



## 6.0 Reagents

- 6.1 Methyl Orange: Dissolve 0.125 g of methyl orange in 1 liter of distilled water.
- 6.2 pH 3.1 Buffer: Dissolve 5.1047 g of potassium acid phthalate in distilled water and add 87.6 mL 0.1 N HCl and dilute to 1 liter. Stable for one week.
- 6.3 Methyl Orange-Buffered Indicator: Add 1 liter of pH 3.1 buffer (6.2) to 200 mL methyl orange solution (6.1) and mix well. Stable for 24 hours.
- 6.4 Stock Solution: Dissolve 1.060 g of anhydrous sodium carbonate (oven-dried at 250°C for 4 hours) in distilled water and dilute to 1000 mL. 1.0 mL = 1.00 mg CaCO<sub>3</sub>.
- 6.4.1 Prepare a series of standards by diluting suitable volumes of stock solution to 100.0 mL with distilled water. The following dilutions are suggested:

mL of Stock Solution	Conc., mg/L as CaCO <sub>3</sub>
1.0	10
2.0	20
4.0	40
6.0	60
8.0	80
10.0	100
18.0	180
20.0	200

## 7.0 Procedure

- 7.1 No advance sample preparation is required. Set up manifold as shown in Figure 1.
- 7.2 Allow both colorimeter and recorder to warm up for 30 minutes. Run a baseline with all reagents, feeding distilled water through the sample line. Adjust dark current and operative opening on colorimeter to obtain stable baseline.
- 7.3 Place distilled water wash tubes in alternate openings on sampler and set sample timing at 2.0 minutes.
- 7.4 Place working standards in sampler in order of decreasing concentration. Complete filling of sampler tray with unknown samples.
- 7.5 Switch sample line from distilled water to sampler and begin analysis.

## 8.0 Calculation

- 8.1 Prepare standard curve by plotting peak heights of processed standards against known concentrations. Compute concentration of samples by comparing sample peak heights with standard curve.

## 9.0 Precision and Accuracy

- 9.1 In a single laboratory (EMSL), using surface water samples at concentrations of 15, 57, 154, and 193 mg/L as CaCO<sub>3</sub> the standard deviation was ± 0.5.

9.2 In a single laboratory (EMSL), using surface water samples at concentrations of 31 and 149 mg/L as CaCO<sub>3</sub> recoveries were 100% and 99%, respectively.

### **Bibliography**

1. Technicon Auto Analyzer Methodology, Bulletin 1261, Technicon Controls, Inc., Chauncey, N.Y. (1961).
2. Standard Methods for the Examination of Water and Wastewater, 14th Edition, p 278, Method 403 (1975).

