- **Warning!** This procedure liberates caustic fumes and heat. Perform in a fume hood.)
- 3% AgNO<sub>3</sub> ([%w/v]): dissolve 3 g silver nitrate in water and make up to 100 mL when fully dissolved.)
- 2N H<sub>2</sub>SO<sub>4</sub>: add 55.5 mL concentrated sulfuric acid to water and make up to 1L
- 2N HCl: add 166 mL concentrated hydrochloric acid to water and make up to 1L

## Sample Preparation

**Liquid samples** are pipetted directly into the sample tube. Most samples use 2 mL volume. If samples are suspected to contain high sulfur contents, use 0.5 mL.

**Solid samples** must be dried, ground, and weighed before introduction into the prepared Coulometer apparatus. The workflow for solid sample preparation is as follows:

1. A scientist or staff member logs wet sample information into *SampleMaster* at the sampling table. The sample is given the name *CARB* to ensure proper routing.
2. Freeze-dry the sample (*Freeze-Drying the Sample*).
3. Homogenize (grind) the sample (*Grinding the Sample*).
4. Weigh the sample, assign a container and code, and upload the mass data to LIMS (*Weighing the Sample*).
5. Prepare the coulometer acidification for analysis (*Preparing Acidification Module and Coulometer Cell*).

### Freeze-Drying the Sample

1. Cut the sample bags or roll back the top to ensure an open orifice during the freeze-drying process.
2. Place the sample in the freeze-drier in the Chemistry Lab under vacuum for 12 hr. If sample is finely divided and is clumpy, freeze-drying may take >12 hr. Sample should appear dry and powder easily (in mortar and pestle).
3. Do not overload the freeze dryer.

### Grinding the Sample

1. Remove the freeze-dried sample from the sample bag and place in a mortar. If the sample volume is too large to be ground in the mortar, grind it in separate smaller portions and recombine.
2. Grind the sample with a pestle to a fine, powder-like consistency with no large clumps. If the sample is too hard to grind in a mortar and pestle, use the mixer mill (see the X-ray technician for assistance in operating the mixer mill).
3. Transfer the sample to a new bag or container.

### Weighing the Sample

1. Log into the Dual Balance system for the **Cahn Balance**. Answer **Yes** or **OK** on all prompts that appear during the log-in process. The user's log-in ID must be same as the LIMS database ID.
2. Click **Test Option**, and enter a number (usually >100 based on sea state; see the technician for guidance). Click **Save/Exit** to return to the main window.
3. Fold a small piece of wax paper (~0.5 cm x 0.5 cm) on opposite edges to create a U-shaped wax paper sample boat. Place the wax paper boat on the left weighing pan. Place a similar size of paper on the tare pan (right). Close the door, click **Tare**, and then **Start** on the plot screen. The current mass shown in the software between the left and tare (right) weighing pan should be no more than 1.0 mg.
4. Once the measurement is finished and the value is acceptable, click **Get Mass**. The tare value will be changed and the display will clear.
5. Put the sample on the weighing pan (~7–13 mg) using the scoop.
6. Press **Weigh** on the screen and then **Start** on the plot panel. The Weigh measurement will not begin if you do not press **Start**.
7. Once the measurement is done and the value is acceptable, click **Get Mass**. Final mass value (under the weigh button) will be changed and the display will clear.
8. Select **Objective** from the list and enter a part of the text ID or label ID of the sample, then click **Search**.
9. Select a sample from the list, then click **Assign** to return to the main window.
10. Enter a container number, and click **Save** to save the mass value into the LIMS. Write down on a piece of paper the mass, container number, text\_id, and core information (ie. expedition, site, hole, section, and interval if applicable).

