# Protocol for Sampling and Testing at PCB Storage Sites in Ontario

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Ontario

**Ministry of the Environment**

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## EXECUTIVE SUMMARY

Legislation for PCB Storage Sites in Ontario is administered under Ontario Regulations 362 (PCB waste management) and 347 (General Waste Management). Historically, wastes stored at these sites have been broadly classified as PCB wastes. In an effort to better manage these sites and in some cases to decommission and close these sites, there is a need for re-evaluation of the wastes, proper characterization (with representative sampling and testing) and permanent disposal of PCB wastes and non-hazardous wastes.

The objective of this document is to provide standard procedures for sampling and testing (laboratory analysis) of contaminated materials stored at these PCB Storage Sites. The protocol can also be used for the decommissioning of these sites or for the clean up of other PCB contaminated sites.

Alternative procedures for sampling can be used for unusual situations, provided that the alternatives have been presented in writing to the Ontario Ministry of the Environment (MOE), and the document reviewed and accepted by MOE. Reference analytical methods are suggested but not required for the analysis or testing of the samples.

Wastes found at these sites are usually stored either in drums or accumulated in piles or in large containers. The protocol provides detailed procedures for sampling and testing for both methods of storage. Sampling and testing of PCB contaminated materials in piles requires segregation of the various types of materials, representative sampling of the segregated piles and statistical analysis of the results.

Various types of materials are stored in these drums or piles. Detailed procedures for sampling are identified for various type of materials, including soil, rocks, concrete/asphalt, wood, electrical cables, liquid/sludge materials, light ballasts and capacitors. This protocol does not include however standards for the classification and management of PCB contaminated transformers, which are covered under the “PCB Transformer Decontamination Standards and Protocols” (CCME, 1995).

Site decommissioning also requires proper sampling and testing of cleaned surfaces inside buildings and cleaned land on the property. Methods for sampling at decommissioned sites are presented in the last chapter of this protocol.

## TABLE OF CONTENTS

## 1 -- INTRODUCTION

This protocol applies to re-classification of wastes stored at registered PCB storage sites located in areas under the jurisdiction of the Province of Ontario and the Ministry of the Environment (MOE). The re-classification of wastes stored at PCB sites can be justified in some cases because of a broad waste characterization initially done at these sites. Re-classification is based on thorough sampling and testing of segregated materials contaminated at different levels and having different physical characteristics. This protocol may be used also for sampling and testing of sites contaminated with PCB, keeping in mind that the protocol does not override any requirements from other guidelines for contaminated sites, or standards and protocols for decontamination of PCB transformers.

This protocol does not apply to PCB transformers. Whenever PCB transformers are stored at PCB sites, the CCME (Canadian Council of Ministers of the Environment) protocols are applicable in Ontario, in accordance with Director's Instruction and Certificate of Approval. The following document should be consulted for details on the classification and management of PCB transformers: "PCB Transformer Decontamination Standards and Protocols", CCME EPC-HW-105E, December 1995.

## 2 -- SAMPLING

### 2.1 RATIONALE FOR SAMPLING

The objective of the sampling methods and procedures outlined in this protocol is to collect a PCB waste sample that represents the concentration of PCB in the waste. The waste may be stored in drums, containers of various sizes, in piles or simply in the field. (Information on representative sampling for soil can be found in Section 5.1 of the "Guidance on sampling and analytical methods for use at contaminated sites in Ontario", MOE, July 1996, PIBS 3266E01).

The procedures for collecting a representative sample are very site specific. The procedures developed in this protocol focus on the majority of the situations that may be encountered. Other procedures may be justified for collecting a representative sample, as long as the rationale is explained by the proponent and the approach is justified in a written submission for review and acceptance by MOE (District Offices in consultation with Approvals Branch and Standards Development Branch).

There is always the no-sampling alternative where, after visual inspection of the container or the pile, the proponent takes the decision not to segregate nor to sample the waste. For practical reasons the proponent assumes that the waste is a PCB waste. In this case, the waste must remain at the site or be managed as PCB waste, under Regulation 347 and Regulation 362.

## 2.2 -- PCB MATERIALS STORED IN DRUMS

### 2.2.1 PCB contaminated soil (in drums)

Soil is defined, for the purpose of this protocol, as follows:

> *"loose or unconsolidated material resulting from the breakdown of rock or organic matter by natural physical, chemical and biological processes and which is capable of supporting plant growth. More than 50% of the material by volume must have a particle size of less than 2 mm"*
>
> *(Guidance on sampling and analytical methods for use at contaminated sites in Ontario, MOE, July 1996, PIBS 3266E01)*

Representative samples from contaminated soils stored in drums (45-gallon type) are collected and analysed as follows:

a) If there is any residual liquid or sludge at the bottom of the drum, the aliquot taken at the bottom layer should include a representative sample of the liquid or sludge. For drums with more than a liquid or sludge residual (i.e. more than 2.5 cm deep at the bottom), the protocol for PCB mixed materials (section 2.7) must be used.

b) If the content of the drum is made of mixed materials (less than 90% of the drum content is soil), the protocol for PCB mixed materials (section 2.7) must be used.

c) Three (3) aliquots (samples forming a composite sample) are taken from 3 representative depths of the soil in the drum (top, middle and bottom depths of the soil).

d) Sampling procedure:

- A core sampler is used for collecting the aliquots at different depths, representing the top, middle and bottom layers of the drum content.

