RAPID PRETREATMENT OF PCB-CONTAINING OIL FOR IMMUNOASSAY

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Abstract

The rapid pretreatment method of polychlorinated biphenyl (PCB) in insulating oil for immunoassay was examined. It is possible to perform elution during purification once in this method by connecting the solid-phase partition column to the solid-phase purification column. As a result, a concentration operation using a vacuum concentration becomes unnecessary and the analysis time is shortened to within 2 h. The solid-phase purification column contains silica gel coated with sulfuric acid and silica gel coated with silver nitrate. The solid-phase partition column contains original high-performance alumina. High purification ability of PCBs were obtained when 14 kinds of insulating oil were subject to rapid pretreatment and satisfactory results were obtained with regard to quantitative capacity and recovery rate. The results were useful for the screening of PCBs in insulating oil.

Introduction

The development of a method to screen the content of polychlorinated biphenyl (PCB) in insulating oil has been expected.

In Japan, it has been declared that all PCB waste must be treated by July 2016. Among approximately 6,000,000 electric apparatuses in Japan, approximately 20%–30% were classified as having a low concentration of PCBs in oil. Although one working policy of the Ministry of the Environment is to burn oil containing a low concentration of PCBs, they cannot estimate the extent of PCB pollution caused by an electric apparatus on the basis of the details or specifications on the nameplate. Several processing periods are required to burn all polluted electric apparatuses; moreover, the processing costs and energy costs involved are very high. Therefore, screening to separate only the apparatuses containing a low concentration of PCB is necessary in order to reduce the number of electric apparatuses that will eventually be burned.

This study was carried out to develop the rapid pretreatment of oil containing PCB for immunoassay. Tests were conducted to determine the following: (1) the efficiency of the purification of insulating oils, (2) dose-response curve, and (3) recovery test of PCB oil.

Materials and Methods

PCBs

The stock solution of PCBs comprised a Kanechlor mixture solution in n-hexane (KC-MIX solution), which was prepared by mixing equal quantities of different Kanechlor formulations: KC-300, KC-400, KC-500, and KC-600. These Kanechlor standards were purchased from GL Sciences, Inc.

Preparation of insulating oils

14 insulating oils were prepared. Two of these oils (“Sample 1” and “Sample 2”) are new insulating oils without PCBs. Sample 1 was purchased from Matsumura Oil, while Sample 2 was purchased from Japan Energy Corporation (JOMO). Another insulating oils (“Sample 3 – Sample 14”) are used-insulating without PCBs that were drained from used-transformers.

Pretreatment method

Three kinds of pretreatment methods were examined. “Method 1” is “multilayer silica gel column (by JIS K0311)”. “Method 2” is “purification column + solvent partition (liquid extraction using dimethylsulfoxide(DMSO) and hexane)”. “Method 3” is “purification column + solid partition (rapid pretreatment)”.

Purification column and solid partition column

A purification column and a solid partition column are both used for rapid pretreatment, as shown in Fig.1. The purification column contains silica gel coated with sulfuric acid and silica gel coated with silver nitrate. The solid partition column is filled with high-performance alumina, which has a high adsorption ability with regard to PCBs. The alumina was improved to the alumina for dioxin analysis[1, 2].

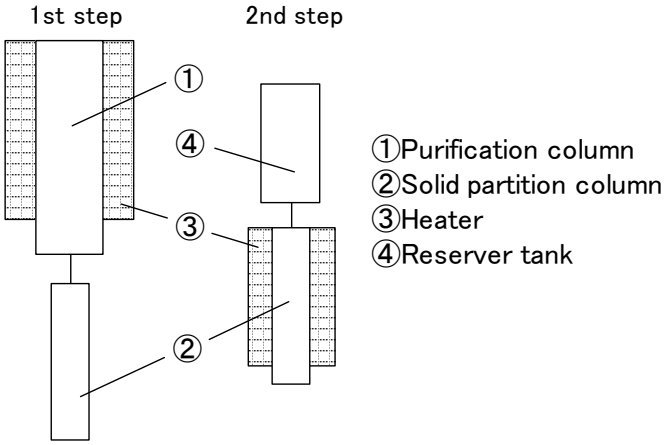

Fig.1 Columns for rapid pretreatment

Rapid analysis of PCBs in insulating oils

The procedure for rapid analysis of PCBs in insulating oil is shown in Fig.2. 0.1 g of the oil sample is added to the purification column. The purification column is heated at 80°C for 30 min. After cooling, 20 ml of n-hexane is eluted. The solid partition column is separated from the purification column and is dried using nitrogen current gas. After drying, the solid partition column is eluted with approximately 200 μ l of DMSO. With the exception of monochlorobiphenyl (M_1CB) and decchlorobiphenyl ($D_{10}CB$), almost all PCBs are included in this eluted solvent.

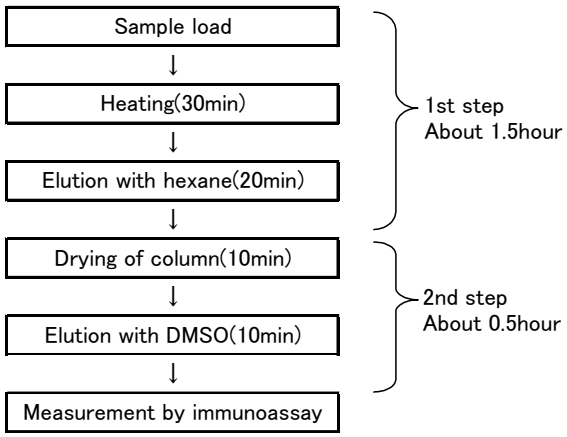

Fig.2 Procedure for rapid analysis

Results and Discussion

Purification ability of the rapid pretreatment columns

The purification ability are evaluated in comparison with intensity of fluorescence of DMSO of chemical reagent by immunoassay. The relative value 1.0 are equivalent to purification ability 100%. Relative fluorescence intensity of after pretreatment of Sample 1－Sample 14 are shown in Fig.3. The purification ability of Method 2 and Method 3 were very high.

