
Coulometer Quick Start Guide

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Introduction

Coulometer results are used to determine carbonate content in samples and in combination with total carbon measurements can be used to calculate organic carbon.

Apparatus, Reagents, and Materials

Hardware

- Coulometer titration cell
- Acidification module

Apparatus

- KOH prescrubber trap
- AgNO₃ postscrubber trap
- Reaction flask/vial
- Bottle-top dispenser (5 mL)
- Agate mortar and pestle

Materials

- Wax paper boats
- Scoop
- Tweezers
- Sample containers

Reagent Solutions

- 45% KOH [%w/v]: dissolve 90 g KOH in water and make up to 200 mL.
- 3% AgNO₃ [%w/v]: dissolve 3 g silver nitrate in water and make up to 100 mL.
- 2 N H₂SO₄: add 55.5 mL concentrated sulfuric acid to water and make up to 1 L.
- 2 N HCl: add 166 mL concentrated hydrochloric acid to water and make up to 1 L.

Sample Preparation

- Liquids:** pipet directly into the sample tube (generally 2 mL). If samples are suspected to contain high sulfur contents, use 0.5 mL.
- Solids:** freeze-dry, grind, and weigh.

Sample Analysis

Preparing Acidification Module and Coulometer Cell

1.	Add ~5 mm KI and 4 cm of anode solution to the small section of the cell.
2.	Add 4 cm cathode solution to the large section of the cell.
3.	Fill the KOH prescrubber trap ½ full of 45% KOH solution.
4.	Fill the AgNO ₃ postscrubber trap ½ full of 3% AgNO ₃ solution and add 3 drops of 2 N sulfuric.
5.	Attach the input gas tube to the KOH trap and turn on gas flow to 100 cm ³ /min.
6.	Connect the KOH trap to the reaction flask and the reaction flask to the horizontal fitting on the AgNO ₃ trap.
7.	Connect the top of the AgNO ₃ trap to the coulometer cell.
8.	Connect the anode/cathode to the titration cell ports.

Running Samples

1.	Turn on the heating unit and the coulometer main unit.
2.	Adjust the transmittance on the titration cell to 100% and turn on the power to the cell.
3.	Calibrate or verify calibration.
4.	Click Get Sample , tip the sample container onto the coulometer, making a good seal, and click Measure .
5.	Slowly add ~5 mL of 2 N HCl to the sample and click Start/Begin Analysis .
6.	Click OK after the measurement is complete.

Shutting down the Coulometer

1.	Turn off power to cell, unit, and heater.
2.	Unplug the electrodes, remove the titration cell, and place the jumper between red and black output fittings.
3.	Remove all traps and dispose of solutions.
4.	Clean and dry all glassware.