- As an alternative to the core sampling procedure, the content of the drum can be transferred to a self contained flat surface where aliquots are collected at locations representing the top, middle and bottom layers of the content of the drum. The content is returned to the drum for later classification and proper management. Proper care must be taken to avoid any spill and/or equipment contamination during the manipulation of the drum and its content.

e) The 3 aliquots are well mixed together to form one composite sample for analysis.

f) A portion of the composite sample is analysed for total PCB concentration for classification of the waste as PCB waste (under Ont. Reg. 362, total concentration above 50 mg/kg). Waste with PCB concentration above 50 mg/kg must therefore be managed as PCB waste. For waste with results less than 6 mg/kg total PCB, the waste is classified as non PCB waste, and non leachate toxic waste (due to dilution in the leachate analysis). There is no need to conduct a leachate analysis on this sample (by-passing procedure 1.8).

g) Another portion of the same composite sample is analysed for PCB leachate concentration, using the LEP method (Leachate Extraction Procedure, Ont. Reg. 347). LEP results above 0.3 mg/L indicate that the waste represented by the sample is classified as hazardous waste (leachate toxic). The waste must therefore be managed as hazardous waste under requirements of Reg. 347.

h) For unusual situations, other representative sampling procedures can be proposed by a proponent, provided that the proposal is well justified and documented. The proposal should be presented in a formal document to MOE District Office for review and authorization (in consultation withe the Approvals Branch and/or the Standards Development Branch), before conducting the waste characterization program.

i) An alternative analytical approach can also be used for reducing the number of leachate tests, as indicated in section “Rationale for testing”, at the beginning of this document.

### 2.2.2 PCB contaminated rocks (in drums)

Rocks are defined, for the purpose of this protocol, as follows:

> *“aggregations of particles composed of one or more naturally occurring minerals for which, in total, more than 50% by volume has a particle size of greater than 2 mm”*
> *(Guidance on sampling and analytical methods for use at contaminated sites in Ontario, MOE, July 1996, PIBS 3266E01)*

This definition does not include rock-like material, such as concrete or asphalt materials (concrete and asphalt sampling procedures are defined in the following section).

a) If there is any residual liquid or sludge at the bottom of the drum, the aliquot taken at the bottom layer should include a representative sample of the liquid or sludge. For drums with a liquid or sludge residual of more than 2.5 cm deep at the bottom, the protocol for PCB mixed materials (section 2.7) must be used.

b) If the content of the drum is made of mixed materials (less than 90% of the drum content is rock), the protocol for PCB mixed materials (section 2.7) must be used.

c) Three (3) aliquots (samples forming a composite sample) are taken from 3 representative depths of the rocks in the drum (top, middle and bottom depths of the rocks).

d) Sampling procedure:

- the aliquots are collected from randomly selected rocks at the 3 depths, by taking a few surface chips from these rocks to make an appropriate volume of the composite sample;

- to access the 3 layers of rocks in the drum, the content of the drum can be transferred to a self contained flat surface where aliquots are collected at locations representing the 3 layers of the content of the drum. The content is returned to the drum for later classification and proper management. Proper care must be taken to avoid any spill and/or equipment contamination during the manipulation of the drum and its content.

e) The surface chips from the surfaces of each of the 3 rocks should be of approximately of equal volume. The 3 aliquots are well mixed together to form one composite sample for analysis.

f) A portion of the composite sample is analysed for total PCB concentration, using a PCB analysis method approved or acceptable by MOE for classification of the waste as PCB waste (under Reg. 362, total concentration above 50 mg/kg). Waste with PCB concentration above 50 mg/kg must therefore be managed as PCB waste. Waste with results less than 6 mg/kg total PCB may also be classified as non leachate toxic waste (for PCB), because it cannot leach more than 50 mg/L due to a dilution by a factor of 20 during the leachate test. Therefore there is no need to conduct a leachate analysis on this sample (bypassing procedure 2.7).

Another portion of the same composite sample is analysed for PCB leachate concentration, using the LEP method (Leachate Extraction Procedure, Reg. 347). LEP results above 0.3 mg/L indicate that the waste represented by the sample is classified as hazardous waste (leachate toxic). The waste must therefore be managed as hazardous waste under requirements of Reg. 347.

g) For unusual situations, other representative sampling procedures can be proposed by a proponent, provided that the proposal is well justified and documented. The proposal should be presented in a formal document to MOE District Office for review and authorization (in consultation withe the Approvals Branch and/or the Standards Development Branch), before any initial sampling, analysis or work are conducted.

### 2.2.3 PCB contaminated concrete and asphalt (in drums)

Concrete and asphalt materials are generally more porous than regular rocks found naturally in the environment. The level and distribution of PCB contamination, where concrete slabs and asphalt surface may have been contaminated locally by spills and industrial/commercial use, is probably different than for rock,.

a) If there is any residual liquid or sludge at the bottom of the drum, the aliquot taken at the bottom layer should include a representative sample of the liquid or sludge. For drums with more than a liquid or sludge residual (i.e. more than 2.5 cm deep at the bottom), the protocol for PCB mixed materials (section 2.7) must be used.

b) If the content of the drum is made of mixed materials (less than 90% of the drum content is concrete/asphalt), the protocol for PCB mixed materials (section 2.7) must be used.

c) Five (5) aliquots (samples forming a composite sample) are taken from 5 representative layers of the concrete/asphalt in the drum .

