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September 11, 2000

The Honourable Joan Sawicki, MLA
Minister of Environment, Lands & Parks
PO Box 9047, Stn Prov Govt
Room 337, Parliament Buildings
Victoria, BC V8W 9E2

Dear Minister:

The Council of Forest Industries (COFI) and the BC Pulp and Paper Association (BCPPA) have been working with the provincial government for several years to facilitate the beneficial use of pulp and paper mill residuals (sludge). The utilization of these materials can improve agricultural land, reduce the use of fertilizers and greatly assist reclaiming disturbed sites. All these uses have positive environmental impacts.

A major impediment to increased beneficial use has been concern regarding the chemical constituents of the residuals produced by the various pulp and paper mills. To solve this problem, COFI, through the BCPPA, commissioned the attached study. After testing the residuals from six pulp and paper mills, representing nearly every pulp and paper process in use in BC, for more than 200 parameters, the conclusion is clear. To quote the report, “pulp and paper residual is a benign substance suitable for use as a soil amendment”.

After you review the report in detail, we believe you will agree the time has come to move forward with a regulation that will facilitate the beneficial use of this material while reducing incineration and use of landfill space.

We would also like to thank you and your staff for their support through this long process. If we can be of further help or assistance, please let us know.

Yours truly,

Ron J. MacDonald

Letter also sent to:

The Hon. Mike Farnworth, MLA
Minister of Health

The Hon. Corky Evans, MLA
Minister of Agriculture, Food & Fisheries

cc. Mr. Harry Vogt, MELP
Dr. Ray Copes, HLTH

Final Report – Pulp Mill Residual Chemistry and Options for Regulation

Prepared for:
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Organix File #9457-02.06

September 2000

EXECUTIVE SUMMARY

Pulp and paper mill "sludge" or residual is considered by many to be a valuable soil amendment. It provides organic matter to the soil, adds nutrients, and improves physical characteristics. Residual is applied to land in Alberta, Ontario, and Quebec, and in several states in the US. These land application programs are conducted under Regulation or streamlined Permit processes. In BC some limited land application programs have been allowed under Permits. Both BC Ministry of Environment, Lands & Parks (BCE) and the industry are working towards establishing a BC regulation for land application of the residual. Earlier steps in this process included participation in the Pulp Mill Sludge Committee of stakeholders including industry, unions, UBC, and environmentalists. The Committee process led to a BCE-prepared Draft 3 of the Pulp Mill Sludge Regulation (PSR Draft 3). This draft is considered unworkable by industry as it was based on out-of-date and incomplete information. Through the British Columbia Pulp and Paper Association (BCPPA), industry initiated a testing program and developed a recommendation. BCPPA commissioned Organix Waste Solutions Inc. (Organix) to conduct a survey of chemistry of residual from six participating mills and develop recommendations for a workable regulation.

Results of the Organix survey indicated that the pulp and paper mill residual is a benign substance suitable for use as a soil amendment. Organix especially focused on chlorinated organic compounds, dioxins and furans, phthalates and alkyl phenol ethoxylates, because some of these compounds are known or suspected endocrine disrupters. Organix found that none of the residuals surveyed contained chlorinated organic compounds and that dioxin and furan concentrations, with many congeners non-detectable, were well below the standards of the PSR Draft 3 for Class 1. Concentrations of phthalates and alkyl phenol ethoxylates were close to or below the detection limits.

Some parameters included in the testing requirements provided "false positive" results due to the characteristics of the residual. Organix recommends removing requirements for testing gross parameters such as light and heavy extractable petroleum hydrocarbons (LEPH/HEPH) and volatile petroleum hydrocarbons (VPH), as analyses for these parameters are sensitive for both petroleum hydrocarbons and compounds of biological origin such as those found in pulp and paper mill residual.

Virtually all samples tested positive in the fecal coliform and some tested positive in the *E.coli* test. These tests are indicator tests for pathogens. They detect both pathogen and non-pathogen organisms commonly found in waste water treatment systems. Results for *Salmonella*, a pathogen, were negative for all samples.

Using the results of our survey and a review of existing acts and regulations in BC, Organix developed recommendations for a new draft of the PSR. These recommendations modify some of the conditions in PSR Draft 3. They also provide flexibility to industry and regulators.

Our proposed classification system contains three classes: Class 1 – unrestricted use, Class 2 – managed use for residual that does not contain organo-chlorine compounds, and Class 3 – managed use for residual with low concentrations of organo-chlorine compounds. Organix proposes to allow only Class 1 and Class 2 residual on agricultural land. Residual adhering to these classes is managed based on agronomic parameters (Class 1) and concentrations of metals (Class 2).

The proposed classification system deals with the potential risk of pathogens. Class 1 sludge will not contain raw domestic sewage but may contain sewage that has been properly disinfected, Class 2 residual adheres to standards for *E.coli* and *Salmonella*, and Class 3 residual is managed as municipal biosolids.

For Class 1 residual, Organix proposes record keeping by the mill with submission of an annual report to BCE. In the case of Classes 2 and 3, a Management Plan must be submitted to the BC Ministry of Environment, Lands & Parks (BCE) prior to land application.

We propose adjusting the testing and monitoring requirements from those in the PSR Draft 3, as our survey results showed a reduced need and recommend testing "at source," rather than in stockpiles.

Residual from different mills vary in agronomic benefits. When properly managed, agronomic parameters will benefit plant growth without affecting the environment. Therefore no "standards" are proposed for these parameters. Organix recommends that mills provide guidance to residual end-users by preparing management options, including application rates. The mill will then work with the end-user to assure he follows the recommended practices.