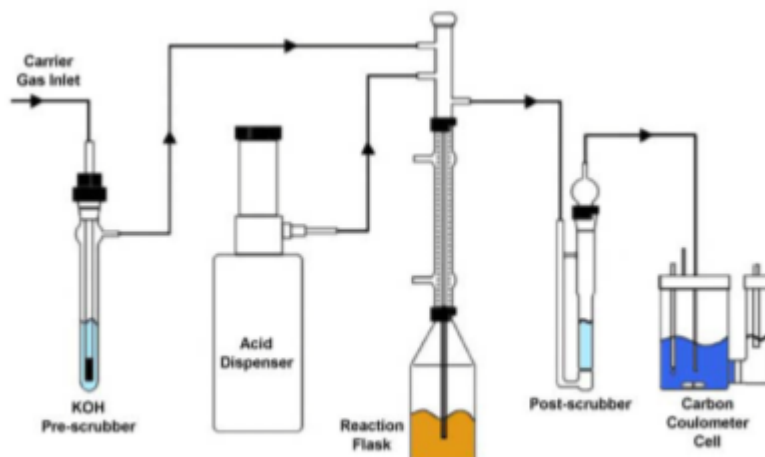
### Preparing Acidification Module and Coulometer Cell

1. Add approximately 1/8- 1/4" layer of granular KI to the bottom of the side arm (small side, anode compartment) of the cell. The junction between the two compartments should be about halfway covered with KI. (*Figure 7*).
2. Add a stir bar to large section (large side, cathode compartment) of the Carbon Coulometer Cell.
3. Fill the large section of the Carbon Coulometer Cell with cathode solution to the 100mL line.
4. Fill the small section of the Carbon Coulometer Cell with anode solution to just under the level of the cathode solution (about 20mL). Do this quickly (within 1 min) after filling the cathode cell, or else the cathode solution will start filtering through the junction between the cells and contaminate the anode solution.
5. Press the cathode top on the cathode compartment and the anode top on the anode compartment. In the anode compartment, make sure the electrode is in the solution but not in contact with the granular KI.
6. Fill the KOH pre-scrubber trap 1/2 full of 45% KOH solution.
7. Fill the AgNO<sub>3</sub> post-scrubber trap 1/2 full of 3% AgNO<sub>3</sub> solution.
8. Add 3 drops of 2N H<sub>2</sub>SO<sub>4</sub> to the AgNO<sub>3</sub> trap.

9. Attach the input gas tube (carrier gas inlet) to the KOH trap.
10. Turn on the gas flow and set to 100 cm<sup>3</sup>/min.
11. Connect the KOH trap to the reaction flask.
12. Connect the reaction flask to the thinner side of the AgNO<sub>3</sub> trap.
13. Connect the top of the AgNO<sub>3</sub> trap to the Carbon Coulometer Cell (through the line entering the back of the Coulometer)

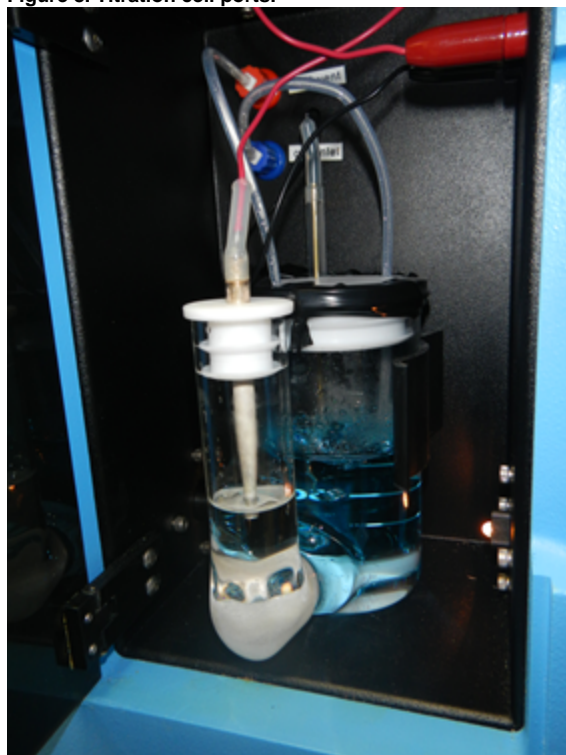
Connect the anode/cathode to the titration cell ports next to the titration cell (*Figure 8*).

**Figure 7. Acidification Module and Carbon Coulometer Cell.**



Level of granular KI

**Figure 8. Titration cell ports.**



## Sample Analysis

Once the sample is placed in the reaction vial, acid is added to release CO<sub>2</sub> gas. This gas is carried through the coulometer cell and into the titration cell, where the sample is titrated by the coulometer automatically and the software plots µg carbon vs. time. The software evaluates the slope of the plot against a drift threshold and then compares the slope against **\$Threshold\_slope** (method-determined value equivalent to 29% transmittance) to determine when the titration is complete. When the threshold is reached, titration halts and the final result is expressed in µg C, from which weight percent (wt%) CaCO<sub>3</sub> can be calculated.

