CHAPTER III
EXPERIMENTAL SECTION

3.1 Materials

Nonyl phenoxypoly(ethyleneoxy) ethanol with an average of 8, 9, and 10 moles of ethylene oxide per mole of nonyl phenol, $NP(EO)_8$, $NP(EO)_9$, and $NP(EO)_{10}$ from Rhodia (Thailand) Ltd. (Igepal CO-610, Igepal CO-630 and Igepal CO-660) were the nonionic surfactants used in this study. Distilled water was used in all experiments.

3.2 Experimental Equipment

3.2.1 Ross-Miles Method Equipment

The equipment for the Ross-Miles method was made in accordance with the ASTM standard D1173-53. The apparatus consists of two parts, the pipette and the receiver. The bulb of the pipette has 45 $\pm$ 1.5 mm outside diameter and its ends are hemispherical. The upper part of the bulb is connected to a stem ending with a stopcock. The lower part of the bulb is connected to another stem of 7 $\pm$ 0.5 mm outside diameter and length 60 $\pm$ 2 mm. At its lower end is fitted an orifice of 2.9 $\pm$ 0.02 mm inside diameter and a length of 10 $\pm$ 0.05 mm constructed from precision bore tube with ends ground square. This orifice is sealed to the stem. The pipette is calibrated to contain 200 $\pm$ 0.2 mL at 20^{o}C. The pipette is shown in Figure 3.1.

The receiver as shown in Figure 3.2 is a jacketed tube of 50 mm internal diameter. The external diameter of the jacket is 70 mm. The lower end of the receiver has a stopcock to drain the liquid. There are three marks on the receiver, one at the 50 mL point measured with the stopcock closed and is at the cylindrical part of the tube.

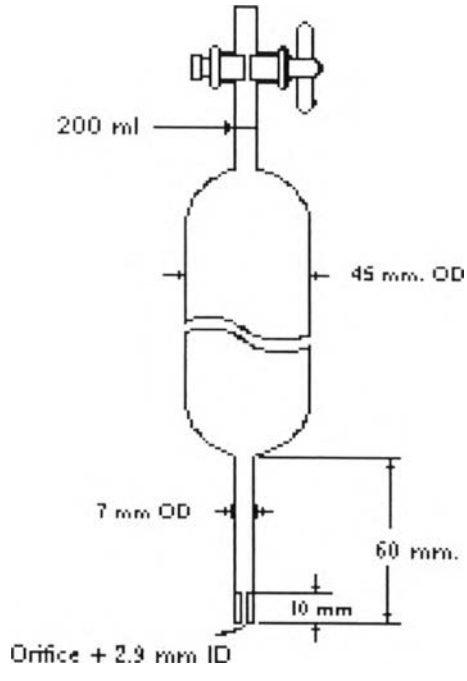

Figure 3.1 The Ross-Miles pipette.

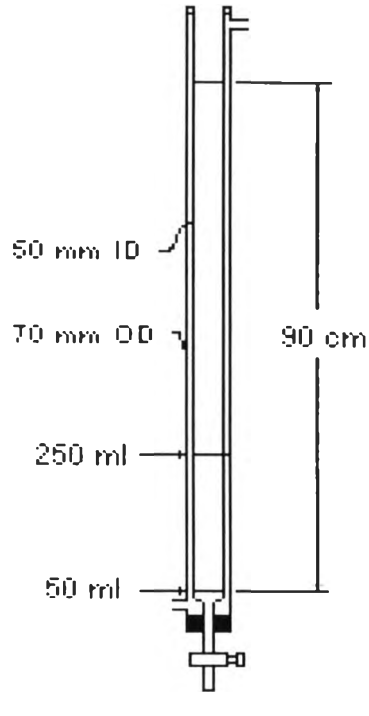

Figure 3.2 The Ross-Miles receiver.

The second mark is at the 250mL point and the third is at 900 mm above the 50 mL mark. Figure 3.3 shows the mounting of the pipette on top of the receiver when in use.

