CHAPTER III
EXPERIMENTAL

Chemicals

1. Neopentyl glycol (2,2-dimethyl-1,3-propanediol, analytical grade) was obtained from Fluka.
2. Trimethylol propane (1,1,1-Tris[hydroxymethyl]propane, analytical grade) was obtained from Fluka.
3. Pentaerythritol (analytical grade) was obtained from Fluka.
4. Heptanoic acid (analytical grade) was obtained from Fluka.
5. Octanoic acid (analytical grade) was obtained from Fluka.
6. 2-Ethyl-hexanoic acid (analytical grade) was obtained from Fluka.
7. Nonanoic acid (analytical grade) was obtained from Fluka.
8. Sulfuric acid (98 %,analytical grade) was obtained from J.T. Baker.
9. Diethyl ether (reagent grade) was obtained from J.T.Baker.
10. Sodium sulfate anhydrous (reagent grade) was obtained from Fluka.
11. Sodium hydrogen carbonate (reagent grade) was obtained from Fluka.
12. Toluene (reagent grade) was obtained from local supplier.

Apparatus and Instruments

1.Fourier-Transform NMR spectrometer: Model AC - F 200 (200 Mhz.), Bruker spectrospin

2.Fourier-Transform IR spectrometer: Model 2000, Perkin Elmer

3.Colourimeter : The Fisher ASTM (D1500)

4.Viscometer : Model K-234 A, Hochler Instrument Co; Inc.

5.Pour point Tester : Model A82, HAKKE

6.Flash point Tester : Model Cleveland semi-automatic

7.Thermogravimetric Analyzer : Model STA 490 C, Netzsch

Experimental Procedure

1.Synthesis of polyol ester : General procedure for the esterification of heptanoic acids with neopentyl glycol.

Concentrated sulfuric acid (0.7 ml) was added into neopentyl glycol (52 g, 0.5 mole) dissolved in toluene (40 ml.). This mixture was added to heptanoic acid (130 g, 1.0 mole) contained in a 500 ml. round bottomed flask fitted with a Dean-Stark apparatus, and magnetic stirrer. The mixture was heated at 130 ^{0}C in an oil bath with continuous stirring for 3 hours. The reaction mixture was allowed to cool to room temperature and then was neutralized with saturated sodium bicarbonate solution. The mixture was dissolved in diethyl ether (50 ml.) and washed with distilled water (3 times, 30 ml.) and organic layer was dried with anhydrous sodium sulfate. Then, diethyl ether and toluene were removed under reduced pressure to obtain neopentyl glycol bis(heptanoate), (153.80 g., 93.76 % yield).

Other polyol esters were synthesized under the same conditions.

2.Determination of physical and chemical properties of polyol ester products.

2.1 Physical properties

2.1.1 Color, ASTM	by ASTM D 1500
2.1.2 Kinematic Viscosity	by ASTM D 445
2.1.3 Viscosity index	by ASTM D 2270
2.1.4 Pour point	by ASTM D 97
2.2.5 Flash point	by ASTM D 92

2.2 Chemical properties

2.2.1 The characterization of polyol ester products were determined by ^{13}C-NMR and FTIR analyses.

2.2.2 The oxidation point and the percentages of oxidative compounds were determined by TGA method under air atmosphere and used the following conditions.

TGA condition

Heating rate	: 5^{0}C/ min.
Temperature range	: Ambient to 650 ^{0}C.
Atmosphere	: Dynamic air 100 ml / min.
Reference	: Al_2O_3