DETERMINATION OF CARBON DIOXIDE IN AMINE GAS SCRUBBING SOLUTIONS

Figure 1: CM140 Total Inorganic Carbon (TIC) Analyzer

PRINCIPLES OF OPERATION AND TYPICAL USE

This procedure is typically utilized for the analysis of amine solutions which are used to remove environmentally-controlled emissions from flue gases. This method measures the amount of carbon dioxide (CO_2) in the scrubbing solution. This result is used along with other analyses to determine the amine scrubbing solution's efficiency and remaining capacity.

The system is composed of an "Acidification Module" and a "CO_2 Analyzer". The Acidification Module is purged of atmospheric CO_2 with an inert carrier gas. After purging the system, an aliquot of the amine solution is injected into the sample flask. Acid is then added using the dispenser on the Acidification Module. The gases evolved from the acidification of the sample then pass through a solution containing silver nitrate, where potentially interfering gases are removed from the carrier gas stream. Finally, the carrier gas is introduced into the CO_2 Analyzer where the carbon dioxide is detected and measured quantitatively.

APPARATUS

1. CM140 Total Inorganic Carbon Analyzer
   Includes: CM5014 CO_2 Analyzer or equivalent
   CM5130 Acidification Module or equivalent
2. CM210-017 Auxiliary Scrubber Assembly with 100 ml Flask, Threaded
3. Ring stand with standard type clamp for holding glassware

REAGENTS

1. Pre-Scrubbing Solution: 40 to 45% Potassium Hydroxide Solution (KOH) (Note: The pre-scrubbing solution is needed only if the "internal" carrier gas of the Acidification Module or a low purity external carrier gas is being used.)
2. Acid: 2 to 4N Sulfuric Acid (H_2SO_4)
3. Post-Scrubbing Solution: 20% Silver Nitrate ($AgNO_3$) (Note: This solution can be made using reagent water containing 3% Hydrogen Peroxide (H_2O_2) and acidified with Nitric Acid (HNO_3) to pH 2.)
4. CM300-001 Carbon Cathode Solution
5. CM300-002 Carbon Anode Solution
6. CM300-003 Potassium Iodide (KI)
7. External Carrier Gas (optional) (Note: An external carrier gas, such as nitrogen, helium, or CO_2-free air may be used in place of the "internal" air source of the Acidification Module if desired.)

PROCEDURE

ASSEMBLY

Assemble the Acidification Module and the CO_2 Analyzer as instructed in their respective manuals. Place the auxiliary scrubber in-line between the sample column assembly and the post-scrubber assembly of the Acidification Module. Hold the auxiliary scrubber in place using a ring stand and clamp. *(The auxiliary scrubber consists of two parts. The bottom is a 100 ml sample flask like the ones used on the Acidification Module. The top is an adapter that allows the gas that is bubbled through the solution to exit. See FIGURE 1 for the assembly of this scrubber.)*

The post-scrubber assembly is snapped into place on the right front of the Acidification Module. This fritted scrubber is used as an indicator of when the auxiliary scrubber solution is spent. When the silver nitrate solution reacts with hydrogen sulfide (H_2S) generated from the sample acidification, a black precipitate forms. When this precipitate begins to form in the post-scrubber assembly at a substantial rate, the silver nitrate solution in the auxiliary scrubber should be replaced.

Place a stir bar in a 100 ml sample flask and attach the flask to the bottom of the condenser on the Acidification Module. Secure the flask with the red locking ring. Place the flask and assembly into the heating and stirring port of the Acidification Module. Heating is not normally required so the condenser does not need to be connected to a cooling source. If an external carrier gas is used, adjust the pressure of the system to 2-5 psi. Set the flow to 100 ml/min. using the flow meter on the front of the Acidification Module. *(See FIGURE 2 for an example of the set-up and flow diagram.)*

ANALYSIS

Determine the background rate of the system by entering “blank” as the sample name in the Sample Entry screen of the CM5014 CO_2 Coulometer. Press “Enter” to begin the analysis. The CM5014 will

automatically determine and store the blank value according to the user selectable settings saved within the system’s memory. The instrument will use the saved blank value in calculating the final result values.

To perform an analysis, draw the sample into a syringe that is fitted with an injection needle. *(See note on "sample integrity".)* Usually 250 to 500 µl of sample is used depending on the sample's CO_2 content. Start the CO_2 Analyzer and inject the sample into the sample flask through the septum at the top of the sample column adapter. The analysis endpoint will be automatically determined by the CM5014 according to the user selectable settings saved within the instrument. Individual coulometer readings and