d) Sampling procedure:

- the aliquots are collected from randomly selected large pieces at the 5 layers, by taking a few surface chips (approx. 2.5 cm deep) from these pieces to make an appropriate volume of the composite sample

- to access the 5 layers of concrete/asphalt in the drum, the content of the drum can be transferred to a self contained flat surface where aliquots are collected at locations representing the 5 layers of the content of the drum. The content is returned to the drum for later classification and proper management. Proper care must be taken to avoid any spill and/or equipment contamination during the manipulation of the drum and its content.

e) The 5 aliquots are well mixed together to form one composite sample for analysis.

f) A portion of the composite sample is analysed for total PCB concentration, using a PCB analysis method approved ( or acceptable by MOE for classification of the waste as PCB waste (under Reg. 362, total concentration above 50 mg/kg). Waste with PCB concentration above 50 mg/kg must therefore be managed as PCB waste. For waste with results less than 6 mg/kg total PCB, the waste is classified as non PCB waste, and non leachate toxic waste (due to dilution in the leachate analysis). There is no need to conduct a leachate analysis on this sample (bypassing procedure 3.7).

g) Another portion of the same composite sample is analysed for PCB leachate concentration, using the LEP method (Leachate Extraction Procedure, Reg. 347). LEP results above 0.3 mg/L indicate that the waste represented by the sample is classified as hazardous waste (leachate toxic). The waste must therefore be managed as hazardous waste under requirements of Reg. 347.

h) For unusual situations, other representative sampling procedures can be proposed by a proponent, provided that the proposal is well justified and documented. The proposal should be presented in a formal document to MOE District Office for review and authorization (in consultation withe the Approvals Branch and/or the Standards Development Branch), before any initial sampling, analysis or work are conducted.

i) An alternative analytical approach can also be used for reducing the number of leachate tests, as indicated in section “Rationale for testing”, at the beginning of this document.

### 2.2.4 PCB contaminated wood (in drums)

Wood waste contaminated with PCB usually comes from PCB contaminated demolition materials, spills or from wood preserving oil contamination. The form and size of PCB contaminated wood is either of the type of wood demolition materials or railway ties.

a) If there is any residual liquid or sludge at the bottom of the drum, the aliquot taken at the bottom layer should include a representative sample of the liquid or sludge. For drums with more than a liquid or sludge residual (i.e. more than 2.5 cm deep at the bottom), the protocol for PCB mixed materials (section 2.7) must be used.

b) If the content of the drum is made of mixed materials (less than 90% of the drum content is wood waste), the protocol for PCB mixed materials (section 2.7) must be used.

c) Three (3) samples (aliquots) are taken from 3 representative depths of the wood in the drum (top, middle and bottom depths of the materials).

d) Sampling procedure:

- the aliquots are collected from randomly selected large pieces at the 3 depths, by taking a core sample of the first 5 cm from the surface (or a full core sample for pieces less than 5 cm thick) to make an appropriate volume of the composite sample;

- to access the 3 layers of wood in the drum, the content of the drum can be transferred to a self contained flat surface where aliquots are collected at locations representing the 3 layers of the content of the drum. The content is returned to the drum for later classification and proper management. Proper care must be taken to avoid any spill and/or equipment contamination during the manipulation of the drum and its content.

e) The 3 aliquots are well mixed together to form one composite sample for analysis.

f) A portion of the composite sample is analysed for total PCB concentration, using a PCB analysis method approved or acceptable by MOE for classification of the waste as PCB waste (under Reg. 362, total concentration above 50 mg/kg). Waste with PCB concentration above 50 mg/kg must therefore be managed as PCB waste. For waste with results less than 6 mg/kg total PCB, the waste is classified as non PCB waste, and non leachate toxic waste (due to dilution in the leachate analysis). There is no need to conduct a leachate analysis on this sample (bypassing procedure 4.7).

g) Another portion of the same composite sample is analysed for PCB leachate concentration, using the LEP method (Leachate Extraction Procedure, Reg. 347). LEP results above 0.3 mg/L indicate that the waste represented by the sample is classified as hazardous waste (leachate toxic). The waste must therefore be managed as hazardous waste under requirements of Reg. 347.

h) For unusual situations, other representative sampling procedures can be proposed by a proponent, provided that the proposal is well justified and documented. The proposal should be presented in a formal document to MOE District Office for review and authorization (in consultation withe the Approvals Branch and/or the Standards Development Branch), before any initial sampling, analysis or work are conducted.

i) An alternative analytical approach can also be used for reducing the number of leachate tests, as indicated in section “Rationale for testing”, at the beginning of this document.