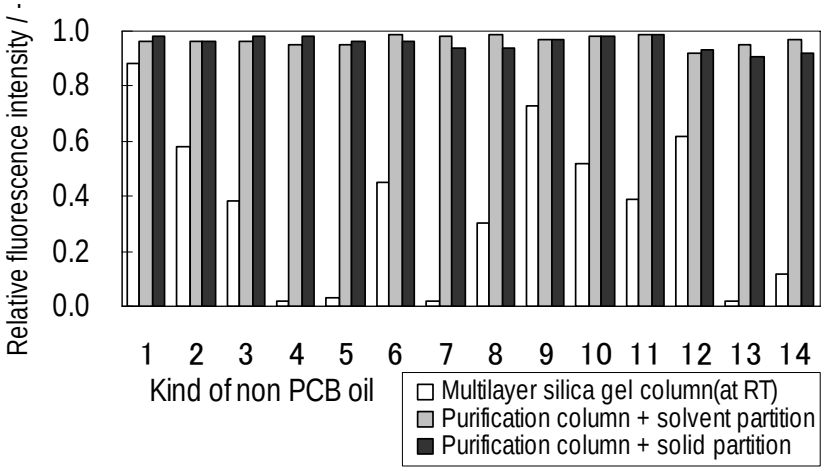

Fig.3 Purification ability of 14 oils

Dose-response curve

For Sample 1, KC - MIX was added to 0.040－10 mg/kg PCBs. This oil was pretreated by Method 2 and Method 3. PCB concentration was measured by immunoassay. From this result, dose response curve was made, as shown in Fig.4. As a result, Method 3 drew the dose-response curve that was similar to Method 2. In the rapid pretreatment method for immunoassay, satisfactory results were obtained with regard to quantitative capacity. The lower detection limit was approximately 0.10 mg/kg, and was less than the Japanese regulation value (0.5 mg/kg).

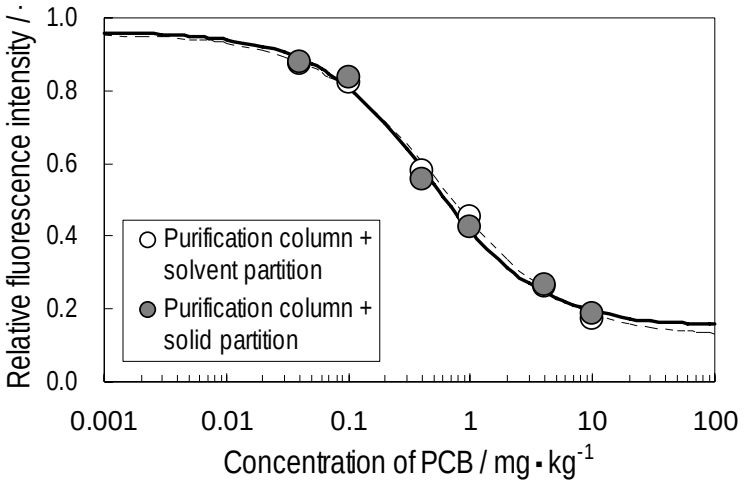

Fig.4 Dose-response curve

Recovery test of PCB oil

After Sample 1 was pretreated by “purification column only”, “purification column + solvent partition (Method 2)” and “purification column + solid partition (Method 3)”, KC - MIX was added to 0.50 m/kg PCBs in pretreated oils and PCB concentration was measured by immunoassay, as shown in Fig.5. The recovery rate was calculated from relative fluorescence intensity in dose-response curve at 0.50 mg/kg PCBs. In “purification column only”, recovery was low because oil contents had remained. In rapid pretreatment, satisfactory results were obtained with regard to recovery rate. These results imply that the rapid pretreatment method of PCB-containing oil in this study can be applied to immunoassay.

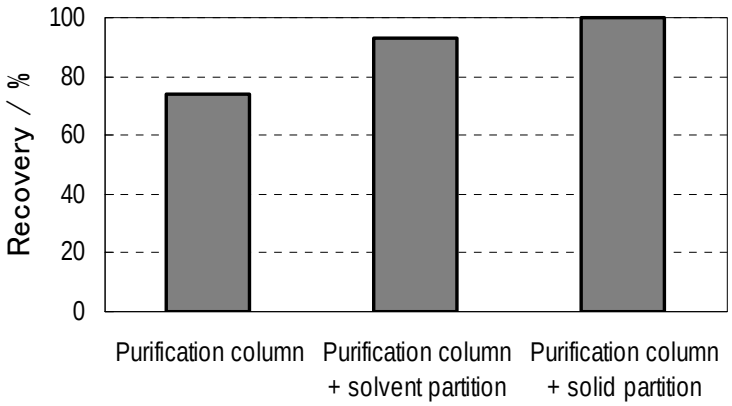

Fig.5 Result of recovery rate in Sample 1

References

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2. Fujita H., Hamada N., Sawadaishi K., Honda K. *Journal of Environmental Chemistry*, 15, 585–596 (2005)