CHAPTER V

CONCLUSION

From the result of this research, it can be concluded that :

Additives in commercial lubricants were isolated by dialysis in a rubber surgery fingercot against hexane in a soxhlet extractor. High-molecular weight additives in dialysis residue were purified by solvent precipation with MEK and analyzed for the purity by GPC. Then, the additives obtained were characterized by IR and NMR technique. The results indicated that all of the lubricating oil samples contained polyisobutylene derivative. Low-molecular weight additives in dialysate fraction were extracted from dialysate using methanol and purified by silica gel column chromatography. The gradient elution was carried out using hexane, carbon tetrachloride, chloroform and acetone respectively. Each fraction of additives was allow to dry. The identity of each additive was characterized by IR and NMR spectroscopy. Phenolic and aminic type antioxidants together with antiwear additives were found to be presence in the dialysate fraction. Antiwear additive was identified as zinc dialkyldithiophosphate.

Thin-Layer Chromatographic technique was developed for convenient identification of additives from commercial lubricants. The commercial lubricants were analyzed by silica gel coated on glass plate using carbon tetrachloride and 20% acetone in carbon tetrachloride as solvents. The spots were visualized by using the suitable reagents such as ammonium molybdate in acidic solution at a elevated temperature (70-80°C.). Each spot of additives was scrapped of the plate and acetone was used to extract the additive from each spot. The acetone solution was concentrated and characterized by FTIR spectrophotometer. The results were compared to those obtained from column chromatographic technique. It was found that TLC-FTIR technique could be used conveniently to characterize the additives in commercial lubricants, in addition, each isolated additive was purer than the one obtained from the column chromatographic technique.

The partial composition of each lubricant studied in this research could be summarized as the following.

Lubricating oil : PTT-V120 (31.40 g.)

Additives found

- Zinc Dialkyldithiophosphates (ZDDP) : multifunctional additive 0.223 % by weight.
- Polyisobutylene derivative : viscosity index improver additive 2.229 % by weight.
- Unknown component < 0.1 % by weight.

Lubricating oil : Caltex Havoline (32.0 g.)

Additives found

- Zinc Dialkyldithiophosphates (ZDDP) : multifunctional additive 0.223 % by weight.
- Polyisobutylene derivative : viscosity index improver additive 4.156 % by weight.
- Sterically hindered phenol : antioxidant additive 0.50 % by weight.

Lubricating oil : Esso Superflo (30.06 g.)

Additives found

- Zinc Dialkyldithiophosphates (ZDDP) : multifunctional additive 0.765 % by weight.
- Polyisobutylene derivative : viscosity index improver additive 13.24 % by weight.
- Aromatic amine : antioxidant additive 0.366 % by weight.

Lubricating oil : Shell Helix (35.40 g.)

Additives found

- Zinc Dialkyldithiophosphates (ZDDP) : multifunctional additive 0.508 % by weight.
- Polyisobutylene derivative : viscosity index improver additive 3.813 % by weight.
- Sterically hindered phenol : antioxidant additive 0.763 % by weight.

Suggestion for future work

1. Solvent system for each type of additive should be optimized in order to identify each additive by type.
2. The result could be better and more convenient if on-line TLC-FTIR experiment could be carried out. However, the on-line experiment should be tried by using information obtained from this research.
3. From this work, the isolation and characterization of additives in commercial lubricating oil is quite complicated, therefore new techniques for characterization and quantification should be developed for future use.