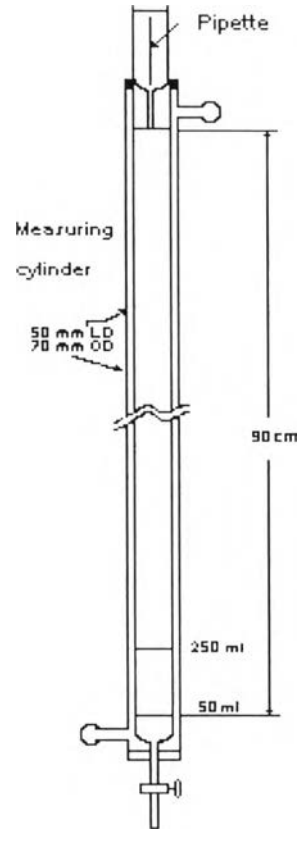

Figure 3.3 Schematic of equipment for Ross-Miles foam test.

3.3 Experimental Methods

3.3.1 Cloud Point Test

The determination of the the cloud point temperature was carried out in accordance with the ASTM D 2024-65. 0.02 M of the surfactant

solution was prepared at a temperature less than 30°C. The solution was agitated until the surfactant was completely dissolved and 50 ± 5 mL of the solution was transfered to a 25x200 mm test tube. The solution was then heated in a water bath until the solution became cloudy by stirring occasionally with a thermometer. The solution was then removed from heat and stirring was given occasionally until the solution was again clear. The temperature at which the solution became clear was recorded to the nearest 1°C. This temperature was taken as the cloud point of the solution.

3.3.2 Ross-Miles Method

This method was carried out by preheating the water to be used to the desired temperature and adjusting the thermostat of the circulation bath so as to bring the water jacket of the receiver to the desired temperature. Then the pipette was mounted by using a clamp to ensure that the axes of the measuring receiver and the pipette must be in line with the upper calibration mark on the measuring receiver. A meter ruler was fastened behind the measuring receiver ensuring that its zero point coincided with the 250 mL calibration mark on the measuring receiver. The walls of the receiver was then rinsed with the test solution using approximately 50 mL solution. When the liquid had drained to the bottom of the receiver, the stopcock was adjusted so that the level of the solution was exactly at the 50 mL mark in the receiver. The pipette was then filled with the test solution up to the 200 mL calibration mark by applying a slight suction. It was immediately placed in position on top of the receiver and the solution was allowed to flow into the receiver until the solution was run out. The foam height was immediately measured at this moment and it was measured again at 5 minutes after the flow had been stopped.

3.3.3 Phase separation

The surfactant solution was prepared at 0.02 M in a 5000 mL beaker which was completely sealed by aluminum foil to prevent evaporation. The solution was kept at the desired temperature above the cloud point in a water bath. The concentration of the dilute phase was sampled and measured by UV spectrometer at 223 nm (Schmitt, 1992) every 12 hours until the separation reached the equilibrium. The dilute phase was then separated out from the coacervate phase. After that the concentration and foamability of both dilute phase and coacervate phase were measured.

3.3.4 Surface Tension and Interfacial Tension Determination

The surface tension was measured in accordance with the ASTM standard D 1331-89. The ring of Digital Tensiometer K 10 ST (KRUSS Instruments) was obtained from Lecompte Du Nouy. The ring specifications are as follows: platinum-irridium type, wetting length 119.95 mm, ring-radius 9.545 mm, and wire radius 0.185 mm. Before measurement, all glass materials were cleaned by using cleaning solution, followed by a thorough rinsing with distilled water. The platinum ring was rinsed by distilled water and then heated to white heat in the oxidizing portion of a gas flame of an alcohol burner. The accuracy of tensiometer was checked by triple-distilled water before use.

The digital display and balance beam of tensiometer were adjusted to zero. The vessel cell and the liquid sample were preheated at the desired temperature. The cleaned vessel cell containing the liquid sample was rinsed three times for each experiment. The sample was loaded into the vessel and the ring was dipped into sample. The pointer of balance beam was moved to a negative value. The temperature of the liquid sample was checked again. If the sample temperature equalled the desired temperature, then the instrument was switched on. When the pointer of the balance beam returned to zero again, the

value of surface tension was shown on the instrument. Each experiment was repeated five times with the same sample. The interfacial tension was measured by following the surface tension method, but the zero adjustment was adjusted at the interface of the coacervate phase then the dilute phase was poured slowly onto the coacervate phase. After that the switch was turned on.

3.4 Determination of Surfactant Concentration

The surfactant concentrations were measured by using a Cecil Instrument model CE 2000 series UV spectrometer at 223 nm.