Organix concludes that the recommended options, based on a chemical survey of residual from a cross-section of mill types and processes, will allow for beneficial reuse of pulp and paper mill residual as much needed organic soil amendments.

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1.0 Introduction

The British Columbia Pulp and Paper Association (BCPPA) is working to establish a regulation for the beneficial reuse of pulp mill sludge or "residuals." Earlier steps in the process included participation in the Pulp Mill Sludge Committee of stakeholders including industry, BC Ministry of Environment, Lands & Parks (BCE), unions, UBC, and environmentalists. The Committee process led to a BCE-prepared Draft 3 of the Pulp Mill Sludge Regulation (PSR Draft 3). This draft is considered unworkable by industry, and the BCPPA initiated a testing program and development of an industry-sponsored recommendation.

The BCPPA commissioned Organix Waste Solutions Inc. (Organix) to develop options for the industry-sponsored recommendation. The work on which the recommendation was based included a chemical characterization of the residual of six pulp and paper mills in BC, and the development of specific options for individual clauses for the regulation.

This report covers the results of the investigation of concentrations of compounds in residual from six mills in BC. It includes screening results for over 200 parameters, of which 119 are covered by the list contained in PSR Draft 3, and Organix conclusions and recommendations for a future draft of the PSR.

2.0 Approach

Organix conducted a survey of residual chemistry from six pulp and paper operations in BC, and developed options for the PSR.

To conduct the survey, Organix prepared a sampling and analyses protocol based on the list of parameters included in PSR Draft 3. This list included metals, chlorinated hydrocarbons, polychlorinated biphenyls (PCBs), polycyclic aromatic hydrocarbons (PAHs), dioxins/furans, alkyl phenol ethoxylates and derivatives, phthalates, and agronomic parameters. Our sampling protocol is attached in Appendix 1.

Organix developed options for each section of PSR Draft 3 from a range of documents. Our work focused on BC documents, as the regulations and standards they include are accepted by BCE, industry, and the public. To complete the scope, we also formulated some options that were not based on existing regulations, and we included the results of the pulp mill residual analyses project. For each identified option, we reviewed their advantages and disadvantages.

2.1 Residual Characterization

The residual characterization was a cooperative effort between Organix and mill staff. Based on a protocol developed by Organix, samples were taken by mill staff from pre-determined locations and process streams. Samples from each mill were stored in several containers to address specific storage requirements for each analysis. Samples were submitted to laboratories generally within 48 hours of being taken, well within the storage requirements for each parameter.

2.1.1 Laboratories

Organix used several laboratories for the analyses. Laboratories were selected based on track record, capability, schedule, and price. All analyses were performed according to recognized methods. A description of these methods is attached in Appendix 2. Samples were submitted to:

CanTest Ltd. in Burnaby, BC, for analyses of:

- Metals.
- Base neutral extractables.
- BETX (benzene, ethylbenzene, toluene, xylene) methanol extractables.
- LEPH/HEPH (light and heavy extractable petroleum hydrocarbons) extraction with Silica Gel cleanup.
- Non-chlorinated phenols.
- PAHs (polycyclic aromatic hydrocarbons).
- PCBs (polychlorinated biphenyls).
- Chlorinated phenols.
- Volatile organic compound (VOC) scan.
- Cyanide.

- Water Soluble Fluoride.
- Fecal coliform, *E.coli*, and *Salmonella*.

Axys Analytical Services in Sidney, BC, for analyses of:

- Dioxins and furans.
- Alkyl phenol ethoxylates and derivatives.
- Phthalate esters.

Pacific Soil Analyses Inc. in Richmond, BC, for analyses of:

- Agronomic parameters including
  - Hot water soluble boron (HWS-B).
  - Available nutrients and micronutrients.
  - Total nutrients.
  - Electrical Conductivity (EC).
  - Exchangeable Cations.

Some of the analyses were duplicated in different laboratories (e.g., phthalates) or with different extractions (PAH, BTEX). This duplication improved confidence in the results. Where results did not confirm historical data (mostly metals), samples were reanalyzed, or new samples were submitted.

2.1.2 Participating Mills

Six mills participated in this sampling program, representing a wide variety of mill types and processes. Mills included one non-integrated paper mill, two bleached Kraft mills, and two mechanical mills including BCTMP and TMP runs, and one mechanical pulp and newsprint operation. All bleaching systems were "elemental chlorine free" (ECF). The bleaching systems covered include no bleaching (1), peroxide/hydrosulphite bleaching (2), peroxide bleaching (1), and ClO_2 bleaching (2).

Each mill was contacted and the sampling protocol was discussed with the environmental manager. Mills were requested to submit archived data on residual analyses results to validate current data with recent historical records.

2.2 Options Analyses

Organix developed options for the major sections outlined in PSR Draft 3. Based on industry needs, Organix reviewed all the options and has recommended one option for each section of the proposed PSR. Recommended options are based on existing BC legislation and numerical standards.

3.0 Results

The results of the analyses showed that the chlorinated organic concentrations were below sample detection limits; dioxin/furan values were below the levels set in PSR Draft 3 for Class 1. The recommended options for a subsequent draft of the PSR lead to a workable, cost-effective regulation based on existing BC regulations and standards, and analysis results.

3.1 Residual Characterization

Residual from the six mills contained virtually no compounds at concentrations over the PSR Draft 3 Class 1 standards. Samples did not contain detectable concentrations of chlorinated hydrocarbons. Concentrations of dioxins and furans were well below the standards outlined in PSR Draft 3 for Class 1 residual. Concentrations of metals and trace elements were below or at the standards outlined in PSR Draft 3 for Class 1 residual.

