

Electrochemical Method for Surface Testing of PCB Contaminated Electrical Equipment

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Traditionally, surface wipe testing of electrical equipment for PCB contamination has been performed by gas chromatography (GC) and reported in units of ug PCB per 100 cm⁴. The actual sampling procedure can be quite varied but generally involves a piece of cotton gauze or filter paper that has been soaked with hexane. This material is repeatedly wiped over a specific area (usually 100 cm⁴) of the equipment in question, often using a template to simplify area measurement. The gauze or filter paper is then sent to the laboratory where it is extracted and analyzed using a procedure very similar to that for an oil sample. The efficiency of surface testing varies greatly with technique and also depends on the smoothness and porosity of the surface being wiped. Although it is often used on porous materials such as concrete or wood, this method does not generally give an accurate indication as to the concentration in the substrate.

Recently, a field method for wipe testing has been developed that uses an electrochemical technique for quantification of the PCBS. The wipe sampling procedure is the same as for the GC methods but the analytical test uses a sodium reaction to remove chlorine from the PCBs which are then quantified using a chloride specific electrode instead of a gas chromatograph. The instrument to perform this test, called the L2000 PCB/Chloride Analyzer, has been used for several years to test for the presence of PCB in transformer oil and in soil samples. The advantage to this method of surface testing over traditional techniques is that the analysis may be performed on site in about 5 minutes without sending a sample to the laboratory.

To compare the accuracy and precision of the method with traditional GC techniques an experiment was designed that would examine as closely as possible the results obtained on identical samples by each method. To analyze the relative accuracy of the two methods, as many outside sources of error as possible were eliminated. Instead of contaminating actual surfaces and performing the wipe test procedure on them, the gauze wipes themselves were directly contaminated with PCBs and then compared using the two techniques. Each 3 by 8 cm piece of gauze was spiked with 2 ml of hexane containing 5% transformer oil. The transformer oil was added to simulate the oil that would be picked up off most surfaces when performing a wipe test. Each piece of gauze was spiked to an amount equivalent to the amount of PCB that would have been removed had an area of 1000 cm² been tested. 30 different samples were spiked; five groups of six samples each at the following concentrations of Aroclor 1260:

<u>Equivalent Surface Concentration</u>	<u>PCB Conc in 2 ml Hexane Spike</u>
0 ug/100 cm ²	0 ug/ml
2 ug/100 cm ²	10 ug/ml
10 ug/100 cm ²	50 ug/ml
50 ug/100 cm ²	250 ug/ml
100 ug/100 cm ²	500 ug/ml

Three-blind samples at each concentration were analyzed by each method so the operators were unaware of the correct concentration for each. The actual procedure used with each method was as follows.

Gas Chromatography

Each piece of contaminated gauze was extracted using 5 ml of chromatographic grade isooctane. The resulting isooctane extract was then washed with concentrated sulfuric acid and injected onto a Varian 3400 GC equipped with electron capture detector. Each sample was evaluated according to EPA method 608 and results were quantified using the practice of Webb and McCall.¹ Results were reported on a ug/100 cm² basis under the previously made assumption that each area wiped totaled 1000 cm². If samples were found to be outside the quantitative range of the instrument they were diluted and re-analyzed until they fell into the appropriate range. All results that showed detectable quantities were identified as Aroclor 1260.

L2000 Electrochemical Technique

Each piece of gauze was placed in a 20 ml vial and ten ml of chromatographic grade isooctane was added. The entire vial was well shaken for one minute. Using a polypropylene pipette, five milliliters of isooctane were removed from each vial and placed into the reaction tube for the L2000 procedure. The sample was processed identically to a transformer oil sample, meaning that the sample was first reacted with a sodium reagent for one minute and then the resulting chloride was extracted using an aqueous based extraction solution.⁴ The extraction solution was then separated from the isooctane and analyzed using the chloride specific electrode on the L2000. The instrument was run using the analytical mode for Aroclor 1260. All results were read directly off the LCD readout and were recorded as ug/100 cm².

Comparison of results

The following results were found for each of the methods

Table 2

Actual Sample Concentration ug/100 cm ²	GC Result Mean of 3 ug/100 cm ²	GC Result Std. Dev. ug/100 cm ²	L2000 Result Mean of 3 ug/100 cm ²	L2000 Result Std. Dev. ug/100 cm ²
0	ND(<2)	NA	ND(<2)	NA
2	1.77	.07	2.26	.45
10	8.11	.13	11.35	.49
50	39.4	1.66	56.1	2.8
100	78.6	3.2	112.8	4.8

CONCLUSION

The results show that in this particular experiment both methods provide consistent results while the GC results are biased somewhat low and the L2000 numbers are biased slightly high. Standard deviations were found to be within the stated limits for each of the methods. The lower standard deviation for the GC method at the 2 ug/100cm² level reflects the lower limit of detection obtainable with the GC. Some of the variation seen in the results may be due to the way the samples were prepared, but most is probably inherent in each analytical technique. Actual wipe test samples would probably show greater variation due to the problems inherent in wipe test sampling.

This study demonstrates that the L2000 is capable of generating surface test results with accuracy and precision comparable to those found using full laboratory GC procedures. With this level of confidence in the results from the L2000 PCB/Chloride Analyzer, it may be the preferred method of analysis when time and cost are primary considerations. Because more error is introduced by actual sampling technique rather than by the chosen analytical method, it is important to follow established guidelines on sampling procedure. Users should make sure to check with regulatory authorities as to how to set up a statistically valid sampling plan.

REFERENCES

¹Webb, R.G. and A.C. McCall, Journal of Chromatographic Science, 11, 366 (1973).

²L2000 PCB/Chloride Analyzer Instruction Manual. Dexsil Corporation, 1990.