CHAPTER FIVE

CONCLUSIONS

From this study, new HPLC and GC methods for the determination of thermal heating fluids and diesel fuel have been developed.

The application of a HPLC with fluorescence detection (247 nm excitation and 310 nm emission) for the determination of the eutectic mixtures of diphenyl oxide and biphenyl in vegetable oils and oleochemicals has been adequately demonstrated. The sample solutions are injected into a reversed-phase column and eluted with a simple isocratic mobile phase mixture of methanol and water (90:10, vol/vol). An intra-laboratory study showed that the mean recoveries of the fluid from various spiked samples ranged from 90.9-108.7%, with an LOQ of 0.1 μg/mL. An inter-laboratory study also gave good mean recoveries of 93.0-116.0%, with the reproducibilities of 1.29-3.84%.

The suitability of a reversed-phase HPLC-fluorescence detection (257 nm excitation and 320 nm emission) method for the analysis of the partially hydrogenated terphenyls in vegetable oils and oleochemicals was also demonstrated in this study. While the oleochemical samples are analyzed directly, saponification of vegetable oils is required for the removal of the interfering fluorescence components prior to analysis. The calibration graph obtained from various concentrations of the fluid is linear with $r^2$of 0.999 and an LOD of 0.01 μg/mL.

Based on the analysis of a range of spiked samples, the mean recovery of the fluid for spiked oleochemicals was 95.3% and 87.0% for spiked vegetable oils.

Further investigations in this study shows that a reversed-phase HPLC with fluorescence detection (270 nm excitation and 320 nm emission) is suitable for the analysis of the synthetic hydrocarbons in oleochemicals. However, the analysis can only be performed after pre-concentration *via* a normal phase preparative column chromatography due to the weak fluorescence property of the fluid. The mean recoveries of the fluid at 3 spiked concentration levels are all within 70-80%, with a CV of lower than 10% based on 6 determinations at each concentration. However, due to the presence of other interfering fluorescent components, the GC-FID technique was found to be more suitable for the determination of the synthetic hydrocarbons in vegetable oils. The mean recoveries obtained from different spiked concentrations ranged from 68.0-86.0%, with CV of between 6.54-9.75%. The LOQ in this method was similar to that of the HPLC method which is 30 μg/g.

Finally, the detection of diesel fuel in vegetable oils by using a normal-phase HPLC system was also investigated. The analysis was performed using a mobile phase of heptane and isopropanol (94:6, vol/vol) and a fluorescence detection at 286 nm (excitation) and at 321 nm (emission). Although baseline resolution between diesel and other background fluorescence components could not be achieved, the quantitative determination is still possible after using the baseline integration method. The mean recoveries ranged from 94.4-101.3%, with an LOQ of 5 μg/g for most vegetable oils.

All the analytical methods developed in this study are quite straightforward and simple. In the case of the HPLC method for the determination of the eutectic