CHAPTER 4

CONCLUSION

The optimized extraction and automated headspace sampler conditions were found to be applicable to quantification of 2-AP in rice seed extract by HS-GC using DB-17MS capillary column. GC conditions are injector temperature at 200 °C, detector temperature at 230 °C, carrier gas flow rate of 3.0 ml/min and the programmed temperature started at 45 °C, hold for 2 min then ramped up to 80 °C with rate 5 °C/min.

In this work, DB-1701 capillary column was used for optimization. GC conditions are injector temperature at 200 °C, detector temperature at 230 °C, carrier gas flow rate of 5.5 ml/min and the programmed temperature started at 45 °C, hold for 2 min then ramped up to 80 °C with rate 5 °C/min. The HS-GC employed both column was run for 11 min. Before subjecting to analysis by HS-GC, the aroma compound, 2-AP was extracted from rice seed by acidic solvent extraction. The extraction parameters affecting sensitivity and separating by HS-GC consists of extraction time, volume of 1.0 M NaOH used for making solution alkaline and equilibrium time. Rice seed extract solution of KDML 105 was used for optimization. The optimum conditions of extraction were found to have included the following: rice seeds were extracted by 0.1 M HCl for 30 min, then the solution was made alkaline by adding 0.5 ml of 1M NaOH and allow to equilibration for 15 min. Then, the headspace of extract solution was analyzed by HS-GC.

The automated headspace sampler parameters consists of temperature of oven, loop and transfer line, vial equilibrium time, pressurizing time, loop fill time, loop equilibrium time and loop injection time. The optimum conditions of automated headspace sampler were as follows: oven temperature, 120 °C; loop temperature, 140 °C; transfer line temperature, 160 °C; vial equilibration time, 3 min; pressurization time, 0.3 min; loop fill time, 2.0 min; loop equilibrium time, 0.04 min and loop injection time, 0.3 min. The rice seed extract solution in vial can be agitated with temperature at 120 °C for 3 min to establish equilibrium. After equilibrium has been reached, the vial is pressurized by nitrogen gas for 0.3 min. Then the vent valve opens and the headspace gas was filled the sample loop for 2.0 min and equilibrated in sample loop for 0.04 min. The carrier gas subsequently flowed through the sample loop for 0.3 min. The headspace gas was then swept into the GC/FID.

Detection limits in term of the lowest amount of rice seed sample required was 4.00 g. Detection limit in term of the least amount of analyte, 2-AP, was 0.23 ppm. Linearity ranges were in the range 0.37-5.93 ppm with a correlation coefficient of 0.9991. The precision was reproducibility. The relative standard deviation of reproducibility was 7.5%. Recovery assay of standard solution extraction was 74.1%. Recovery assay of standard solution extraction of KDML105 Chiang mai, KDML105 Tungkularonghai and Howm Supanburi were 79.27, 76.58 and 76.28%, respectively.

The optimal conditions of extraction and automated headspace sampler were employed for quantification of 2-AP in brown rice samples. The headspace of the extract was subjected to analysis by HS-GC. The DB-17MS column was utilized for quantification of 2-APin rice seed extracts in order to obtain higher resolution, so that peaks overlapping with interfering peaks could be avoid. In this work, DB-17MS column was found suitable for seperation components in four of brown extracts. Four of brown rice samples were analyzed by HS-GC. 2-AP was detected in KDML105 Chiang mai, KDML105 Tungkularonghai and Howm Supanburi at the concentration of 1.18 ppm, 2.41 ppm and 0.41 ppm, respectively. No 2-AP was detected in Howm Patumtani rice.