

CHAPTER III EXPERIMENTAL

3.1 Materials

Barium Chloride Certified A.C.S. (Crystal) and Sodium Sulfate Certified A.C.S (Anhydrous) (10-60 Mesh), supplied from Fischer Scientific, were used to prepare 1 mM of stock solutions. Deionized water (MILLI-Q Water System) was used as a solvent for all sample preparations. Barium chloride and sodium sulfate solutions were used as investigating materials. Diethylenetriamine-pentaacetic acid (DTPA, 98+%) solution with pH of 12 was used as a dissolving agent for barium sulfate. The pH of the solution was adjusted by adding potassium hydroxide (KOH), 99.98% (trace metal basis), to obtain the desired concentration. Barium standard solutions were prepared at different concentrations in the range of 0.01 to 1.5 ppm for concentration measurement which will be discussed later on. 1,000 ppm Barium ICP-MS Standard (Cat. No. MSBA1KN-100) and 1,000 ppm Sodium ICP-MS Standard (Cat. No. MSNA1KN-100) are used as reagents.

3.2 Methodology

3.2.1 Experimental Apparatus

HPLC pumps (Waters 510 HPLC pump) were used to pump the fluids through the capillary. The sodium sulfate line flows through a low volume tee (Upchurch Scientific, U-428) which allows for measurement of the pressure at inlet (differential pressure transducer, Honeywell, Z Low, 30 psi range, ± 0.25 psi was used). The pressure line was connected to a pressure gauge and a 25 psi back pressure regulator. After the mixing section, there is a 5 cm long section of 0.02 inch inner diameter capillary to connect another mixing tee (Upchurch Scientific, U-428) to allow for mixing of fluids. The fluid then flows through a 30 cm stainless steel test section (Upchurch Scientific, U-104) and into a collection container. The pressure line at outlet was exposed to the atmosphere at the same level in

all experiments. A diagram of the experimental apparatus used in this study is shown in Figure 3.1.

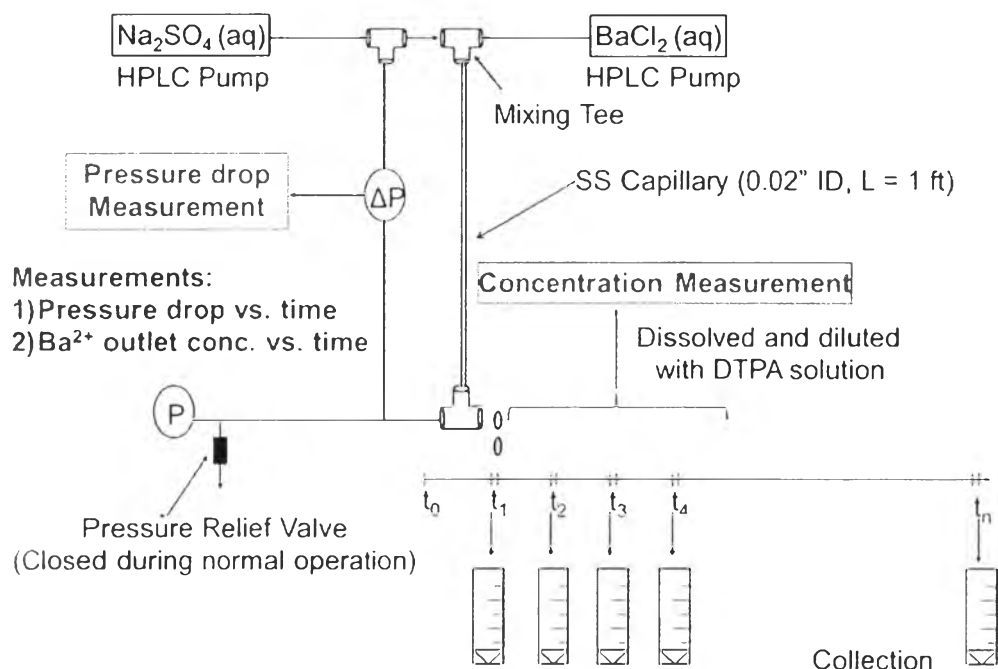


Figure 3.1 Experimental apparatus used in this study.

The system was designed to prevent the pressure transducer damage from pressure buildup during experimental performing by switching the fluid flow to a glass container when the elevated pressure in the system exceeds the set pressure.

When starting a deposition experiment, the system was run with deionized water to remove air in the capillary test section. To make sure pressure to be measured is correct, small pocket of air in all lines were removed through pressure transducer. Once the system was started, the lines were switched from deionized water to brine solutions; the pressure drop measurement and counting the time were started. The time when the barium chloride and sodium sulfate enter the system is defined as $t = 0$. The effluents were collected at different time.

3.2.2 Dilution Scheme

After the fluid passed through the capillary for each period of time, approximately 1 mL of sample was collected at capillary outlet using a 50 mL centrifuge tube which was weighed before. The sample was weighed and immediately filled with DTPA solution to dissolve barium sulfate particles in the solution. Approximately 3 mL of the solution was then transferred to a 15 mL centrifuge tube, the sample was weighed and diluted again with DTPA solution. The collections were kept stirring overnight to guarantee that all the barium sulfate particles have been redissolved.

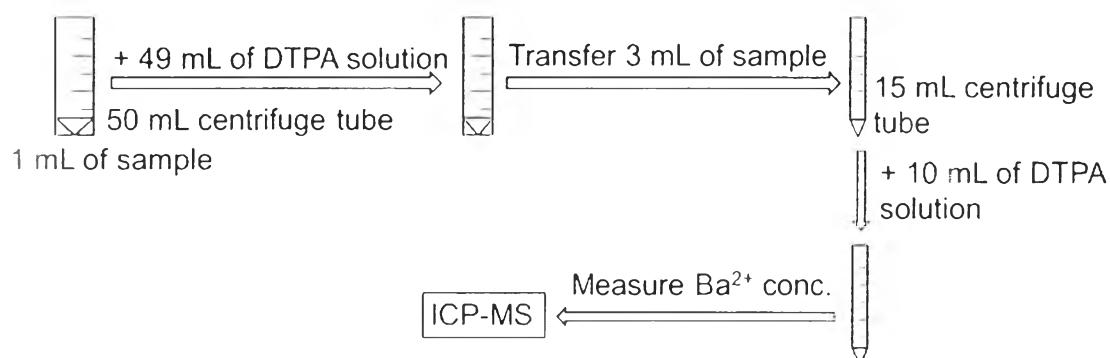


Figure 3.2 Schematic diagram of the dilution procedure.

3.2.3 Pressure Drop Analysis

All of the pressure drop profiles are shifted based on the initial pressure drop at steady state where no deposition occurs. New capillaries were used for each new run.

3.2.4 Concentration Measurement

In order to measure barium ion concentration, the samples were analyzed using Inductively-Coupled Plasma Mass Spectroscopy (ICP-MS). The equipment was calibrated before measuring the concentrations using barium standard solutions and DTPA solution was used as blank solution. The acceptable relative error for calibration curve in this work is typically at least 0.999. The calibration was repeated in every set of experiment.