## Running Samples

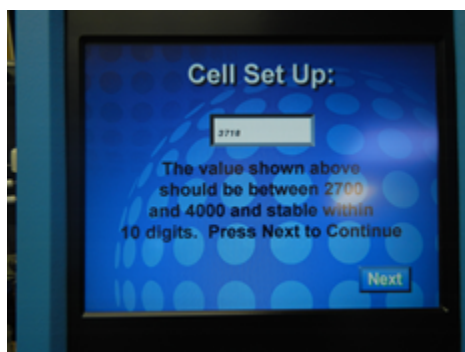
1. Turn on the **heating unit** and **power** to the main coulometer unit.
2. Choose **Emulation Mode** on the screen.



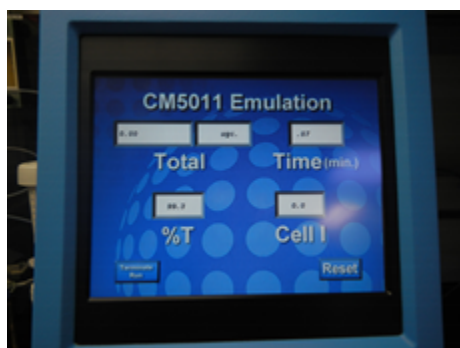
1. Click **Run Cell Setup** on the screen.



1. On the transmittance screen that appears, check to see that the value is **between 2,700 and 4,000**. If not, swivel the carbon coulometer cell until a value in this range is acquired. Do not move the cell once this position has been found.



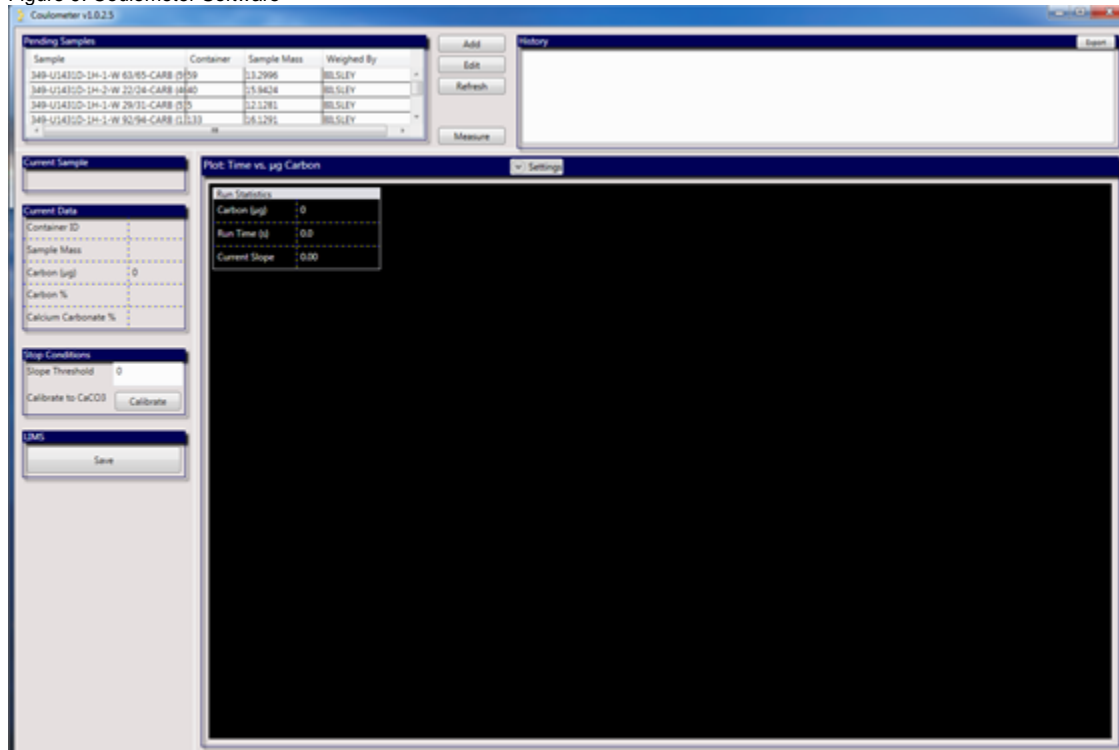
1. Click **Next**.
2. Click **Start Analysis**. The Cell Activity screen will appear. The %T should be between 99.8-100.1 and the Cell I should be 0.0.