### 2.2.5 PCB contaminated electrical cables (in drums)

The contamination of used electrical cables with PCB oil is located in the paper wrapping between the copper conductor and the outside plastic or rubber jacket. The source of contamination may be either from migration of PCB from the PCB filled transformers, improper maintenance or use of contaminated equipment or manufacturing process. Representative sampling and testing involve the following procedures:

a) If there is any free liquid or sludge at the bottom of the drum, the free material should be sampled and tested for total PCB concentration. If the results are above 50 mg/kg, the content of the drum is classified as PCB waste, and further sampling is not required. If the results are less than 50 mg/kg, the following procedures apply.

b) Five (5) aliquots are collected from randomly selected sections of cable from each drum. The aliquots consist of short sections (1 to 5 cm) of the selected cables.

c) The 5 aliquots are well mixed together to form one composite sample for analysis, by shredding the cable sections and washing the inert parts (e.g. copper wire, metal casing) with n-hexane distilled in glass.

d) A portion of the composite sample is analysed for total PCB concentration, using a PCB analysis method approved or acceptable by MOE for classification of the waste as PCB waste (under Reg. 362, total concentration above 50 mg/kg). Waste with results above 50 mg/kg must therefore be managed as PCB waste.

e) Another portion of the same composite sample is analysed for PCB leachate concentration, using the LEP method (Leachate Extraction Procedure, Reg. 347). LEP result above 0.3 mg/L indicates that the composite sample is classified as hazardous waste (leachate toxic), along with the volume of waste that the sample represents. The waste must therefore be managed as hazardous waste (leachate toxic waste).

f) For unusual situations, other representative sampling procedures can be proposed by a proponent, provided that the proposal is well justified and documented. The proposal should be presented in a formal document to MOE District Office for review and authorization (in consultation with the Approvals Branch and/or the Standards Development Branch), before any initial sampling, analysis or work are conducted.

g) An alternative analytical approach can also be used for reducing the number of leachate tests, as indicated in section “Rationale for testing”, at the beginning of this document.

### 2.2.6 PCB contaminated liquid/sludge (in drums)

Various types of PCB (i.e. biphenyls with various levels of chlorination) have a density higher than that of water, ranging from 1.38 to 1.62 kg/L. In a mixture of water and PCB, the PCB will tend to precipitate at the bottom of the drums or form layers within the sludge. Only minor concentration of PCB are expected to remain in solution (PCB solubility in water range from approximately 6 mg/L for the less chlorinated PCB to 0.007 mg/L for the most chlorinated ones). Consequently, proper sampling of PCB contaminated liquid or sludge in drums requires firstly a separation of the various phases or layers.

a) Once the drums have been allowed to rest for the natural separation to take place, the visible layers that may be contaminated by PCBs are separated. The surface liquids are carefully pumped out. Any visible layer between the surface liquid and the bottom sediments is separated as waste likely to be contaminated with PCB.

b) One representative sample is collected from each of the separated phases or layers within each drum. Alternatively for a large number of drums showing the same type of separation, the supernatant water can be collected from several drums and sampled and analysed as one type of PCB waste. The other layers must be sampled however from each drum and tested individually.

c) A portion of the sample is analysed for total PCB concentration, using a PCB analysis method approved or acceptable by MOE for classification of the waste as PCB waste (under Reg. 362, total concentration above 50 mg/kg). Waste with PCB concentration above 50 mg/kg must therefore be managed as PCB waste. For waste with results less than 6 mg/kg total PCB, the waste is classified as non PCB waste, and non leachate toxic waste (due to dilution in the leachate analysis). There is no need to conduct a leachate analysis on this sample.

d) Another portion of the same composite sample is analysed for PCB leachate concentration, using the LEP method (Leachate Extraction Procedure, Reg. 347). LEP results above 0.3 mg/L indicate that the waste represented by the sample is classified as hazardous waste (leachate toxic). The waste must therefore be managed as hazardous waste under requirements of Reg. 347.

e) For unusual situations, other representative sampling procedures can be proposed by a proponent, provided that the proposal is well justified and documented. The proposal should be presented in a formal document to MOE District Office for review and authorization (in consultation with the Environmental Assessment and Approvals Branch and/or the Standards Development Branch), before any initial sampling, analysis or work are conducted.

f) An alternative analytical approach can also be used for reducing the number of leachate tests, as indicated in section "Rationale for testing", at the beginning of this document.

### 2.2.7 Mixed PCB materials (in drums)

a) In some cases, drums may contain various mixed materials, including the materials mentioned above (soil, rocks, concrete, wood, transformer, capacitors, electrical cables, packaging materials) and in some case protective equipment and materials (respirator filters, coverall suits, gloves, etc.). A drum is considered to contain mixed PCB materials if more than 10% of the content of the drum is of a different type than the remaining.

b) Drums that have visible phase separation either between liquids, between solids or between solid-liquid, must be separated and analysed separately for classification as PCB waste and leachate toxic waste. Procedures outlined in Section 2.6 (PCB contaminated liquid/sludge) must be used.

c) The procedure for handling mixed PCB materials in drums is to separate the mixed materials in varius types of materials and in visibly contaminated materials and visibly non-contaminated materials. Sampling and testing must be done on both type of materials (i.e. visibly contaminated and non-contaminated), using the protocol for materials stored in piles (see the following sections: PCB materials stored in piles).

d) The alternative approach is to simply consider the content of the drum as a PCB waste, and to leave the drum at the PCB site, or manage it as a PCB waste under Regulation 362 (PCB waste management) and Regulation 347 (Hazardous Waste Management).