Some sample results showed concentrations of some metals, PAH, and BTEX that were marginally above the standards set in PSR Draft 3 for Class 1. Additional samples were collected to confirmed these results. Residual from all but two mills included large numbers of fecal coliforms and *E.Coli* in tests that are commonly used as indicator tests for potential presence of pathogens. None of the residual tested positive for the pathogen *Salmonella*.

Below we address metals, PAHs, dioxins/furans, phthalates, alkylphenol ethoxylates, general indicators, microbial parameters, and agronomic parameters.

3.1.1 Metals

The concentration of metals in pulp mill residual depends on the quality of the mill's feedstock and on process chemicals used. The results for HWS-B showed that the current boron concentrations are well below the PSR Draft 3 limits for Class 1 (11mg/kg). The boron is likely related to the bleaching process where borate is used as a bleaching agent. Boron is a micronutrient rather than an environmental contaminant, but can be phytotoxic when applied in excessive amounts. Historic boron concentrations over the PSR Draft 3 standards may be due to differences in extraction technology (total boron vs. HWS-B) and reporting, or due to fluctuating use of borates in the bleaching process.

The residual from one mill contained concentrations of cadmium and zinc higher than the others. Results from this round of analyses confirmed the historical values.

Concentrations of tin in the samples were below detection limits and below the Class 1 limits of 5mg/kg. Samples were analyzed with a new technology using ICAP (Inductively Coupled Argon Plasma Spectroscopy) with an array detection system. This system is more accurate in detecting tin, as this metal is detected in the lower end of the emission spectrum. Older ICAP technology using a focussed beam is prone to interference and background noise, especially in samples with high organic content, resulting in artificially elevated concentrations as seen in historic data.

3.1.2 PAHs

Historical data and current data were generally matched in the PAH analyses. Some PAH components were found in the residual from one mill. Phenanthrene was detected at a concentration above the standards of PSR Draft 3 Class 1. Naphthalene in this sample was detectable but well below the Class 1 standard in PSR Draft 3. We resampled the residual and analysis results confirmed our earlier results.

3.1.3 Dioxins/Furans

Concentrations of dioxins and furans in the residual of all six mills were well below the standards of PSR Draft 3 Class 1 (10pg/g TEQ). Concentrations ranged from 0.184pg/g to 1.6pg/g TEQ, based on using a value of half the detection limit to calculate the TEQs. An alternative method of calculation is based on the value of zero for a non-detect result. With this method of calculation, the dioxin and furan results ranged from 1.48pg/g to 0.002pg/g TEQ.

A review of analyzed congeners showed that most of the concentrations were non-detect with sample detection limits of 0.1pg/g and 0.2pg/g. The detectable congeners included the 08CDD and the 08CDF, but not the more toxic 2,3,7,8 TCDD, which is a possible indicator for chlorine bleached processes.

Our results were obtained with very low sample detection limit. Recent work by Environment Canada[1] suggested that the estimated LoQ (Level of Quantification, the lowest level at which a product can be quantified confidently) for PCDD/PCDF is 9pg/g TEQ. This level was proposed by Environment Canada to virtually eliminate PCDD/PCDF from soil. This LoQ level is below the standard of 10pg/g TEQ used in BC regulations but is well above the range found in our analyses results. From the point of view of Environment Canada, dioxins and furans are no longer present in pulp mill residual.

3.1.4 Phthalates

Samples were analysed for phthalates by two laboratories. CanTest included these compounds as part of their assessment for base-neutral extractables, and Axys used their specialized methods of analyses with clean-up steps, serial dilutions, and addition of spiked compounds.

Results from both laboratories showed that the concentrations of phthalates, including bis(2-ethylhesyl phthalate (DEHP), were well below the standards included in PSR Draft 3. Due to the more complicated clean-up procedure, the results obtained by Axys showed lower detection limits. As part of the process, Axys was required to complete significant method development to

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1 Raouf, Morcos. 2000. Determination of Levels of Quantification for Measuring HCB and PCDD/PCDF in Soil – Draft. Environment Canada.

reach the required clean-up. This still resulted in some reported concentrations that must be considered maximum concentrations, as requirements for an absolute reading were not met. These concentrations are labelled with (*) in Table 1. Phthalate analyses results are therefore obtained with the best available technology.

3.1.5 Alkyl Phenol Ethoxylates

Results of the analyses for alkyl phenol ethoxylates showed that pulp mill residual contained either concentrations below the detection limits or small concentrations. One mill's residual contained NP1E0, a breakdown product of nonylphenol, at 2.2μg/g, a concentration in the lower range of those found in sediments[2]. Concentrations of octylphenol, considered to have a greater relative estrogenic response than other aklyl phenol ethoxylates[3], were non-detect or had potential maximum values at the detection limits.

3.1.6 Indicators for Petroleum Hydrocarbons

Gross indicators for petroleum hydrocarbons including volatile petroleum hydrocarbons (VPH), LEPH, and HEPH were high in all samples. These indicators for general presence of petroleum hydrocarbons are sensitive for both petroleum based compounds and biological based compounds. A review of chromatograms for these samples revealed that compounds were likely of biological origin. Therefore, these gross indicators are not suitable for identification of petroleum hydrocarbons in pulp mill residual as they result in false positive readings, even after cleanup with Silica Gel and correction for PAHs. We recommend removing these parameters from the testing protocol and assess the presence or absence of petroleum hydrocarbons with the specific parameters included in other analyses.

