



## Short Communication

# Comparison of solvent extraction and solid-phase extraction for the determination of polychlorinated biphenyls in transformer oil

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## ABSTRACT

Solid-phase extraction (SPE) of nine polychlorinated biphenyls (PCBs) from transformer oil samples was evaluated using octadecyl (C18)-bonded porous silica. The efficiency of SPE of these PCBs was compared with those obtained by solvent extraction with DMSO and hexane. Average recoveries exceeding 95% for these PCBs were obtained via the SPE method using small cartridges containing 100 mg of 40 pm C18-bonded porous silica. The average recovery by solvent extraction with DMSO and hexane exceeded 83%. It was concluded that the recoveries and precision for the solvent extraction of PCBs were poorer than those for the SPE.

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## 1. Introduction

One important area of modern environmental analysis is the determination of polychlorinated biphenyls (PCBs) and polycyclic aromatic hydrocarbons (PAHs), both of which commonly occur in the environment. They are highly toxic, mutagenic and persistent in the environment, and therefore are on the US Environmental Protection Agency (EPA) list of priority pollutants (Mahindrakar et al., 2012). Poly chlorinated biphenyls (PCBs) are chemical compounds which have been used as dielectric fluids in transformers and capacitors, as plasticizers and in hydraulic fluids (Berg et al., 1998; Bogdevich and Cadocinicov, 2004). PCBs are toxic, and they accumulate in soil, sediment and biota when they are released into the environment. Although most countries have strict regulations on the use of PCBs (UNEP, 2002), enormous amounts of PCBs did go into use before they were regulated. Indeed, they are still being used as insulators because devices such as transformers have lifetimes extending several decades. Many countries that have developed a classification system for PCB-containing fluids and materials have considered 50 mg kg<sup>-1</sup> as the benchmark level for PCB regulation (UNEP, 1999). In accordance with PCB management, analytical test methods for determination of PCBs in insulating oil have been published as ASTM-D4059, EPA-600/4-81-045 and NIST

(USEPA, 1982; ASTM D4059, 2000). In these methods, the oil, diluted with the appropriate solvent, is treated with acid and/or an adsorbent to remove interferences, and then analyzed by GC-ECD. However, the method detection limits (MDLs) of PCBs by these test methods are only 1–2 mg kg<sup>-1</sup> because the oil is diluted 50–100 times to minimize the influence of the oil during the analytical procedure (Yun-Cheol et al., 2008). PCBs are not easily separated from oil because the physical and chemical characteristics of PCBs are very similar to those of mineral oil. The components of oil-based liquid wastes co-elute with the PCBs during a GC separation and the baseline shifts due to the oil matrix. The key to lowering the MDL is to effectively remove the remaining oil during the analytical procedure. To remove the oil, a few analytical methods have been introduced to apply liquid–liquid partition using an appropriate solvent. Although these methods are useful in identifying and determining the PCBs in oil, the separation of PCBs by liquid–liquid extraction (LLE) is dependent on the skill of the analysts. To separate PCBs from oil matrix, researchers have used partition between non-polar solvent and non-proton polar solvent, such as dimethylsulfoxide (Orazio et al., 1989; Larsen et al., 1991; Mahindrakar et al., 2011a,b,c), acetonitrile (Gordon et al., 1987) and dimethylformamide (Lawn and Toffel, 1987). Solid phase extraction (SPE) system is also reported for concentration of sample (Kot-Wasik et al., 2004; Wolska et al., 2005; Conka et al., 2005). This method provides a powerful technique to overcome the drawback of LLE. A comparative study of the SPE technique

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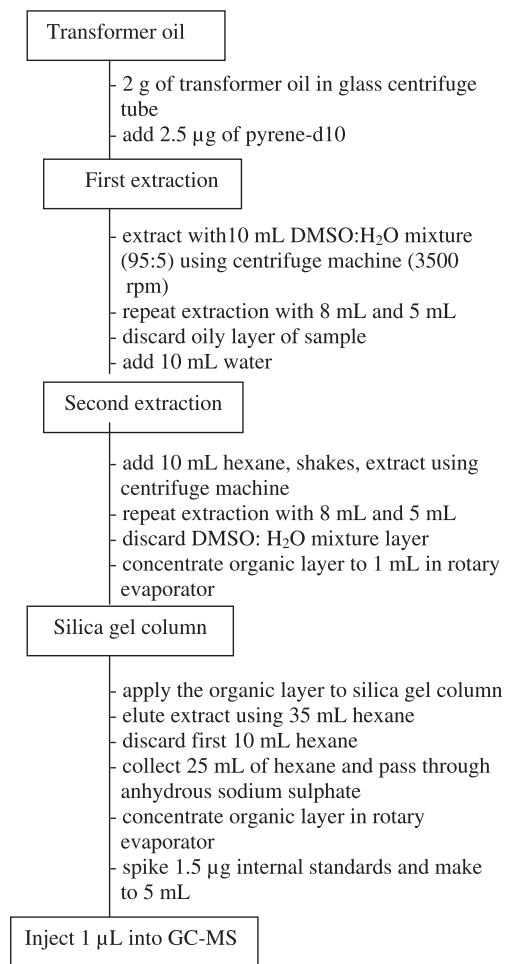


Fig. 1. Analytical procedure for determination of PCBs in transformer oil.

using commercially available cartridges and the conventional solvent extraction method was carried out for nine PCBs in transformer oils.

