ANALYSIS OF PCBS IN FOOD BY THE CONGENER SPECIFIC METHOD

Junghyuck Suh, Dongmi Choi, Kyoungpoong Won, Changmin Kim, Insang Song

#5 Nokbun-Dong, Eunpyung-Ku, Korea Food and Drug Administration, Seoul 122-704, Korea

Introduction

PCBs are assumed as the endocrin disrupters as dioxins that are highly persistent compounds due to their bioaccumulated property and long half-life[1]. Since it is increased general concern of the human exposure on PCBs derived from food, it is needed to study the level of PCBs in food. However, there is a limited report about PCBs in food comparing to other environmental samples such as soil or air[2,3]. During 1992 ~ 1996, the Korea Food and Drug Administration (KFDA) conducted the monitoring on PCBs in foodstuff and determination was based on a peak pattern method[4]. But such information is very complecated to interpret the measured PCBs regarding commercial PCB formulations(Aroclors). Because GC patterns produced by PCBs in food samples are different from Aroclor patterns. To improve these kinds of disadvantage, the congener-specific approach has been proposed for analyzing PCBs in food. So far several contries in Europe have the standardised method by the indicator PCBs such as PCB congener 28, 52, 101, 118, 138, 153 and 180. The selection of the indicator PCBs was based on aboundance, chromatographic resolution, response and availability as a standard[5]. In addition USA and Japan have the established congener specific method[6,7] that estimates the homolog total by the level of chlorination. Thus, the aim of this study is to apply the congener specific method for analyzing PCBs in food and estimate total PCBs in a sample by sum of the concentration of the PCB congeners and congener groups. For the present study, 62 PCB congeners were selected as the target chemicals as shown in Table 1 and to evaluate the method performence, beef was chosen as reference matrix which was demonstrated not to contain PCBs.

Method and Material

The composited standard(BP-EC, Wellington) and the internal standard were prepared in iso-octane. For cleaning up, Silica gel column(glass, 2cm x 3200cm) was prepared by packing with sodium sulfate anhydrous(1g, Wako, PCBs grade), Silica gel(15g, Sigma, 60 ~ 200 mesh) and sodium sulfate anhydrous(1g), in order. The reference matrix(beef) was homogenized by meat mincer three times and was kept at -20℃ until analyzed. The lipid content was measured as described previously[5]. About 10g of sample was put into a flat bottom flask(500mℓ), spiked with 1

㎖ of the composited standard, added 50㎖ of 1M potassium hydroxide/ethanol solution and alkali digested in water bath at 80℃ for 1 hour with the condenser. After cooling down the digested solution to the room temperature, 30㎖ of n-hexane was added from the top of the condenser into the flask followed by rinsing the entrance of flask with 20㎖ of n-hexane. After transferring the digested solution to a separatory funnel, 20㎖ of ethanol:n-hexane(1:1) solution and 50㎖ of distilled water were added into it, shaken vigorously for 10 minutes, and then stood for 30 minutes. The organic layer was taken and the remaining aqueous layer was extracted with 50㎖ of n-hexane, twice, and combined to the organic layer. The organic layer was washed up with 50㎖ of concentrated sulfuric acid until the sulfuric acid layer became clean. And then it was back washed with 50㎖ of distilled water. To clean up the sample, the extracts were loaded onto Silica gel column and eluted with 80㎖ of n-hexane. The eluates were concentrated by rotary evaporator at 30℃. The concentrates were transferred to the test tube containing the internal standard solution and evaporated down to about 2㎖ under the nitrogen. Instrumental analysis was performed by GC/ECD. The operating conditions of GC/ECD are shown in Table 2.

Results and Discussion

On searching special property of this analytical method, it takes only one hour to operate the KOH saponification step in comparing with the soxhlet extraction system which takes 16 ~ 18 hours to extract. In addition, to clean up the sample, the extracts were directly loaded onto Silica gel column. Thus, there is no concentration step, in other words the cost and time to operate were reduced by this analytical method. In addition, it is able to decrease the loss of low chlorinated biphenyls like mono-, di- and tri chlorinated biphenyl that are strongly affected by concentration step. For qualification, the detected PCB congeners were cross-cheked with the dual column(DB-5MS and Rtx-1701) to exclude mis-interpretation, possibly. As results, the recoveries of PCB congeners are ranged from 60% to 96% as shown in Table 1. Therefore, the analytical method applied was very effective to determine PCB residues in meat sample. Further studies will be needed to include all the PCB congeners to the present congener specific method for analyzing PCBs in food.

References

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Table 1. The recovery range for the selected PCB congeners.

(n=5)

IUPAC Number		Concentration (μg/mL)	Recovery range
Monochlorobiphenyl	1, 3	5	60 – 74%
Dichlorobiphenyl	4, 8, 10, 15	1	68 – 92%
Trichlorobiphenyl	18, 19, 22, 28, 33, 37	0.5	68 – 94%
Tetrachlorobiphenyl	44, 49, 52, 54, 70, 74, 77, 81	0.1	74 – 96%
Pentachlorobiphenyl	87, 95, 99, 101, 104, 105, 110, 114,118, 119, 123, 126	0.1	86 – 96%
Hexachlorobiphenyl	128, 138, 149, 151, 153, 155, 156, 157, 158, 167, 168, 169	0.1	84 – 96%
Heptachlorobiphenyl	170, 171, 177, 178, 180, 183, 187, 188, 189, 191	0.1	84 – 96%
Octachlorobiphenyl	194, 199, 201, 202, 205	0.1	84 – 96%
Nonachlorobiphenyl	206, 208	0.1	84 – 96%
Decachlorobiphenyl	209	0.1	82 – 92%

Table 2. The operating conditions of GC/ECD

	GC/ECD
Model	HP 5890
Column	DB5-MS capillary column (0.25mm, 60m, 0.25μm) Rtx-1701 capillary column (0.25mm, 60m, 0.25μm)
Oven Temperature	80℃(2min) → 20℃/min → 200℃(10min) → 1℃/min → 215℃(20min) → 2℃/min → 260℃(20min)
Carrier gas	N_2 (2mℓ/min)
Detector Tem.	280℃
Injection mode	Splitless
Injection Tem	250℃
Injection volume	1μℓ