3.1.7 Microbial Indicators

Residual from several mills tested positive for fecal coliforms and *E.coli*. Both the "total coliforms," "fecal coliforms, " and "*E.coli*" tests are commonly used to indicate the potential presence of pathogenic micro-organisms.

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2 Bennie, D.T. 1999. Review of Environmental Occurrence of Alkyl phenols and Alkylphenol Ethoxylates. Water Qual. Res. J. Canada 34(1), 79-122.

3 Servos, M.R. 1999. Review of the Aquatic Toxicity, Estrogenic Responses and Bioaccumulation of Alkyl phenols and Aklylphenol Polyethoxylates. Water Qual. Res. J. Canada 34(1), 123-177.

In biological treatment systems (secondary systems), micro-organisms proliferate and can cause high positive results in the total coliform, fecal coliform, and *E.coli* tests without indicating pathogens. A literature review by Organix showed that pathogens could not be detected in analyses of process water and residual from Canadian pulp mills. A non-pathogen bacteria strain, *Klebsiella*, was reported to increase in the waste water system of pulp mills. This bacteria tests positive in a fecal coliform test (Gaudet and Coleman, 1998)[4]. Testing for specific groups of pathogens such as *Salmonella* is a recognized method to identify pathogens in pulp mill residual (Archibald, 2000)[5]. These tests have been incorporated in the Quebec regulation on pulp mill residual, and acceptable levels have been set.

We sampled and analyzed samples for *Salmonella* as parameters for the presence of pathogens. We found that the levels of S*almonella* were negative, even though some mills did include a domestic sewage in their secondary treatment system.

3.1.8 Agronomic Parameters

Several agronomic parameters varied widely among the six residual samples tested. The wide range comes from differences in the pulping process and the waste water treatment process. Below we describe some of these parameters including nitrogen and ammonia; sodium, electric conductivity, and Sodium Adsorption Ratio (SAR); and total and plant available iron. Other agronomic parameters are present in the residual. Their low concentrations will not cause them to limit agronomic applications.

Nitrogen and Ammonia

Levels of nitrogen in the pulp mill residual depend on the waste water treatment technology used. Secondary treatment generates more nitrogen in the residual than does primary treatment. This difference is visible in the test results. Concentrations in residual ranged from 0.06% to 4.18% total nitrogen. This difference is also visible in the levels of ammonia. Both the levels of total nitrogen and ammonia in the residual should be included in the calculation for agronomic application rates.

4 Gaudet, I.D. and R.N. Coleman. 1998. A Search of Available Literature to Determine Knowledge on the Persistence of Klebsiella Species and other Microorganisms Present in Land Applied Chemi-Thermo-Mechanical Pulp Mill Sludge. Alberta Research Council Report to Standards and Guidelines Branch, Alberta Environmental Protection. 28pp.

5 Archibald, F. 2000. The Presence of Coliform Bacteria in Canadian Pulp and Paper Mill Water Systems – A Cause for Concern? Water Qual. Res. J. Canada. 34:1-22.

Sodium, Electric Conductivity, and SAR

Sodium is a constituent of the chemicals used in the Kraft pulping process. Concentrations of sodium in the residual ranged from 540mg/kg to 3000mg/kg. The SAR (Sodium Adsorption Ratio) combines the activities of sodium, calcium, and magnesium. Where the SAR is >4, effects on sodium sensitive plants may be anticipated, while many plants may be affected with the SAR >8. We calculated the SAR for the pulp mill residual of the six mills. The SAR for five of the mills was well below 4. One residual sample showed a SAR of 6, indicating that the balance of sodium to calcium and magnesium is affected by the levels of sodium in the residual. We recommend that this mill incorporate SAR in the calculations leading to agronomic application rates.

The EC is a measurement of the total salt level in soil, residual, or irrigation water. An EC level of <2mS/cm in soil and irrigation water is generally considered a safe level for plants. However, most plants can tolerate soils with an EC of 3-4mS/cm. The EC of the six residual samples ranged from 0.4mS/cm to 4.2mS/cm, causing no concern to plants. Higher EC levels need to be included in agronomic application rates.

Iron

Three of the six residuals analyzed contained appreciable concentrations of both total and available iron. Concentrations in these samples were virtually identical at 2800mg/kg and 450mg/kg for total and available iron, respectively. Concentrations of total iron in the other residual products are in the 400mg/kg to 800mg/kg range, while plant available iron is in the 20mg/kg to 100mg/kg range. The origin of iron is likely the use of ferrichloride or ferrisulphate as a coagulant or flocking agent in the residual dewatering process. The total iron (0.2%) is above the standard cited in the Fertilizer Act for concentrations that could be declared on a label (0.1%). No standards exist for concentrations of iron in soil or in compost/soil amendments. Residual from these three mills could provide significant amounts of iron as valuable micronutrients, and iron should therefore be included as a basis for agronomic application rates.

3.1.9 Discussion

Method detection limits are important in characterizing chemistry in residuals. The residual is an organic material, releasing compounds that may affect methods for analyzing other organic parameters. The sensitivity of some analyses can be improved by taking more material in an extraction method. This works well in materials that are dry. In wet materials, the water content may affect the extraction method and increase the sample detection limits. This results in sample specific detection limits for certain compounds, some of which may be over the "allowable" concentrations.

Detection limits of some methods can be improved by adding cleanup methods, or by selecting specific compounds. We have used all methods available to improve the quality of the data and lower the detection limits. Duplication of analyses provided a cross-check on the reliability of methods. For instance, the monocyclic aromatic group (BTEX) was included in two different extractions, as was the PAH group. The analyses for phthalates was performed by two laboratories. One laboratory performed a specific (and expensive) analyses with lower detection limits, while the other laboratory included phthalates as part of the general extraction and adjusted the sample detection limits.