1. Switch the cell to **On** on the main coulometer unit.
2. Allow the cell to **equilibrate for 30-45 minutes** before continuing.
3. Open the Coulometer software and log in.

- Heat the vial with the weighed standard (100% CaCO<sub>3</sub>) in the heating unit for 2-3 minutes before measuring.
- Attach the top of the reaction cell to the sample vial. Bubbles should appear in the pre and post scrubber traps. If not, make sure all connections are tight.
- Select the **sample** from the list you wish to measure on the Coulometer software (*Figure 9*).

Figure 9. Coulometer Software



- Then slowly add 5 mL of 2N HCl using the connected repeater dispensette.
- Quickly press **Measure** in the sample list page of the coulometer software. If the measurement is delayed the results may underestimate the calcium carbonate percentage. A measurement screen will appear displaying real time data acquisition, the options to abort or stop the measurement, and to save/not save the results. The slope threshold is a measurement of the µg carbon with respect to time, and may be adjusted to specify the stopping point of the titration. Setting the slope threshold too low increases measurement times with the possibility of including circuit noise in the results, whereas setting the threshold too high will cause the measurement to prematurely terminate. The default slope threshold is 0.1.
- Once measuring is complete, **click Save** and record the %CaCO<sub>3</sub>, % carbon, and carbon mass in the log book. The %CaCO<sub>3</sub> standard should give a %CaCO<sub>3</sub> value of 100%. See the technician if this value is not acquired after running 3 standards.
- Wait 10-15 minutes for the system to restabilize between samples.
- Repeat steps 10-16 for the samples.

## Shutting Down the Coulometer

Shut down the instrument after each run.

- Turn off cell power, unit power, and heater power.
- Unplug the electrodes and remove the titration cell.
- Place the appropriate jumper between the red and black cell output fittings.
- Remove all traps and dispose of solutions appropriately.
- Rinse/dry all glassware.

## Cleaning the Glassware

- Sample tubes:** rinse sample tubes with DI water and place into the oven to dry. They do not need to be acid washed.
- Carbon Coulometer Cell:** Clean the cathode/anode cell in a fume hood by adding acetone to the anode cell. The acetone will leach through the bridge between the cells and clean it. Follow the acetone rinse by placing DI water in the anode cell and letting that leach through.
- Platinum electrodes:** Electrodes can acquire surface coatings from the solutions. Remove this coating by placing the electrode in a concentrated nitric acid solution for 1 hr.
- AgNO<sub>3</sub> post-scrubber trap:** Clean the post-scrubber trap by adding ammonium hydroxide to the trap and letting it sit in the fume hood for about 24 hours.



## Data Handling

Weight percent calcium carbonate is calculated from  $\mu\text{g}$  carbon measured during the titration as follows:

$$\% \text{CaCO}_3 = \mu\text{g C} \times 8.333 / \text{sample mass}$$

Sample mass is stored in LIMS associated with the container ID that the coulometer analysis is associated with.

## Quality Assurance/Quality Control

QA/QC for Coulometer analysis consists of instrument calibration and continuing calibration verification using check standards, along with blanks and replicate samples.

### Range and Rate

The working range of the  $\text{CO}_2$  coulometer is 0.01  $\mu\text{g}$  to 100mg C. . The coulometer cell solution can absorb >100 mg of C. Titrating at maximum current (200 mA), the coulometer can titrate 1500  $\mu\text{g}$  of carbon (or 5500  $\mu\text{g}$   $\text{CO}_2$ ) per min.

### Analytical Batch

An analytical batch is a method-defined number of samples with which QC samples including calibration verification, blank check, and replicate samples are run. Because samples are grouped into QC batches, if problems arise, affected samples can be identified and reanalyzed. Analytical batches for the coulometer are typically 10 samples.

