
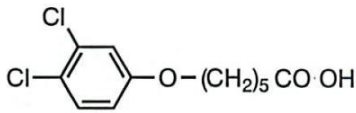


(※本報告書は英語で記述してください。ただし、産業利用課題として採択されている方は日本語で記述していただいても結構です。)

 MLF Experimental Report	提出日 Date of Report 15/02/2017
課題番号 Project No. 2016B0108 実験課題名 Title of experiment Non-destructive detection of organochlorine compound in insulating oil using neutron induced prompt gamma ray analysis (PGA). 実験責任者名 Name of principal investigator Yasumoto Date 所属 Affiliation Central Research Institute of Electric Power Industry	装置責任者 Name of responsible person Yosuke Toh 装置名 Name of Instrument/(BL No.) Accurate Neutron-Nucleus Reaction Measurement Instrument (BL-04) 実施日 Date of Experiment 21-24/11/2016

試料、実験方法、利用の結果得られた主なデータ、考察、結論等を、記述して下さい。(適宜、図表添付のこと)
 Please report your samples, experimental method and results, discussion and conclusions. Please add figures and tables for better explanation.

1. 試料 Name of sample(s) and chemical formula, or compositions including physical form.
<p>Measured samples.</p> <p>Sample name (chemical formula, physical form)</p> <p>(1) Oil samples Insulating oil (CnHm, containing 0, 5, 44, 437, 876 mg-chlorine*/kg, <i>solid</i>)</p> <p>(2) Interference samples Borosilicate glass (SiO₂, <i>solid</i>) Stainless steel (Fe, <i>solid</i>) Copper (Cu, <i>solid</i>)</p> <p>(3) Composite sample Composite sample (Borosilicate glass, Copper, Mineral oil containing 876 mg-chlorine*/kg, <i>solid</i>)</p> <p>* Trace chlorine in oil derive from compound A shown in following chemical structure (fig.1)</p> <div style="text-align: center;">  </div> <p>Fig1. "Compound-A" 6-(3,4-dichlorophenoxy)hexanoic acid (Mw: 277 g/mol)</p>

2. 実験方法及び結果（実験がうまくいかなかった場合、その理由を記述してください。）

Experimental method and results. If you failed to conduct experiment as planned, please describe reasons.

Methods.

The experiment has been carried out using ANNRI installed at beam line BL-04. We measured the trace chlorine (0 – 876 mg-Cl/kg-oil) in five mineral oil samples. Three interference materials and one composite sample composed of Cu, borosilicate, and mineral oil were measured. All samples were enclosed in doubled FEP sheet, and were set on the sample holder. The relationship between the chlorine content and peak intensities obtained by analyzing these spectra was studied. The energy-integrated neutron intensities under a proton beam power of 150 kW at the sample position are approximately 0.5×10^{11} n/s/m² in the neutron energy range of 1.5–25 meV, and 0.7×10^{10} n/s/m² in 0.9–1.1 keV. The thermal neutron capture cross section for Cl-35 is approximately 43.6 barns.

Initially, prior examination was carried out (shot: 42100, 84200, and 168400, with Pb filter. Eventually, Pb filter was removed and the number of pulse was decides as 163725 shot.

Result.

The results of mineral oil measurement (0, 437 and 876 mg-Cl/kg-oil), and detection limit of chlorine in mineral oil were shown in Table 1. The detection limit was 166–191, and weight average was 182 mg-Cl/kg-oil. Chlorine content in polychlorinated biphenyls (PCBs, KC-500) is 54 wt%. Therefore, the calculated detection limit of PCBs was 337 mg-PCBs/kg-oil. In addition, the chlorine measurement in composite sample was interfered by copper.

Environmental standard of PCBs in oil is 0.5 mg-PCB/kg-oil, and the high-level contaminated waste standard is 5000 mg-PCB/kg. From the result of present study, PGA based PCBs detection using chlorine as the evaluation index seems applicable not for the decision of environmental standard, but for the decision of high-level contamination.

In the next step, we will test the following subjects.

1. The effect of the sample weight on the detection limit of chlorine.
2. Interference of borosilicate, and other materials which exist in measurement situation.
3. Actual PCBs measurement (containing 209 of congeners).

Table 1

measurement	Sample weight (g)	Chlorine (mg-Cl/kg-oil)	count/shot	error	*Detection limit (mg-Cl/kg)
#4083	0.05	0	0.002281225	0.00237727	
#4087	0.05	437	0.013304022	0.00220419	166
#4085	0.05	876	0.023149725	0.00235073	191

*The detection limits were calculated from three sigma method.

Table 2

Average detection limit (mg-Cl/kg-oil)	*Calculated PCB detection limit (mg-PCBs/kg-oil)
182	337

*Chlorine content in PCBs (KC500) is 54 wt%.