

## CHAPTER V

### CONCLUSIONS

A simultaneous analysis of 17 organochlorine pesticides by GC- $\mu$ ECD was developed. A good separation of 17 compounds was achieved by using GC conditions as mentioned in the previous chapters (Table 4.1). The limit of detection was in the range of 0.3 – 12.3 ng/mL and the limit of quantitation was at 1.1 – 41.0 ng/mL. Quantitation is by the matrix calibration curve with an acceptable range of  $R^2 > 0.99$ . A new multi-residue method covering 17 organochlorine pesticides in Turmeric powder was developed. The simple preparation of interested pesticides was created to extract pesticides by using sample 2 g and adding a mixed solvent between hexane and dichloromethane in the ratio of 5:2, 25 mL and then the fortified solutions 10 mL were passed through the mixed mode of SPE—silica gel, florisil and anhydrous sodium sulfate—for clean-up. In the preparation of SPE, it had to be conditioned with dichloromethane first and then the sample solutions were loaded. In the final stage, elute of interested compounds were mixed with the solvents in the ratio of 5:2. After the liquid had evaporated and the final volume was reduced to 1 mL in vial, it was finally determined by GC- $\mu$ ECD. The developed method has been validated in many relative terms. Its method detection limit is in the range of 0.5 – 15.4 ng/g and its method quantitation limit is in the range of 1.4 – 51.2 ng/g.

Quantitation is by matrix calibration curve with an acceptable range of  $R^2 > 0.99$ . The result of the study of matrix effect showed that the sample in matrix is significantly different from the standard solution in hexane. The precision of developed method and the %recoveries of all pesticide were studied intra-assay precision and intermediate precision at the spiking level of 5 ng/g, 25 ng/g and 125 ng/g. The %RSD of carbophenothion and  $\beta$ -BHC, HCH can't be detected because

carbophenothion had MQL higher than 5 ng/g and  $\beta$ -BHC, HCH had MQL higher than 25 ng/g.

All of pesticides at the spiking level of 125 ng/g, intra-assay precision are less than 15% from regulation of AOAC. Concerning the intermediate precision, all of pesticides are of less than 15%. About %recovery at spiking level 125 ng/g that more than MQL of all interested organochlorine pesticides are in the range of 60.33 – 180.64%. Therefore, the developed extraction method is acceptable for used to standard method except endrin because it is less than 70% recovery from the regulations of AOAC and  $\beta$ -BHC, HCH, alachlor, P,P'-DDD and carbophenothion because it is more than 125% recovery from the regulations of AOAC (70 – 125 %recovery in ng/g level). As for  $\beta$ -BHC, HCH and carbophenothion, they yielded a more %recovery than that specified in the regulations. Thus, the extraction by means of the multi-residue method that had been developed earlier was not suitable for the analysis of these two substances. This prompted the author to make changes to the method so that it could be used effectively in extracting the two substances. In this thesis, heptachlor,  $\beta$ -endosulfan, endosulfan-sulfate and methoxychlor had to study but %recovery of theirs couldn't acceptable because %recovery is much less than 70.

As for the stability of organochlorine, 17 pesticides could be kept, in general, no longer than three days starting on the day they were extracted and they had no effect on the results analyzed except  $\beta$ -BHC, HCH (5 days) and O,P'-DDE (15 days). In Turmeric powders from 3 Thai markets and 3 commercial products, all of them have the concentration of DDT and metabolites of DDT (DDE and DDD) in low concentrations and under the limit of the USP regulations, 0.05 – 3.00 ppm (mg/kg). In general, both DDT and DDT metabolite were found in the Thai Turmeric powder. The amounts of these contaminants were usually very slight. Yet, the method the author developed could detect the contaminants even though they existed at a very low level. This shows that the method is very effective and can detect slight traces of contaminants. From all the results, as described above about the method of validation for the determination of organochlorine pesticides in Turmeric powder, the developed sample preparation technique is most suitable for the standard method for Thailand.

**Table 5.1** Summary of retention time, resolution, correlation coefficient ( $R^2$ ) of matrix calibration curve, MDL and MQL of the developed method for organochlorine pesticides in Turmeric.

No.	Pesticides	Retention time (min)	Resolution	$R^2$ of matrix calibration curve	MDL (ng/g)	MQL (ng/g)
1	$\alpha$ -BHC, HCH	15.394	-	0.9980	0.9	2.9
2	$\beta$ -BHC, HCH	16.697	10.24	0.9952	15.4	51.2
3	$\gamma$ -BHC, HCH	16.922	1.70	0.9978	0.5	1.8
4	Alachlor	20.478	2.21	0.9977	1.2	3.9
5	Aldrin	22.279	15.73	0.9973	0.6	2.0
6	Heptachlor epoxide isomer B	24.479	18.80	0.9970	0.9	2.9
7	Heptachlor epoxide isomer A	24.728	2.10	0.9946	0.7	2.2
8	O,P'-DDE	26.175	11.66	0.9966	1.1	3.8
9	$\alpha$ -endosulfan	26.436	2.06	0.9982	1.3	4.3
10	Dieldrin, P,P'-DDE	27.942	9.13	0.9978	0.4	1.4
11	O,P'-DDD	28.416	2.85	0.9978	1.4	4.5
12	Endrin	29.073	5.02	0.9986	1.0	3.5
13	P,P'-DDD	30.303	4.71	0.9978	1.4	4.8
14	O,P'-DDT	30.434	0.91	0.9979	1.0	3.4
15	Carbophenothion	31.813	9.47	0.9969	7.1	23.8
16	P,P'-DDT	32.366	2.23	0.9977	0.6	2.1