

INTER-LABORATORY CROSS-CHECK ON THE ANALYSIS OF PCBs IN INSULATING OIL.

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Introduction:

Polychlorinated biphenyl (PCBs) has excellent chemical stability and electrical insulation, and had been used as insulating oil, heat transfer medium, and carbonless copying paper, etc. However, it has strong toxicity, and the use and production are prohibited at present. In Japan, PCBs contaminated wastes must be disposed before April 2027 according to "Law concerning Special Measures for Promotion of Proper Treatment of PCB Wastes". Accordingly, in 2010, the Ministry of Environment established "Measurement methods for evaluating very small amounts of PCB contained in insulating oil" (hereinafter referred to as "MOE methods") to promote the disposal of PCBs waste. In this method, in order to ensure analytical accuracy in analytical laboratories, it is required to strive for technological improvement through external precision management. As part of the external accuracy management required by the MOE methods, we have carried out inter-laboratory cross-check since 2010. In this paper, we report on the results of nine inter-laboratory cross-checks conducted so far.

Materials and Methods:

All inter-laboratory cross-checks were carried out by distributing 4 samples with different PCBs composition, concentration and insulating oil species. The samples were prepared by mixing a virgin insulating oil with a PCBs product (KC-300, KC-500 or mixed). In order to confirm the progress of the proficiency, a sample having a PCBs content of 0.5 mg/kg (which is a PCBs treatment regulations in Japan) was prepared at all rounds. The participants analyzed the samples distributed according to the MOE methods and reported results to us. The results were statistically analyzed, and the z-scores of the values were evaluated using a robust statistical method. The results of the evaluation were fed back to the participants.

Results and Discussions:

The total 9 inter-laboratory cross-checks were reported from a cumulative total of 849 laboratories and 922 reported values were obtained. The statistics for each round in the same composition and concentration samples are shown in Table 1. The CV(%)rob which was 15.2% in round 1 converged to about 8%, and it is considered that the analysis accuracy is improved and maintained. This conclusion became evident from the results of laboratories which participate a lot of times. Therefore, it was confirmed that the technical verification by the continuous participation is important.

Table 1. The statistics for each round in the same composition and concentration samples

Type of insulating oil			Mineral oil (JIS C 2320 Class1)						
PCBs concentration			0.5 mg/kg						
PCBs composition			KC-500						
Round	1	2	3	4	5	6	7	8	9
Year	2010	2011	2012	2013	2014	2015	2016	2017	2018
Number of values	113	94	90	100	99	102	95	99	95
Min (mg/kg)	0.151	0.215	0.216	0.130	0.099	<0.150	0.223	<0.130	<0.150
Max (mg/kg)	1.88	1.15	1.56	0.791	4.20	0.632	4.49	47.0	0.933
Mean (mg/kg)	0.481	0.489	0.489	0.490	0.559	0.506	0.568	0.981	0.475
Median (mg/kg)	0.477	0.480	0.482	0.500	0.504	0.513	0.521	0.523	0.492
SD	0.171	0.114	0.140	0.076	0.439	0.068	0.415	4.67	0.083
NIQR	0.073	0.066	0.046	0.041	0.038	0.036	0.042	0.043	0.042
CV(%)	35.5%	23.2%	28.6%	15.6%	78.6%	13.4%	73.0%	476%	17.6%
CV(%)rob	15.2%	13.8%	9.6%	8.2%	7.6%	7.0%	8.1%	8.2%	8.6%
$2 < z < 3$	9.7%	6.4%	13.3%	10.0%	10.1%	6.9%	5.3%	12.1%	5.3%
$3 \leq z $	7.1%	5.3%	10.0%	6.0%	14.1%	3.9%	7.4%	11.1%	8.4%