Analysis of some parameters is affected by the sample matrix. This has been recognized for cadmium, arsenic, and mercury. Laboratories use alternative methods for their detection. For tin, this has not been an issue in the past and methods were based on a typical matrix of soil with a low carbon content. The sample matrix from pulp mill residual samples appears to increase the detection levels of tin when an ICAP with conventional detection is used. We analyzed the samples with both old and new technology, the results from the old technology compared with historical data (also analyzed with this technology), while the data from the new, more accurate technology showed concentrations of tin below the detection limit. Tin in pulp mill residual should be analyzed with new equipment using array detection technology.

Some of the "standards" included in PSR Draft 3 Class 1 appeared to be unobtainable with the (approved) analyses methods used. Background "noise" in the detection masked the concentration peaks of certain compounds. The laboratories therefore reported concentrations with a higher sample detection limit or with an expected maximum value. This could partly be prevented by advanced clean-up procedures as was attempted in the analyses of phthalates and nonylphenols. Virtually all compounds with a sample detection limit over the "standard" are chlorinated compounds. As elemental chlorine is not used in the bleaching process in any of the six mills, we expect that chlorinated compounds are not present in residual. The "standards" may need to be re-evaluated to reflect best available technology for detection of compounds. We have indicated in Table 1 where sample detection limits are over the "standards," and where maximum expected values were generated.

3.1.10 Conclusion

From comparing the results of the analyses of residual from six pulp and paper mills with the PSR Draft 3 standards, we conclude that all mills will produce Class 1 or Class 2 residual. In fact, three mills could be classified as Class 1 mills and three mills as Class 2.

No residual contained chlorinated organics results above the sample detection limit. No residual contained dioxins or phthalates above standard levels specified in PSR Draft 3 for Class 1.

Gross parameters or screening parameters such as VPH, LEPH and HEPH, total coliforms, fecal coliforms, and *E.coli* cannot be used in the analyses of pulp and paper mill residual due to the high incidence of false positive results. Pulp mill residual should be analyzed for individual compounds and specific pathogens such as *Salmonella*.

Some compounds in pulp mill residual, while using recognized best available technology, cannot be analyzed to the levels included in the PSR Draft 3 Class 1 standards.

3.1.11 Recommendation

Testing of residual from six mills showed that no residual contained chlorinated organic compounds and that dioxin/furan concentrations were below the standards listed in the PSR Draft 3 for Class 1 residual. We recommend reducing testing for chlorinated organic compounds and dioxins/furans to once in the classification process.

Results of the tests showed that mill residual can be classified based on concentrations of metals and of some non-chlorinated hydrocarbons. We recommend focussing in a classification procedure on these parameters.

Gross indicators for petroleum derived substances such as LEPH, HEPH, and VPH are affected by biologically based compounds. We recommend removing these tests from any testing protocols.

Micro-organisms in secondary treatment facilities affect generally accepted test methods for pathogens. We recommend that domestic sewage is excluded from the wastewater treatment process, disinfected before inclusion, or that specific pathogen groups are used for testing the microbial safety of pulp and paper mill residual.

Several agronomic parameters vary widely between mills. We recommend having these parameters included in the calculation for agronomic/silvicultural rates. These include boron, molybdenum, total nitrogen, ammonia, iron, EC, and SAR.

The levels of tin measured through a regular ICAP detection method may be affected by the high organic matrix. We recommend using another method to detect tin, such as the ICAP with array detection.

We recommend matching "standards" with the results of best available, recognized analysis techniques, or using the sample detection limit as an acceptable standard.

3.2 Recommended Option

Organix has reviewed options for a regulatory framework. This framework is designed for the safe utilization of pulp and paper mill residual without the requirement of significant government resources. Our recommended option is based on existing BC legislation and numerical standards.

3.2.1 Review of Acts and Regulations

Several jurisdictions in North America have implemented or are implementing regulations for land application and beneficial reuse of pulp and paper mill residual. Most of these regulations combine the land application of sewage biosolids and other organic materials including pulp mill residual. These jurisdictions (e.g., Maine, Ontario) require permits but have streamlined protocols for pulp and paper mill residual. Alberta has a guideline for land application of residual from CTMP mills as a basis for a permit. Currently, all land application activities in BC are covered under the Waste Management Act's permit system. Under the Waste Management Act, BC can adopt regulations that waive the permit requirement when a set of rules and guidelines are followed. These regulations are commonly developed with input from and in consultation with industry and affected parties. Examples are the OMRR Draft 2, the Mushroom Composting Regulation, the Contaminated Sites Regulation, the Agricultural Waste Regulation, and the Agricultural Code of Practice. Other relevant acts and guidelines in BC that affect land application of residuals include the Fertilizer Act and the National Standard of Canada for organic soil conditioner and composts. Each of these documents have aspects that are applicable to the PSR.

The *OMRR Draft 2* will regulate land application of municipal biosolids and related products such as compost and soil amendments. This regulation includes classes for sewage residual and guidelines for use, and requires the submission of a Management Plan.

The *Mushroom Composting Regulation* describes the process and physical structure required for preparing substrate for the production of mushrooms. It requires the submission of a Pollution Prevention Plan to BCE prior to establishing a composting facility.

