CHAPTER III
EXPERIMENTAL SECTION

3.1 Materials

Cationic polyelectrolyte used in this research was poly (diallyldimethyl ammonium chloride) tradename MERQUAT 100 obtained from Calgon Corporation as a 40 % solution (QUAT). Average molecular weight of QUAT was approximately 240 K and the empirical formula monomer was $(H_2C{=}CHCH_2)_2N(CH_3)_2Cl$. Sodium chromate was analytical grade supplied by Seelze - Hannover, Germany and analytical grade barium chloride dihydrate obtained from Merck company were used as received.

Purification of polyelectrolyte to remove low molecular weight components was done in 400 mL stirred cell (Spectrum company) with 10K MWCO cellulose ester membranes (Spectrum company). The cell was operated at 60 psi pressure applied from a nitrogen tank and the stirring speed was 250 rpm. The initial QUAT concentration was 0.0742 M (monomer) and 85 % of the initial volume was removed as permeate. Then, distilled water was added to the retentate to the original solution volume, this solution then being treated. The purification process was repeated for at least 6 cycles.

3.2 Methods

3.2.1 Determination of Equilibration Time

In order to determine minimum contact times to attain equilibrium for the protocol used, purified QUAT and chromate ions were mixed as shown in Table 3.1 and then shaken constantly for 5 min. Barium chloride was added to the solutions and shaken again for 7 min. After the solutions were cooled at 5 °C for 24 hrs., the crystalline barium chromate was forced to settle by centrifugation at the speed of 2,000 rpm. As the result, the phases were allowed to separate. The solutions were then held in temperature controlled bath at 30°C. The supernatant was withdrawn periodically and analyzed for chromate concentration. The analysis of chromate was performed using an atomic absorption spectrophotometer (Varian Spectr AA-300). The equilibrium time was established when there was no further change in the concentration of chromate.

Table 3.1 Conditions for determining equilibration time

Concentration (M)		
QUAT	CrO_4^{2-}	Ba^{2+}
0.4	0.8	0.4
0.1	0.002	0.002

3.2.2 Equilibrium Precipitation

The experimental procedure for barium chromate precipitation was the same as just given except an equilibration time of 48 hrs. 30 °C was used. The experimental conditions are given in appendix A.