Development of Simple Analytical Method for Trace Chemical Substances such as PCB Using High Temperature Thermal Degradation

Background

In 2002, The Japan Electrical Manufacturers' Association (JEMA) announced that trace PCB was contaminated in the insulating oil, in which it had been considered that PCB was not contained. Insulating oil containing PCB is required to be processing for non-toxicity in Japan. Therefore, the organizations such as the electric power industries, which use and keep insulating oil, are driven by necessity to check that PCB is contained or not in the oil that they are possessing. It is considered that the insulating oil samples which are needed to be analyzed are up to a few hundred thousands, so the analytical method is required to be simple and rapid. On the contrary, measurement demand of trace heavy metals in coal and heavy oil are increased according to the surge of concern to the recent environmental problems. However, the measurement procedure is usually complex and must take much time. Accordingly, the needs for a simple and rapid analytical method for trace heavy metals are high.

Objectives

To develop a simple and rapid analytical method for the chemical substances in insulating oil, and to evaluate the performances of the developed method.

Principal Results

1. Planning and manufacture of the high temperature thermal degradation system

The author planned and manufactured the high temperature thermal degradation system to analyze PCB in insulating oil and heavy metals in fuels simply and rapidly. (Fig. 1)[1] In this system, the sample is thermally degraded immediately in closed and oxygen pressurized container, and then the target chemicals are recovered by trapping the combustion gas in absorbent solution. Measuring time for PCB in insulating oil with this system is 20-30 minutes, being much shorter than the usual analytical method (-3 hours).

2. Simple analysis of PCB in insulating oil by the high temperature thermal degradation system

PCB standard samples, in which PCB concentrations were known, were applied to the system, resulting that almost all PCB in samples were recovered as hydrogen chloride (Fig. 2). In addition it was confirmed that 0.5ppm of PCB, which is processing standard value of PCB waste in Japan, could be analyzed using this system. Furthermore, several old insulating oil samples, in which PCB concentrations were known, were analyzed using this system, resulting that PCB were measured almost quantitatively (Table 1). Slight overestimation of chloride is considered to result from trichlorobenzene in insulating oil.

3. Simple analysis of trace heavy metals in fuels by the high temperature thermal degradation system

Heavy oil and coal samples, in which heavy metal concentrations were known, were applied to the system. As a result, As and Se in heavy oil were almost recovered, whereas As and Se in coal were measured only 50-60 % of original samples. Required time to measure heavy metals in fuels with this system is 2 days, being much shorter than the usual analytical method (-10 days).

Accordingly, the high temperature thermal degradation system established in this study was evaluated to have potency as simple and rapid analytical method for PCB in insulating oil and heavy metals in fuels.

Future Developments

Examining the new thermal degradation system for coal samples, and trying instrumentation of this system.

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Reference

N. Tanaka, 2004, "Development of simple analytical method for trace chemical substances such as PCB using high temperature thermal degradation", CRIEPI Report, T03034.
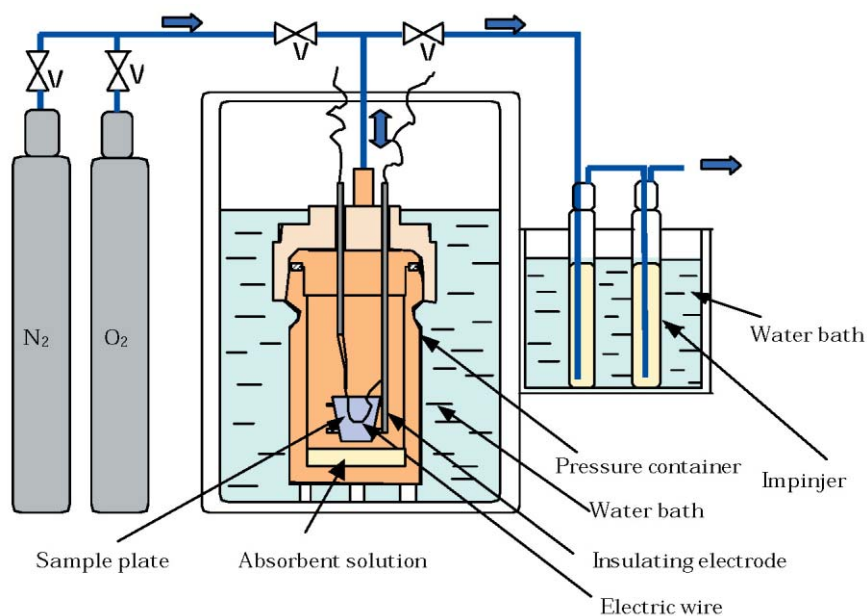
Fig.1 An overview of thermal degration system examined in this study.

The system is divided into gas supply part, thermal degradation part and sample collection part. The procedureis following: Sample is put on a sample plate. After a pressure container is closed, O₂ is filled in the container to30MPa. The sample is thermally degraded by high voltage using insulating electrode. After degradation,sample gas is collected in absorbent solution.
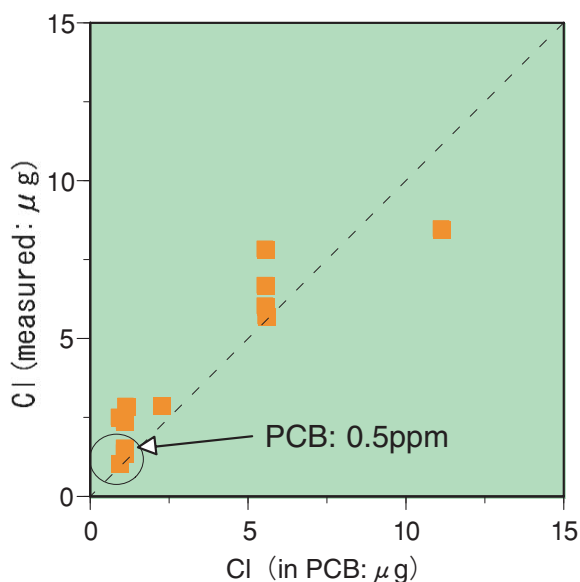

Fig.2 Relationship between Cl in PCB and Cl measured by thermal degradation.

Almost 100% of Cl in analyzed PCB samples were degraded to hydrogen chloride by thermal degradation and were recovered.

Table 1 Analytical result of Cl in used insulating oil.

	Cl (μg)	
	Input	Measured
oil 1	15.5	20.0
oil 2	15.7	18.7
oil 3	16.3	20.7
blank[1]	0	0.20

[1]Insulating oil without PCB.