### Control Limits

Each QA/QC sample has one the following results:

- In Control
- In Control (exceeds warning limit)
- Out of Control (exceeds control limit)

For a system to be considered in control, all QA/QC samples (blanks, calibration verification [CV] standards, and replicate samples) must be in control.

### In Control

A QA/QC sample is in control when the sample analysis result is within a certain tolerance of acceptable limits (usually  $1\sigma$ ). Calibration verification standards should be within acceptable limits of the actual value of carbonate, blanks should be within acceptable limits of background levels of carbonate, and replicate samples should be within acceptable limits of precision. When the system is in control, as indicated by acceptable results on QA/QC samples, analytical results for unknown samples are considered to be reliable.

### In Control (Warning Limit Exceeded)

When QA/QC samples exceed the warning limits (generally  $2\sigma$  but  $\sigma$  to  $3\sigma$ ), the system is considered to be in danger of becoming out of control (but is not yet out of control). Typically, the warning situation indicates that the operator must decide whether to take action. The operator can continue the analysis if he or she does not think that the control limit will be exceeded.

### Out of Control

If the control limits are exceeded (generally  $3\sigma$ ), the instrument system is considered out of control and all samples in the current analytical batch are invalid and should be reanalyzed once corrective action has been taken to put the system back in control.

### Blanks

A blank is run every  $N$  (defined by method) samples. The blank result is evaluated against  $\$CL$ , the method-defined percent threshold that the measured blank value can deviate from standard value and still be considered in control, and  $\$WL$ , the method-defined percent threshold that the measured blank value can deviate from the standard value before setting a warning flag.

- If the blank result is  $<\$WL$  and  $<\$CL$ , the system is in control and analysis can continue.
- If the blank result is  $>\$WL$  and  $<\$CL$ , the system is flagged with warning limits, although analyses can proceed.
- If the blank result is  $>\$CL$ , the system is out of control and samples in the analytical batch (between the previous blank and the current blank) are invalid and must be rerun.

### Calibration

The Coulometer instrument electronics are calibrated by the manufacturer. Each time the reagents are changed a calibration curve is constructed by running the following standards:

- Blank: 0% CaCO<sub>3</sub>
- STD 1: standard level to bracket the lower end of expected sample value range
- STD 2: standard level to bracket upper end of expected sample value range
- CaCO<sub>3</sub>: 100% CaCO<sub>3</sub>

The calibration curve is calculated using linear fit, least-squares method as measured CaCO<sub>3</sub> vs. STD CaCO<sub>3</sub>:

Variable	Calculation
y = STD_CaCO3	$(mass\_C\_std / mass\_std) \times (100.087/12) \times 100\% = 834\% \times mass\_C\_std / mass\_std$
m = slope	$(STD\_CaCO3 / Sample\_CaCO3)$
b = intercept	STD_CaCO3
x = meas_CaCO3	$(mass\_C\_sample / mass\_sample) \times (100.087/12) \times 100\% = 834\% \times mass\_C\_sample / mass\_sample$
y = mx + b	$(834\% \times mass\_C\_std / mass\_std) = m \times (834\% \times mass\_C\_sample / mass\_sample) + b$

A transfer function relates measured µg carbon from the instrument to normalized %CaCO<sub>3</sub>. This transfer function is applied to all measurements in the range for which the calibration is valid.

## Calibration Verification

A check standard is run every 6 hr of Coulometer instrument operation or every 10 samples (whichever comes first). Check standards consist of a 100% CaCO<sub>3</sub> standard (reagent grade calcium carbonate).

The check standard result is evaluated against the threshold for %variance limits for calibration verification standard (\$X) against true value as follows:

$$(834\% \times mass\_C\_normal / mass\_normal) = m \times (834\% \times mass\_C\_check / mass\_check) + b$$

$$(834\% \times mass\_C\_normal / mass\_normal) = normalized\%CaCO3\_$$

- If the check standard \$X >1%, then rerun the standard.
- If the check standard \$X >1% on the rerun, then change the reagent solution, recalibrate the instrument, and rerun all samples in the corresponding analytical batch.
- If the check standard rerun falls within actual value ±1%, then run the check standard again to determine one of the following:
- If the verification check standard run falls within actual value ±1% then the check standard is considered successful and analysis can continue.
- If the verification check standard \$X >1%, then change the reagent solutions, recalibrate the instrument, and rerun all samples in the corresponding analytical batch.