### 2.2.8 Empty drums

Empty drums will be generated from the re-classification and management of the wastes stored at the PCB Storage Sites. Because the drums have been in contact with PCB contaminated wastes, it is important that these drums be properly cleaned and tested, before they can be recycled or disposed of as non hazardous wastes.

a) A drum is considered empty when there is less than 2.5 cm of residual wastes at the bottom. Empty drums from PCB waste storage are NOT excluded from the definition of hazardous waste, like other empty containers containing certain hazardous wastes (i.e. hazardous industrial waste, hazardous waste chemicals, or ignitable, corrosive, leachate toxic or reactive wastes).

b) Empty drums must be triple rinsed with appropriate solvents for removing any traces of PCB contamination. The solvents can be a mixture of water, steam, emulsifier, surfactant or non toxic degreaser, suitable for cleaning up the drums with 3 rinses. The rinse wastewater must be tested for total PCB concentration before disposal. Local municipal sewer bylaws should be considered before disposal into the sanitary sewer. The Ontario Water Resource Act will also dictate the quality of the wastewater that can be disposed on the land.

c) To confirm the efficiency of the 3 rinse operation, 10% of the cleaned drums are tested using a wipe test sampling methodology (see Appendix B). A minimum of 1 drum and a maximum of 15 drums should be tested from the inventory of rinsed drums at one site. This implies that at least one drum is tested for an inventory of 10 drums or less, and a maximum of 15 drums are tested for an inventory of 150 drums or more.

d) It is generally accepted that results of wipe tests remaining less than 10 ug/100 $cm^2$ determine that the empty drums can be recycled or re-used for industrial applications ("PCB Transformer Decontamination Standards and Protocols", CCME, 1995).

e) Empty drums that have been cleaned with a triple rinse operation can be considered non PCB waste and non hazardous waste. This subjective classification is applicable to steel or metal drums. Drums made of other materials such as plastics/polymers or other porous materials should be tested for total PCB and leachate.

## 2.3 – PCB MATERIALS STORED IN PILES

### 2.3.1 Segregation of mixed piles

Handling of PCB contaminated materials must be done according to the requirements of Ontario Regulation 362 (PCB waste management) regarding health and safety procedures, and environmental care in the handling of hazardous wastes.

a) The objective of segregating suspected PCB contaminated materials into various piles is to minimize the number of samples to properly characterize the materials as PCB and non-PCB wastes. Once properly segregated, each pile is sampled statistically to confirm that the pile is indeed classified as PCB waste or non-PCB waste. In the first step, the segregation is done by doing a visual inspection of the level of contamination, and assessing if this level is above or below 50 mg/kg of PCB. If the proponent feels that there is no visual reference for this concentration, the proponent would have to conduct some basic tests (relating the visual or apparent contamination with the actual PCB concentration in the waste) before segregating the suspected materials.

b) Mixed materials stored in drums are required to be segregated into piles or in storage containers before conducting proper sampling and analysis. The alternative is to sample each type of materials from each drum.

c) At some PCB storage sites, materials may already be stored in piles. Piles are often made of various types of materials (e.g. soil, sludge, rocks, concrete and asphalt, construction wood waste, metal waste/containers, etc.). For piles showing significant mixed materials (i.e. more than 10% of any material types), the piles must be segregated first in various types and in two levels of apparent (i.e. visual) contamination, namely one above, and the other, below 50 mg/kg of total PCB. Sampling and testing of each segregated pile is required to confirm the level of PCB contamination in both piles.

### 2.3.2 Representative sampling

The proper segregation of various PCB wastes into piles will ensure a certain degree of homogeneity in the concentration of PCB in each individual pile. A representative sampling program should be developed to confirm, with an acceptable level of confidence, that the concentration of total PCB in the pile is indeed below the Ontario standard of 50 mg/kg (PCB waste definition).

The following steps will ensure the proper characterization of the segregated piles.

a) For a pile representing a volume less than or equal to 3 cubic metres, a minimum of 3 aliquots are taken from each cubic metre of material. A composite of the 3 aliquots is analysed for total PCB, for each cubic metre. If any of the results indicate a concentration above 50 mg/kg total PCB, the pile is considered PCB waste.

b) The coning and quartering method is the usual method for compositing aliquots. The aliquots are thoroughly mixed with a shovel or scoop and piled into a cone. Each shovelful should be placed at the apex of the cone and allowed to run down the sides. After repeating this several times, the cone is flatten into a rough circle of uniform thickness and divided into four quarters. The two opposite quarters are rejected and the two other ones are retained. The procedure of coning and quartering, and then rejecting opposite quarters, can be repeated until the approximate required sample size is obtained. If required the rejected quarters may be stored as back-up or reference samples until testing is completed.

c) For a pile representing a volume above 3 cubic metres, a total of 15 random samples are collected from the pile. No compositing is done on the samples. Each sample is analysed for total PCB concentration.

d) The sampling procedures for the specific types of materials (i.e. soil, rock, concrete/asphalt, wood, etc) are the same as the ones for the specific materials in drums (see sections above in this document).

### 2.3.3 Statistical approach

The 15 results can be statistically analysed to confirm, with an acceptable level of confidence, if the pile of material can be classified as non PCB waste (i.e. less than 50 mg/kg).

The Student t-test is used as the statistical analysis for the 15 samples. The test assumes that the population (i.e. waste pile) is homogeneous, and consequently the distribution of the 15 sample results is normal around the average concentration of the pile. The higher the variation of the results and the closer (or higher) the results are to the target concentration (i.e. 50 mg/kg), the less confident we can state that PCB concentration in the pile is below 50 mg/kg.

In the t-test analysis, we want to demonstrate that the null hypothesis $H_0$ (i.e. the mean PCB concentration in the pile is greater than or equal to 50 mg/kg PCB) can be rejected for the alternative hypothesis $H_1$, (i.e. that the PCB concentration is less than 50 mg/kg).