The *Contaminated Sites Regulation* provides standards for levels of contaminants in soil in BC, based on land use and site factors, and prescribes procedures for dealing with contaminated sites. Under this regulation, BCE can review consultants reports and issue letters of compliance, certifying proper remediation of a site. The standards from the Contaminated Sites Regulation are often (improperly) used for soil amendments and compost. BCE appears to favour the standards from this regulation because of its extensive list of parameters.

The *Agricultural Waste Regulation* and *Agricultural Code of Practice* describes how agricultural waste (mostly manure) must be handled and stored to prevent environmental damage. This includes setbacks from ditches and streams, storage facilities, both permanent and for field storage, and requirements for agronomic application (e.g., what the plant needs).

The *Federal Fertilizer Act* regulates quality and labelling standards for fertilizer. It also includes maximum cumulative additions of trace elements to soil.

The *National Standard of Canada – organic soil conditioners and compost* sets standards for the quality of compost and soil amendments. This standard includes identical conditions for metal levels to the Federal Fertilizer Act.

3.2.3 Recommended Option

As models to follow for a recommended option, we chose the OMRR and Mushroom Compost Regulation. These include the submission for review to BCE of a draft Management Plan, or a draft Pollution Prevention Plan. Below we present our selected option.

Coverage of Regulation – *The regulation will cover all mills and all residual uses unless restricted by Class requirements.*

This basis has been chosen to prevent the development of sub-regulations for a specific group of mills or for specific uses.

Class Types – *Regulation will have three residual classes:*

Class 1 – Unrestricted use, record keeping, and submission of an annual reports is required.

Class 2 – Residual Management Plan required for management of agronomic and silvicultural parameters, and pathogens.

Class 3 – Residual Management Plan required for management of chlorinated organic contaminants. Residual cannot be used in agriculture.

These classes provide the industry with flexibility while meeting BCE's concerns of safeguarding the environment. A full permit process is required for any residuals not included in the classification standards.

Class Limits –

***Class 1**:*

- *Concentrations below National Standard of Canada for metals – Soil amendments for metals Level A; CSR (AL) for other metals, or as modified.*

- *Concentrations for chlorinated hydrocarbons (CHC), PAH and BTEX as per CSR (AL) or PSR Draft 3 Class 1, or sample detection limit for approved methods.*
- *PCDD/F <10pg/g TEQ.*
- *All sewage is excluded from treatment process, or sewage is disinfected using an approved method prior to inclusion in the mill's process water treatment system.*

Class 2:

- *Metals concentrations below National Standard of Canada-Soil amendments Level B, and PSR Draft 3 Class 2.*
- *PSR Draft 3 Class 1 for chlorinated HC or sample detection limit.*
- *PAH, BTEX at PSR Draft 3 Class 2.*
- *PCDD/F <10pg/g TEQ.*
- *E.coli <1000MPN/g dry; Salmonella <3MPN/4g dry weight.*

Class 3:

- *Metals concentrations below National Standard of Canada-Soil amendments Level B, and PSR Draft 3 Class 2.*
- *PSR Draft 3 Class 2 for chlorinated HC, PAH, BTEX.*
- *PCDD/F <250pg/g TEQ.*
- *E.coli <2,000,000MPN/g.*

Class 1 and 2 are based on the trace metal concentrations. We found in our test program of pulp mill residual of six mills that chlorinated compounds were below the sample detection limit, and were below the standards set in PSR Draft 3. All sewage is excluded from Class 1 sludge, or it is disinfected using an approved method. Where this is not possible, the residual will be in Class 2 with limits for *E.coli* and *Salmonella* as indicators for pathogens. As pathogen control is manageable with composting, lime additions, or other management techniques commonly used in the municipal biosolids industry, pathogen levels are controlled in Class 1 only. Class 3 residuals may contain chlorinated hydrocarbons below specified concentrations or concentrations of dioxins/furans <250pg/g TEQ. These compounds require careful management and monitoring. Residual that does not meet the specified limit is not covered. It will convert to the conventional requirements for permit.

Classification Testing – *Testing at source (residual press).*

Testing at source is the simplest and most consistent way for classification testing, and is consistent for each mill. Downstream residual management may change from mill to mill, e.g. composting, and would be part of a Management Plan where required. Mills must be vigilant that each residual type (based on process, furnish) adheres to its own classification, as different wood sources (hardwood vs. softwood) or (bleaching) processes may affect the residual quality.

The classification testing at the source is actually more restricted than the provisions presented in PSR Draft 3, while being more cost-effective. Testing at source prevents the change of classification prior to land application through dilution, composting, or other techniques.

Test Procedure – *Three samples within a three month period; a three day, 10 part composite each. Samples to be submitted for analyses: one out of the three samples will be submitted for PCB, PAH, Phenols (chlorinated, non-chlorinated), metals, HWS-B, PCDD/F, NPE, phthalates, E.coli, and Salmonella. Two samples out of the three taken will be submitted for metals, HWS-B, E.coli, and Salmonella as a check on variability.*

Classification to be confirmed annually. Confirmation includes results of analysis of one 3 day, 10 part composite sample to be analysed for metals, HWS-B, E.coli, Salmonella. The Regional Manager may require additional parameters.

