Chapter 4

CONCLUSION

A method for acetaldehyde analysis was developed using a gas- liquid chromatographic coupled with airspace techniques. The injector of the gas chromatograph was modified to be used as a gas sampling valve system resulted in good reproducibility, precision, and simple injection. Acetaldehyde standard gas was prepared by airspace technique in a 500 mL gas sampling bulb and was analyzed with a DB^{-1} capillary column, 15.0 m × 530 μm ID. × 3.00 μm film thickness and flame ionization detector (FID). The optimum conditions for the GC technique were, carrier gas flow rate 8.0 mL min^{-1}, column temperature 130 °C (isothermal), detector temperature 270 °C, valve temperature 140 °C, fuel gas flow rate 30 mL min^{-1}, oxidant gas flow rate 300 mL min^{-1}, make up gas flow rate 20 mL min^{-1} and valve heating time 0.3 min. For the airspace technique, the equilibration time was 2 min. These optimum conditions provided a short analysis time (1.5 min), low detection limit (0.3 ng mL^{-1}) and wide linear dynamic range (0.3 ng mL^{-1} to 6.6 μg mL^{-1}) with a linear regression coefficient of more than 0.99.

To reduce sample preparation time of the conventional method for acetaldehyde analysis, a lab- built heating box was used. The proposed method, can reduce the time from 24 hours to one hour at an incubation temperature of 60 °C. This technique allowed the rapid release of acetaldehyde from plastic material into the airspace within the bottle. The gas was then analyzed using gas chromatographic technique at optimum conditions. Using the *t*-test, it was shown that there was no significant difference between the conventional and proposed method.

To reduce the complication of sample transportation and preconcentration another sample preparation was also developed to be used in a purge and trap technique. Acetaldehyde migrated from PET bottle was purge with N_2 at a flow rate of 70 mL min^{-1} for 15 min through the absorbent (Porapak Q 80/ 100 mesh) in the absorbent tube. The trapped acetaldehyde on the adsorbent was then desorbed at 215 °C for 0.6 min with a desorption flow rate of 70 mL min^{-1}. Using Mann Whitney rank

significant different at 99% confidence level. The results showed that qualitative and quantitative analysis of trace level of acetaldehyde residue in PET bottle could be obtained with high precision.

For qualitative and quantitative analysis of acetaldehyde in real sample, PET bottles were collected from Haad Thip Co. Ltd., (the Coca-Cola bottled company in the South). Three types of bottle, Fanta, Sprite and Coke, 1.25 and 2 l, were analyzed using conventional, proposed and purge and trap methods as sample preparation techniques. Gas chromatography at optimum conditions was used as the analysis method. The relative standard deviation (% RSD) was lower than 10%. The amount of acetaldehyde residue in PET bottle was in the range of 0.4 to 1.1 ng mL^{-1}. This was less than the EU SCF limitation (0.6 mg kg^{-1} bottle weight) and Coca-Cola specification (5 μg L^{-1}). When comparing the results, there were no significantly different between conventional and proposed method as well as the proposed method and purge and trap technique.

In conclusion, the proposed method, with a lab- built heating box and purge and trap system, can be used for qualitative and quantitative analysis of acetaldehyde residue in PET bottle with good precision. The advantages of the proposed method are simple, rapid, no solvent needed, low detection limit, can be used in industrial application and all sizes of the bottles can be analyzed. The purge and trap technique needed more time (15 minutes) to adsorbed acetaldehyde on adsorbent and needed a thermal desorption unit to desorbed acetaldehyde. However, it can be used in order to reduce the cost, the complication of sample transportation, and also preconcentration. For example, when there is not enough time to analyze the sample, or the laboratory is far away from the field.