$H_0$: $\overline{X} \geq$ 50 mg/kg PCB Null hypothesis

$H_1$: $\overline{X} <$ 50 mg/kg PCB Alternative hypothesis

From Student t-test statistical tables, the critical value of "t" is found to be -1.76 (one tailed t-test, with a confidence level of 95% and 14 (i.e. n-1) degrees of freedom). The calculated value of "t" is also obtained from the results of the 15 samples as:

$$\text{t (calculated)} = \frac{\overline{X} - \mu}{s/\sqrt{n-1}} = \frac{\overline{X} - 50}{s/\sqrt{14}}$$

Table 2.3a below provide the working sheet to derive the average value " $\overline{X}$ " and the standard deviation "s".

If the calculated t-value is less than the critical t-value of -1.76, the null hypothesis is rejected at the 95 % confidence level. Therefore the alternative hypothesis that the PCB concentration is less than 50 mg/kg is accepted.

If the calculated t-value is greater than or equal to the critical t-value of -1.76, we cannot confirm, with an acceptable level of confidence, that the pile is not PCB waste. The following actions can be taken for management of the pile:

a) The waste pile is classified as PCB waste and is managed as hazardous PCB waste, under Regulation 347.

b) The waste pile is segregated again, taking additional care to separate PCB wastes and non-PCB waste. The two newly segregated piles are then sampled again, following the same initial statistical approach.

c) NON-ACCEPTABLE APPROACH
   a) Taking more samples (more than 15) from the initial pile and selecting the lowest results is not acceptable.
   b) Taking additional samples from the initial pile to replace the ones with results that exceed 50 mg/kg is not acceptable.
   c) Resampling the initial pile with 15 new samples is not acceptable either.

Once again, this conclusion is applicable only if the pile is considered to have an homogeneous level of PCB contamination. This approach would not be acceptable if there was a potential for hot spots in PCB concentrations or the PCB waste pile has not been segregated before sampling.

**Legend (to Table 3a):**

Xi : individual analytical results for total PCB concentration (in mg/kg or ppm)

$\overline{X}$ : average concentration of the 15 samples, calculated as

$$\overline{X} = \frac{Xi}{[n]} = \frac{Cell:B16}{[15]}$$

S : standard deviation of the 15 samples, calculated as

$$S = \sqrt{\frac{[Xi - \overline{X}]^2}{[n-1]}} = \sqrt{\frac{Cell:D16}{[14]}}$$

Table 3a: Calculation Table for Student t-test

| Row/Column | A | B | C | D |
|---|---|---|---|---|
| 0 | SAMPLE # | Xi | Xi - $\overline{X}$ | [Xi - $\overline{X}$ ] $^2$ |
| 1 | 1 | | | |
| 2 | 2 | | | |
| 3 | 3 | | | |
| 4 | 4 | | | |
| 5 | 5 | | | |
| 6 | 6 | | | |
| 7 | 7 | | | |
| 8 | 8 | | | |
| 9 | 9 | | | |
| 10 | 10 | | | |
| 11 | 11 | | | |
| 12 | 12 | | | |
| 13 | 13 | | | |
| 14 | 14 | | | |
| 15 | 15 | | | |
| 16 | Summation for 15 samples | | | |
| 17 | | $\overline{X}$ = | | S = |

# 3 -- TESTING

## 3.1 RATIONALE FOR TESTING

Once a representative sample has been collected, adequate testing or analysis must be done on the sample to meet the requirements of the regulations pertaining to PCB waste classification and management.

Under Ontario Regulation 362 (PCB waste management), a PCB waste is generally defined as a material having a total PCB concentration of more than 50 mg/kg or parts per million (ppm). In the regulation, there are a number of definitions for special types of material potentially contaminated with PCB. For more details, Regulation 362 and the "PCB Transformer Decontamination Standards and Protocols " (CCME, December 1995, CCME EPC-HW-105E) should be consulted.

Under Ontario Regulation 347 (General Waste Management), a waste is also classified as hazardous waste (leachate toxic waste), when the PCB leachate concentration is above 0.3 mg/L (i.e. 100 times the Leachate Quality Criteria ( LQC) of 0.003 mg/L, in Schedule 4 of Reg. 347). The leachate test is done according to the Leachate Extraction Procedure (LEP) in Reg. 347. Wastes defined as PCB wastes in Reg. 362 are also classified as hazardous wastes under Reg. 347.

An alternative analytical approach to testing for both total PCB concentration and PCB leachate concentration can be used when a large number of analyses have to be performed. Because PCBs have a low water solubility and the LEP uses a mild acidic water leachant, a waste sample with a total PCB concentration of less than 50 mg/kg will most likely results in a leachate with a concentration considerably lower than 0.3 mg/L ( the leachate toxic level in Reg. 347). The alternative to testing all samples for both total concentration and leachate concentration is to verify that the leachate concentration in samples less than 50 mg/kg total PCB are indeed below the leachate toxic level (0.3 mg/L). This verification is done by analysing 10 % of the samples (previously analysed for total PCB) for leachate concentration. The leachate tests are performed on samples with a total PCB concentration <u>below and closest</u> to 50 mg/kg. If any of the leachate concentration are above 0.3 mg/L, a more comprehensive leachate testing program should be developed, in consultation and authorization by MOE.