Classification Report – *Prepare a Classification Report.*

The Classification Report includes:

- *Name of mill.*
- *Description of sampling point.*
- *Description of process that generates the residual to be classified.*
- *Sampling technique.*
- *Analyses results.*
- *Details of management of domestic sewage.*

Notification (Class 1) – *Mill keeps records of all shipments; records are available upon request. Mill prepares an annual report to be submitted to BCE, including all records.*

The annual reports includes:

- *Classification report.*
- *Volumes shipped.*
- *Application rate.*
- *Name and address of owner of the land, or operator of facility.*

The ***residual user*** *will receive a product description including agronomic parameters and a recommendation for use (including fertilizer requirements/recommendations and loading rates).*

Management Plan (Class 2) – *Management Plan contains requirements for Class 1 notification plus:*

- *Agronomic application rates for any limiting factor in use of the residual.*
- *Soil management plan.*
- *Odour control plan.*
- *Monitoring plan.*
- *Auditing plan.*
- *Reporting plan.*

Management Plan (Class 3) – *Residual and residual products cannot be used in agriculture. Management Plan contains requirements for Class 1 notification and Class 2 management plan plus:*

- *Pathogen management plan conform to OMRR.*
- *Public notification and public response plan*
- *Monitoring plan including quarterly monitoring of residual and pre and post application monitoring of the soil at the application site for the compounds that caused the residual to be Class 3 residual.*

Management Plan to be submitted to the Regional Manager 45 days prior to application of Class 2 and Class 3 residual. In case of a Class 3 Management Plan, the results of a public notification program must be included in the Plan. Any changes to the Management Plan must be submitted to the Regional Waste Manager for review 45 days prior to implementation.

A Draft Management Plan should be submitted for the Regional Manager's comments prior to final submission. The Regional Manager may require additional tests, measurements, and reporting to be incorporated in the final Management Plan. This model is based on the Pollution Prevention Plan requirements of the Mushroom Compost Regulation, and on Schedule 7 of the OMRR. The mill develops an auditing plan. This plan establishes how the mill will manage the residual application, data handling, and compliance issues.

General Regulatory Requirements – *General regulatory requirements will be developed. These will parallel the Agricultural Code of Practice. Concentrations of constituents in soils cannot exceed the criteria of the Contaminated Sites Regulation for the land use of the site. Applications are not to exceed the Maximum Cumulative Trace Element Addition to Soil (National Standard of Canada).*

These regulatory requirements are accepted in the agricultural community and by BCE. The Agricultural Code of Practice was developed for manure, which has the potential to be a more potent pollutant than pulp and paper mill residual. These regulatory requirements will be in effect for all uses of pulp and paper mill residual.

4.0 Conclusion

Two of the six residuals tested would be classified as Class 1 residual under the recommended classification. Several mills are planning to separate sewage from their effluent screen and more mills will be classified as Class 1 mills. The survey of quality of residual from six mills in BC showed that pulp and paper mill residual do not contain concentrations of chlorinated organic compounds over the standards set in PSR Draft 3 for Class 1.

Application of pulp and paper mill residual can be managed based on the concentrations of metals and of other agronomic parameters.

The options for a regulation will cost-effectively manage land application of pulp and paper residual meeting the requirements of BCE.

5.0 Standard Limitations

Organix has prepared this report for the British Columbia Pulp and Paper Association. The report includes a survey of contaminants in pulp and paper mill residual, and an option for a workable Pulp Mill Sludge Regulation.

Respectfully submitted,

ORGANIX WASTE SOLUTIONS INC.

Per:

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TABLE 1
Overview of Residual Quality for Six Pulp and Paper Mills in BC
British Columbia Pulp and Paper Association
Organix File: 9457-02-06