## Precision

Every N (defined by method) samples, a single sample is analyzed in replicate. The deviation between the two sample results is evaluated against \$CL, the method-defined maximum percent deviation allowable for the precision to be considered in control, and \$WL, the method-defined percent deviation allowable for the precision before setting a warning flag.

- If precision is <\$WL and <\$CL, the system is in control and analysis can continue.
- If precision is >\$WL and <\$CL, the system is flagged with warning limits, although analyses can proceed.
- If precision is >\$CL, the system is out of control and samples in the analytical batch are invalid and must be rerun.

## Accuracy

Typical accuracy using the UIC Coulometer is as follows:

- Carbonate carbon in calcium carbonate: 12.00%/12.00% ± 0.05%
- Titration accuracy is ±0.15% in samples with >1000 µg C.
- If sample volume limits CO<sub>2</sub> evolution to small amounts, accuracy is better than ~1 µg C.

## LIMS Integration

### Sample Characteristics

- Analysis is typically performed on a homogenized powdered subsample
- Sample type can be homogenized powder or aqueous
- Analysis is destructive

### Analysis Characteristics

## Weight Analysis

Data have the following dependencies on weight analysis:

- Mass of carbonate sample (measured)
- Container ID (directly input)

## Coulometer Analysis

The following analysis components are uploaded from the coulometer into the LIMS with each sample result:

- Sample ID
- Instrument serial number
- Analysis timestamp
- µg carbon measured (measured)
- Slope threshold
- Analysis duration
- Method reference
- Calibration information
- Slope ( $m$ )
- Intercept ( $b$ )
- $R^2$
- Timestamp

## LIMS Analysis Components

Analysis	Component	Definition	Unit
COUL	calcium_carbonate_percent	Concentration of CaCO <sub>3</sub> in sample	wt%
	carbon_mass	Mass of carbon in sample	µg
	carbon_percent	Concentration of carbon in sample	wt%
	container_number		
	mass	Mass of sample	mg
COUL_QAQC	calcium_carbonate_expected_percent	Concentration of CaCO <sub>3</sub> expected in standard	wt%
	calcium_carbonate_percent	Concentration of CaCO <sub>3</sub> in sample	wt%
	carbon_expected_mass	Mass of carbon expected in a standard	µg
	carbon_expected_percent	Concentration of carbon expected in standard	wt%
	carbon_mass	Mass of carbon found in standard	µg
	carbon_percent	Percent carbon found in standard	wt%
	container_number		
	corr2	Correlation coefficient R <sup>2</sup>	
	intercept		
	mass	Mass of sample	mg
	slope		
	standard_percent	Percent of carbon expected in standard as determined from standard	wt#

## Health, Safety, & Environment

### Safety

#### Carbon Cathode Solution (CM300-001)

–Hazardous components: Dimethyl sulfoxide, Monoethanolamine, Tetraethylammonium bromide (TEAB)

–Hazards:

- Inhalation: irritant; TEAB toxic
- Absorption: irritant; TEAB toxic/potential mutagen
- Ingestion: TEAB toxic

–Handling: absorbs CO<sub>2</sub>; keep tightly closed.

–Storage: keep away from oxidizers, heat, and ignition sources

–PPE: gloves, safety glasses

–Reactivity: stable; incompatible with oxidizers, acids, alkali metals, CO<sub>2</sub>

### **Carbon Anode Solution (CM300-0002)**

–Hazardous components: Dimethyl sulfoxide, potassium iodide

–Hazards:

- Inhalation: irritant
- Absorption: irritant

–Storage: keep away from heat/ignition sources and oxidizing agents

–PPE: gloves, safety glasses

–Reactivity: stable; incompatible with oxidizers, acids, alkali metals, CO

### **Potassium Iodide (CM300-003)**

–Hazards:

- Inhalation: irritant
- Absorption: irritant
- Ingestion: irritant

–Incompatible materials: alkaloid salts, chloral hydrate, potassium chlorate, metallic salts, tartaric and other acids, bromine trifluoride, fluorine perchlorate

## **Waste Management**

Waste may be washed down drain with flowing water.

## **Maintenance/Troubleshooting and Parts/Consumables**

For maintenance and troubleshooting or parts and consumables, see the Coulometer User Guide.