## 3.2 TESTING METHODS

Traditionally, analytical methods used in environmental monitoring and compliance have been included in the regulations for references. The principles identified in the "Guidance on Sampling and Analysis", 1998 Model Sewer Use By-Law, Laboratory Analysis, MOE) reflect those derived from MOE methods. Rather than imposing specific analytical methods, a Performance Based Method System (PBMS) is applied here in this document. This system defines what is to be accomplished in the form of performance targets rather than how it should be done through prescriptive methods. It allows laboratories participating in the analysis to use any suitable method as long as it meets the specified Analytical Performance Criteria.

The MOE has available copies of the analytical procedures used routinely at the Laboratory Services Branch (LSB) for PCB analysis in soil and oil, and for leachate extraction. These methods are approved internally for use and are currently accredited by the SCC/CAEAL (Standards Council of Canada/Canadian Association for Environmental Analytical Laboratories). For the purpose of this Protocol, the LSB methods should be considered *reference* procedures and are not a requirement for use by external laboratories.

The following three methods, as described above, are currently used by the LSB:

b) Ministry of the Environment, 1998. The Determination of Polychlorinated Biphenyls (PCB) in Soil and Solid Industrial Waste by Gas Chromatography-Electron Capture Detection (GC-ECD) (PSAPCB-E3153A). Laboratory Services Branch, Etobicoke, Ontario.

c) Ministry of the Environment, 1998. The Determination of Polychlorinated Biphenyls (PCB) in Liquid Industrial Wastes by Gas Chromatography-Electron Capture Detection (GC-ECD) (POAPCB-E3145A). Laboratory Services Branch, Etobicoke, Ontario.

d) Ministry of the Environment, 1997. The Preparation of Samples by Regulation 347 and Distilled Water Procedures Including Determination of Moisture Content (REG347LEACH-E9001A). Laboratory Services Branch, Etobicoke, Ontario.

Technical inquiries for these methods can be directed to:

Quality Management Unit,
Laboratory Services Branch
Ontario Ministry of the Environment;

All inquiries related to method sales can be directed to:

Customer Services Unit
Laboratory Services Branch
Ontario Ministry of the Environment.

## 4 -- DECOMMISSIONING OF PCB STORAGE SITES

The final objective for certain PCB Storage Sites may be to clean up and decommission the site for other land use or application. In the planning for the decommissioning of a PCB Storage Site, there are 3 general issues to be addressed:

a. what is the required levels of cleanup for the soil on the property ?
b. what is the cleanup level for re-using the site structures and buildings ?
c. what is the classification and management of the wastes generate from the clean up ?

### 4.1 SOIL CLEAN-UP LEVELS

The general requirements for decommissioning contaminated sites are presented in the "Guideline for use at contaminated sites in Ontario" (Revised February 1997, MOE). In the guideline, there are criteria for cleanup levels for various land use applications and various contaminants in the soil. A summary of the PCB criteria for soil is presented in the following Table 4.1; PCB criteria for potable groundwater use and criteria for non-potable groundwater use are the same. For further details or explanations on the meaning of the criteria for PCB and other contaminants, please consult the Guideline.

**TABLE 4.1: Soil remediation criteria for PCB (all units in µg/g)**

| Ref: Ontario Contaminated Sites Guideline (1997) | Agricultural land use | Residential/ Parkland land use | Industrial/ Commercial land use |
|---|---|---|---|
| Full depth restoration | 0.5 | 5.0 | 25 |
| Stratified restoration | | | |
| -- Surface to 1.5 m | 0.5 | 5.0 | 25 |
| -- Below 1.5 m | 0.5 | 25 | No Value |

## 4.2 STRUCTURES AND BUILDINGS

Structures refer to any human made works, ranging from landing/loading pads, covered surfaces with concrete/asphalt, decks or in-ground trenches, tanks or piping. Buildings refer to any enclosed, or partially enclosed structures which can be used for storage, shelter or habitation.

Structures and buildings used for the storage of PCB wastes may have been contaminated during the course of the storage period, either from historical use of the structure/building, handling of PCB materials, spills, fire or explosion. The contamination may be either on the internal or the external walls of the storage building, or both. The floors are obviously the most likely to be contaminated, along with floor drain and catch basins..

a) The initial evaluation of the contamination of these buildings and structures includes the review of historical incidents and operations at the site. The collection of this background and historical information will facilitate the development of a representative sampling program, described further in the following paragraphs.

b) After the initial evaluation, a visual inspection of the surfaces and containment areas is conducted. The presence of oil stains, discolouration or oily surfaces may be the indication of PCB contamination, thus making the sampling program easier to develop. However, the absence of these visual clues are not an indication that the surfaces are free of PCB contamination.

c) The visually contaminated surfaces must be sampled, following the sampling methods described in this documents, depending if the surfaces are non-porous hard surfaces, or porous surfaces.

d) For non-porous hard surfaces and visually contaminated areas, the wipe test method (Appendix B in this document) is used to take a minimum of three (3) non-composited samples for a general contaminated area of the building, excluding floors and catch basins. The criterion for a surface to be considered relatively clean is less than or equal to 10 µg /100 $cm^2$. This criterion is based on health-risk studies and is generally accepted in the Canadian and US legislation as a reasonable risk level to human health and safety, and the environment.

e) For porous surfaces (such as wood, concrete) and visually contaminated areas, the sampling method is described in section 2.3 and 2.4 of this document. A minimum of three (3) non-composited samples is taken for a general contaminated area of the building. A total PCB concentration less than 0.3 µg/g in each of the individual samples is considered a cleaned surface. This criterion is based on the health-risk studies and is the level generally accepted for unrestricted use of materials as inert fill.

f) Confirmation of cleaned surfaces is done with random representative sampling. For relatively small areas ( ranging up to 10 $m^2$) one representative composite sample made of 2 or more wipe tests is adequate. For larger areas, composite samples are taken in areas where it is most likely that the vertical walls or ceiling were exposed to possible PCB contamination. Procedures for wipe test and compositing are included in Appendix B of this document.

