

CHAPTER V

CONCLUSION

According to the gas chromatographic separation of phthalate esters, i.e., DMP, DEP, DBP, BBP, DEHP and DOP on two GC columns shown in Figure 4.1-4.2. The results show that HP-1 (Crosslinked Methyl Silicone Gum) capillary column can give better separation of phthalate and less analysis time. Therefore, HP-1 (Crosslinked Methyl Silicone Gum) capillary column is chosen to analyze some phthalate esters, i.e., DMP, DEP, DBP, BBP, DEHP and DOP. To increase sensitivity of GC and obtaining reproducible, accurate and representative quantitative results, various parameters, i.e., purge time, split ratio and make-up gas flow rate are studied and results are shown in Table 4.1-4.3. The purge time at 1.0 min, split ratio of 25:1 and make up gas flow rate at 60.0 mL/min are chosen as the optimum condition of GC for the analysis of phthalate esters.

Analysis of phthalate esters i.e., DMP, DEP, DBP, BBP, DEHP and DOP in water by solid phase extraction technique, various factors having effect on the percent recovery are studied and results are shown in Tables 4.5-4.21. The pH value of the extracted solution at 2.0, 500 mg of sorbent mass, toluene as elution solvent and elution volume at 3.0 mL are chosen as the optimum conditions of solid phase extraction for the determination of phthalate esters in water samples. The results of study pH effect, elution solvent and volume of elution solvent in single component and mixture solution with two concentrations are

presented in Tables 4.5-4.12 and Tables 4.14-4.21, respectively. The results can be concluded that the percent recovery of each phthalate ester is independent of the concentration of the compounds. Moreover, the percent recovery of each phthalate ester in single component and mixture solution is shown insignificantly different. Therefore, this technique can be used to analyze each phthalate ester in a single component the same as in mixture components.

The precision of each phthalate ester is studied in water by using toluene and ethyl acetate as eluent solvent and is shown in Tables 4.24 and 4.25, respectively. By using toluene as eluent, the percent recovery and the percent relative standard deviations for the determination of DMP, DEP, DBP, BBP, DEHP and DOP are 102.83 ± 2.19 , 105.24 ± 1.83 , 96.39 ± 1.37 , 93.11 ± 2.66 , 88.22 ± 2.28 , 88.01 ± 2.61 , respectively. By using ethyl acetate as elution solvent, the percent recovery and the percent relative standard deviations for the determination of DMP, DEP, DBP, BBP, DEHP and DOP are 95.27 ± 2.81 , 95.75 ± 2.64 , 92.03 ± 1.80 , 91.61 ± 1.98 , 87.74 ± 1.59 and 87.42 ± 1.97 , respectively. This result indicates that ethyl acetate which is less toxic than toluene and also gives the high percent recovery can be used as another elution solvent for determination phthalate esters in water with good precision the same as using toluene as eluent.

The method detection limit of DMP, DEP, DBP, BBP, DEHP and DOP presented in Table 4.23 are 1.24 ppb, 1.26 ppb, 0.69 ppb, 0.52 ppb, 0.51 ppb and 0.68 ppb, respectively. The accuracy of this technique is also evaluated by determining the synthetic unknown solution of

mixture phthalate esters and the results are shown in Table 4.26. The percent errors of DMP, DEP, DBP, BBP, DEHP and DOP are 1.24, 0.03, 0.12, 0.41, 2.00 and 0.73, respectively. It is shown that this technique can be determined these phthalate esters in the ppb concentration with high confidential results.

To verify that this technique is suitable for the analysis of real samples, fifteen water samples collected from several places are analyzed by this solid phase extraction technique and the results of the analysis that are shown in Table 4.27. The results indicate that thirteen water samples have DEP, DBP and BBP in the range of 0.024-1.92 ppm, 0.012-0.151 ppm and 0.023-0.024 ppm, respectively and the interested phthalate esters could not be found in two drinking water samples.

In this work, the solid phase extraction technique can be developed for the trace analysis of the phthalate esters in water samples. High percent recoveries are obtained as a result of none formation of emulsion, a few sample transfers, and small volumes of extraction solvent usage that is little or no evaporation. It is also simple, rapid, precise, accurate, economical and safe method.

For the future work, the study of higher sample volume should be done in order to enrich the final concentration of the extraction. The flow rate in retention and elution step should be studied to enhance the percent recovery and reduce analysis time with increasing the flow rate. The investigation of the other phthalate esters in water samples and various environmental samples should be also considered.