## 2. Materials and methods

### 2.1. Reagents

The solvent hexane was HPLC grade, DMSO and sulfuric acid (98%) was A. R. grade used in the present work. Silica gel (100–200 mesh, Sigma–Aldrich) activated at 200 °C for 6 h and anhydrous sodium sulfate (A.R. grade) was used in the clean-up stage. Water was purified with a Milli-Q plus system (Millipore). Supelclean LC-18 SPE cartridges from Supelco, pre-packed in a polypropylene housing cartridge containing 100 mg of C18-bonded porous silica and with a 0.45 µm filter attached, were used.

### 2.2. Standard solutions

The mixture of PCBs was purchased from AccuStandard (M-680A). Phenanthrene-d10 and chrysene-d12 from AccuStandard (M-680-IS-10X) and pyrene-d10 from Sigma Aldrich were used as an internal standards and surrogate standard respectively. All the solutions were prepared in hexane. Solutions of 0.0156–12.50 mg kg<sup>-1</sup> were prepared from the stock solution for calibration of different congeners and 0.3 mg kg<sup>-1</sup> of internal standard was added to each.

### 2.3. Samples

The samples of transformer oil were stored in 250 mL pre-cleaned dark glass bottles with Teflon caps at a maximum temperature of 4 °C. Moisture determinations were made according to ASTM D 3976 (Mahindrakar et al., 2011a,b,c). The concentration of PCBs was calculated on dry weight basis.

### 2.4. Analytical procedure for Solvent extraction

The analytical procedure was used from the previous work (Mahindrakar et al., 2011a,b,c), the sample was transferred into a glass centrifuge tube and 1 mL of 2.5 µg of surrogate standard pyrene-d10 was added. The first extraction was carried out by 10 mL DMSO and water (95:5) then repeated the same with 8 mL and finally 5 mL after keeping the tubes into centrifuge machine (3500 rpm) for 10 min. Then 10 mL water was added in extraction portion. This was further extracted with 10 mL hexane, repeated the same with 8 mL and finally with 5 mL after keeping in centrifuge machine for 10 min. The extracted portion was reduced in rotary evaporator and made up in 5 mL after adding 1.5 µg of internal standards and finally injected 1 µL into GC–MS. The detailed analytical procedure is shown in Fig. 1.

### 2.5. Sample cleanup

A variety of substances in oil can be simultaneously extracted and cause interference in the determination of PCBs by GC–MS. Therefore the extraction was purified using silica gel column. The clean-up procedure was as follows: The concentrated extracts were applied to open glass column (0.45 m, 1 cm i.d.) filled with (from bottom to top) glass wool, silica gel (100–200 mesh) and anhydrous sodium sulfate (2 cm). The extracts were eluted with the mixture of hexane and acetone (1:1) from silica column, the first 10 mL of elute were discarded and remaining 25 mL of elute was collected and reduced the volume in rotary evaporator and

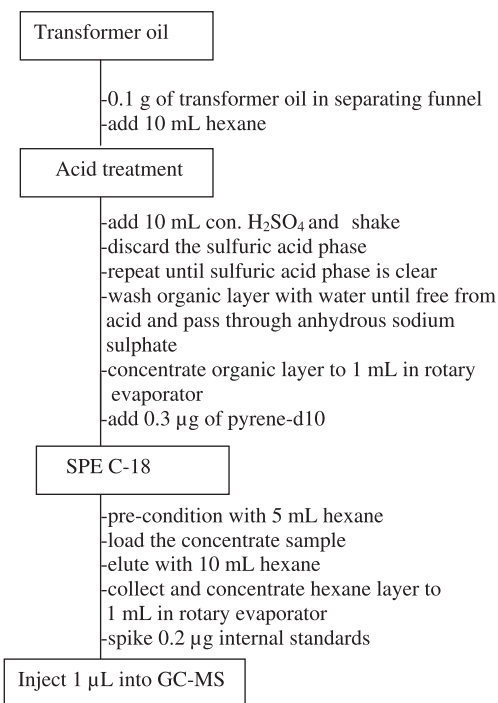


Fig. 2. Analytical procedure for determination of PCBs in transformer oils.

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