## 4.3 MANAGEMENT OF CLEAN-UP MATERIALS

Any waste materials generated from the decommissioning of the PCB storage site must be classified properly for disposal. The sampling and classification is the same as for PCB contaminated wastes in drums or in piles, as detailed in the previous sections.

a) Materials are classified as hazardous waste under Regulation 347, if the total concentration of PCB in the waste exceeds 50 mg/kg (classified at PCB wastes) or the leachate concentration exceeds 0.3 mg/L (leachate toxic waste),

b) Non-hazardous waste can usually disposed at non-hazardous landfill sites or municipal landfill sites. Alternative uses of non-hazardous materials is possible, as long as the application respect the conditions identified in the draft “Criteria for the management of inert fill”.

Ministry of the Environment
Ministère de l'Environnement
STORAGE SITES
Appendix A

# POST NOTIFICATION FORM
FOR CLEAN-UP AT PCB

Ontario

**(in accordance with Protocol for Sampling and Testing at PCB Storage Sites in Ontario)**

**Part A: IDENTIFICATION**

1. Site Name: _____
2. PCB site Number: **__ __ __ __ __ A __ __ __**
3. Address _____
4. Generator Registration Number (GRR): **ON __ __ __ __ __ __ __**
5. Contact person: _____ Phone:_____

**Part B: CLEAN-UP OPERATION**

1. Date/Period of operation: _____

| 2. Waste removed from the site | Quantity (kg) | State (solid/liquid) |
|---|---|---|
| 2.1 Waste re-classified as NON-PCB wastes | _____ | _____ |
| 2.2 Waste classified as PCB wastes | _____ | _____ |
| 3. Waste remaining at the site | | |
| 3.1 Waste re-classified as NON-PCB wastes | _____ | _____ |
| 3.2 Waste classified at PCB wastes | _____ | _____ |

| **Part C: ATTACHMENTS and SIGNATURE** | | **YES** | **NO** |
|---|---|---|---|
| 1. Copy of Certificate of Destruction | | ____ | ____ |
| 2. Proper description of waste type and quantity | | ____ | ____ |
| 3. Updated PCB Inventory Form | | ____ | ____ |
| 4. Included Deregistration Component | (mandatory) | | |
| 5. Lab results (as per protocol) | (mandatory) | | |

Signature: _____
Title: _____ Date: _____

**This form, including the mandatory attachments, must be completed and submitted to the local MOE District Office within 30 days of reclassification.**

**APPENDIX B**

**WIPE TEST**
**(sampling procedures for PCB contaminated surfaces)**

(Source: this methodology is derived from the CCME "PCB transformer decontamination standards and protocols", December 1995)

**Purpose**

This wet wipe sampling methology is recommended for the verification of PCB concentrations on metal surfaces inside rinsed drums, or any other hard and non porous surfaces that are suspected of PCB contamination . If the results of the wipe test meet the cleanup criteria (i.e. less than 10 ug/100 $cm^2$), the metal drums can be sent to metal recyclers or re-used for industrial applications.

**Pre-sampling preparation**

1. In pre-cleaned 250-mL glass jars, place in each a 10 cm by 10 cm, 12 ply, sterilized gauze pad. The jars are then sealed with lids lined with hexane-cleaned aluminum foil or teflon.

2. Disposal templates are prepared such that the inner edges represent a minimum of 100 $cm^2$ surface area. The template can be of any shape appropriate for the surface being tested (e.g. square or rectangular) but should be at least 100 $cm^2$. Templates are normally made of heavy paper or cardboard.

**Required sampling equipment**

1. Prepared sampling jars, each containing a gauze pad.
2. Disposable templates.
3. Latex gloves.
4. Markers and labels.
5. Container for disposing of used templates and gloves.
6. Chain of custody sheets.
7. Metal forceps.
8. Solvent bottle of pesticide-grade hexane.

**Sampling methodology**

1. Using a disposable template and wearing latex gloves, mark with a piece of chalk a 100 $cm^2$ area on the inside metal surface to be tested. Dispose of the template and gloves. Alternatively, the template may be taped to the surface.

2. Open one sample jar. Wearing a clean pair of latex gloves, remove the gauze pad from the jar. Add 4 or 5 mL of hexane to the gauze pad quantitatively from a pump buret on the solvent bottle.

3. Starting in one corner, wipe the pre-marked area with the gauze pad in rows ensuring the entire area is covered. Use a uniform and steady pressure.

4. Open the gauze pad and refold to expose fresh surfaces.

5. Wipe the marked area in rows which are perpendicular to the previous ones. Ensure the entire area is swabbed equally. Use a uniform, steady pressure.

6. Place the gauze pad back in the jar. Close the lid and label the sample appropriately.

7. Dispose of the goggles.

8. Complete a chain of custody form prior to removing the samples for analysis.

**Analysis**

Analysis method is normally a capillary GC-ECD using Aroclor standards for quantification. Alternative analytical methods include GC-ELCD or GC-MS.