CHAPTER V

CONCLUSION

According to the analysis of volatile organic compound i.e., methylene chloride, chloroform, benzene, trichloroethylene and 1,4-dioxane in drugs by using headspace can increase the sensitivity by various factors such as temperature, equilibration time, liquid to gas phase ratio and salting out effect are studies and the results are shown in Table 4.1-4.22. The temperature at 70°C, the equilibration time 40 minutes and the liquid to gas phase volume ratio of 5:5 in 10 mL vial for headspace are selected as the optimum headspace analysis condition and it would give a better sensitivity for the determination of the volatile organic compounds in drug samples. The effect of no adding salt and adding anhydrous sodium sulfate into the solution on the sensitivity and the percent recovery are also studied and the results show that adding anhydrous sodium sulfate gives the higher sensitivity and percent recovery for each interested compound. Hence, the adding anhydrous sodium sulfate is more suitable to used for their determination in aqueous drug samples than non adding salt. Therefore, the following condition namely liquid to gas phase volume ratio of 5:5, equilibrating at 70°C for 40 minutes and 1.00 g anhydrous sodium sulfate is selected as the optimal headspace analysis condition for the determination of the volatile organic compound in drug samples.

The precision of this technique is also evaluated by determining the known mixture solution and the results are shown in Table 4.26 for using FID as a detector and Table 4.27 for using ECD as a detector. The average concentration and the

percent relative standard deviations for the determination of methylene chloride, chloroform, benzene, trichloroethylene and 1,4-dioxane using FID as a detector are 4.17 ± 1.80, 4.21±2.48, 4.19 ± 3.04, 4.21 ± 3.00 and 99.26± 2.02, respectively and using ECD as a detector are 20.23± 1.97, 20.40 ± 2.43 and 20.21 ± 2.24 for methylene chloride, chloroform and trichloroethylene, respectively.

The method detection limit of methylene chloride, chloroform, benzene, trichloroethylene and 1,4-dioxane using FID as a detector are 0.10 ppm, 0.20 ppm, 0.08 ppm, 0.20 ppm and 12.41 ppm, respectively and using ECD as a detector are 2.5 ppb, 0.4 ppb and 0.2 ppb for methylene chloride, chloroform and trichloroethylene, respectively.

The linearity of this technique is also evaluated by determining the various known mixture solution and the results are shown in Table 4.30 and Table 4.31 for using FID and ECD as a detector, respectively . The concentration range and correlation coefficient using FID as a detector of methylene chloride, chloroform, benzene, trichloroethylene and 1,4-dioxane are 0.42-151.60 ppm , 0.9988 , 0.42-151.72 ppm , 0.9994 , 0.38-148.90 ppm , 0.9993 , 0.42-151.58 ppm , 0.9997 and 12.41-223.34 ppm , 0.9973, respectively for using ECD as a detector are 4.21-1052.80 ppb, 0.9990 , 4.24-1060.55 ppb, 0.9989 and 4.21-1052.65 ppb , 0.9984 of methylene chloride, chloroform and trichloroethylene, respectively.

The accuracy of this technique using FID as a detector is also evaluated by determining the unknown synthetic mixture solution using external standardisation method and the results are shown in Table 4.32. The percent error is in the range of 0.42-5.06. To verify that this technique is suitable for the analysis of real samples,

fifteen drug samples bought from several company are analysed by this technique and the results of the analysis indicated that three drug samples seem to have methylene chloride in ranges of 38.10 - 202.10 μg/g, one drug sample seem to has chloroform 957.10 ng/g. Moreover, some drug samples can be found the other organic compound i.e., cyclohexane, methylamine, pentanone, ethanol, methanol, tetrachloroethylene, acetamine, and carbondisulfide.

For the future work, the studies of the different salts should be interested in order to enhance the sensitivity and the percent recovery of this technique. The investigation of the other organic volatile impurities and other residual solvents in drug samples i.e., liquid, colloidal and solid should be also considered by using the headspace technique.