	Draft 3 - Class 1 Sludge	Mill A, March 2000	Mill B, March 2000	Mill D, March 2000	Mill E, March 2000	Mill F, March 2000	Mill G, March 2000, Aspen	Proposed Class 1 Limits	Proposed Class 2 Limits	Proposed Class 3 Limits
Inorganic substances (mg/kg)										
Antimony (total)	<20	<10	<10	<10	<10	<10	<10	20	40	40
Arsenic (total)	<15	<0.5	<0.5	<0.5	<0.5	1.6	<10	13	75	75
Barium (total)	<500	227	44	169	142	110	188	500	2000	2000
Beryllium (total)	<4	<1	<1	<1	<1	<1	<1	4	8	8
Boron HWS (confirmed analysis)	<11	1.8	3.4	1.2	0.6	3.5	6.4	11		
Cadmium (total)	<1.5	<0.3	0.5	3.2	<0.3	1.2	<0.3	3	20	20
Chromium (hexavalentl)	<9						<5			
Chromium (total)	<60	12	<2	<2	20	15	10	210	1060	1060
Cobalt (total)	<40	<1	<1	<1	<1	<1	<1	34	150	150
Copper (total)	<40	19	13	35	21	23	16	100	757	757
Cyanide (weak acid dissociable)	<0.5	<0.15	<0.15	<0.15	<0.15	<0.15	<0.15	0.5	0.5	100
Cyanide (strong acid dissociable)	<5	<0.15	<0.15	<0.15	<0.15	<0.15	<0.15	5	5	500
Fluoride	<200	83	54.9	59	59	32	199	200	2000	2000
Lead (total)	<100	<1	<1	26	3	<1	<30	150	500	500
Mercury (total)	<0.8	0.005	0.015	0.961	0.0385	0.031	0.01	0.8	5	5
Molybdenum (total)	<5	<4	<4	<4	<4	<4	<4	5	20	20
Nickel (total)	<100	10	<2	21	7	<2	4	62	180	180
Selenium (total)	<2	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	20	14	14
Silver (total)	<20	8	<2	<2	5	<2	<2	20	40	40
Sulphur (elemental) %	<500		<200	<200	<200	<200	315	-	-	-
Thallium (total)	<2	<1	<1	<1	<1	<1	<1	2	10	10
Tin (total)	<5	<5	<5	<5	<5	<5	<5	5	5	50
Vanadium (total)	<200	6	2	2	8	12	8	200	-	-
Zinc (total)	<150	75	32	209	24	59	15	500	1850	1850
Aluminium (Al)		2910	307	2180	45200	471	438			
Calcium (Ca)		33800	8510	9210	370,000	5240	3830			
Iron (Fe)		2990	408	2180	3120	2730	320			
Magnesium (Mg)		1900	437	1490	4990	1670	1070			
Manganese (Mn)		973	25	1490	191	1060	13			
Sodium (Na)		3010	2270	4170	3380	1310	5820			
Strontium (Sr)		73	18	47	247	31	17			
Titanium (Ti)		187	14	67	97	9	6			
Organic Substances (mg/kg)										
Monocyclic aromatic hydrocarbons (mg/kg)										
Benzene	<0.04	<0.04	<0.04	<0.04	<0.01	<0.1	<0.1	0.04	4	4
Ethylbenzene	<0.1	<0.4	<0.1	<0.4	<0.01	<0.1	<0.1	0.1	7	7
Styrene	<0.1	<0.01	<0.01	<0.01	<0.01	<0.1	<0.1	0.1	50	50
Toluene	<0.1	<0.4	<0.1	<0.4	<0.01	<0.1	<0.1	0.1	2.5	2.5
Xylene	<0.1	<0.4	3.2	<0.4	<0.01	<0.1	<0.1	0.1	20	20
Phenolic substances										
Nonchlorinated										
2-Methylphenol (o-cresol)		<0.2	<2	<0.2	<0.2	<3	<0.2			
4-Methylphenol (p-cresol)		<0.3	<3	<0.3	<0.3	<4.5	<0.3			
[cresol]	<0.1									
2-nitrophenol	<0.1	<0.2	<2	<0.2	<0.2	<3	<0.2	0.1	0.1	10
2,4-dimethylphenol	<0.1	<0.2	<2	<0.2	<0.2	<3	<0.2	0.1	0.1	10
2,4-dinitrophenol	<0.1	<1.0	<10	<1.0	<1.0	<15	<1	0.1	0.1	10
4-Nitrophenol		<0.2	<2	<0.2	<0.2	<3	<0.2			
2-methyl 4,6-dinitrophenol	<0.1	<1.0	<10	<1.0	<1.0	<15	<1	0.1	0.1	10
phenol	<0.1	<0.07	<0.7	<0.07	<0.07	<1	<0.07	0.1	0.1	10
Chlorophenols										
Chlorophenol isomers (ortho)	<0.05	<0.5	<2.5	<0.5	<0.5	<5	<0.5	0.05	0.05	5
Chlorophenol isomers (meta)	<0.05	<0.5	<2.5	<0.5	<0.5	<5	<0.5	0.05	0.05	5
Chlorophenol isomers (para)	<0.05	<0.5	<2.5	<0.5	<0.5	<5	<0.5	0.05	0.05	5
dichlorophenol (2,3-)	<0.05	<0.1	<0.5	<0.1	<0.1	<1	<0.1	0.05	0.05	5
dichlorophenol (2,4-)	<0.05	<0.1	<0.5	<0.1	<0.1	<1	<0.1	0.05	0.05	5
dichlorophenol (2,5-)	<0.05	<0.1	<0.5	<0.1	<0.1	<1	<0.1	0.05	0.05	5
dichlorophenol (2,6-)	<0.05	<0.1	<0.5	<0.1	<0.1	<1	<0.1	0.05	0.05	5
dichlorophenol (3,4-)	<0.05	<0.1	<0.5	<0.1	<0.1	<1	<0.1	0.05	0.05	5
dichlorophenol (3,5-)	<0.05	<0.1	<0.5	<0.1	<0.1	<1	<0.1	0.05	0.05	5
trichlorophenol (2,3,4-)	<0.05	<0.02	<0.02	<0.02	<0.02	<0.02	<0.01	0.05	0.05	5
trichlorophenol (2,3,5-)	<0.05	<0.02	<0.02	<0.02	<0.02	<0.02	<0.01	0.05	0.05	5
trichlorophenol 2,3,6-)	<0.05	<0.02	<0.02	<0.02	<0.02	<0.02	<0.01	0.05	0.05	5
trichlorophenol (2,4,5-)	<0.05	<0.02	<0.02	<0.02	<0.02	<0.02	<0.01	0.05	0.05	5
trichlorophenol (2,4,6-)	<0.05	<0.02	<0.02	<0.02	<0.02	<0.02	<0.01	0.05	0.05	5
trichlorophenol (3,4,5-)	<0.05	<0.02	<0.02	<0.02	<0.02	<0.02	<0.01	0.05	0.05	5
tetrachlorophenol (2,3,5,6)	<0.05	-	-	-	-	-				
tetrachlorophenol (2,3,4,5-)	<0.05	<0.01	<0.01	<0.01	<0.01	<0.01	<0.005	0.05	0.05	5
tetrachlorophenol (2,3,4,6-)	<0.05	<0.01	<0.01	<0.01	<0.01	<0.01	<0.005	0.05	0.05	5
pentachlorophenol	<0.02	<0.01	<0.01	<0.01	<0.01	<0.01	<0.005	0.02	0.02	2

Notes - ## Measurement over Draft 3 Class 1 Standard
Sample detection limit over Draft 3 Class 1 Standard

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British Columbia Pulp and Paper Association
Organix File: 9457-02-06

	Draft 3 - Class 1 Sludge	Mill A, March 2000	Mill B, March 2000	Mill D, March 2000	Mill E, March 2000	Mill F, March 2000	Mill G, March 2000, Aspen	Proposed Class 1 Limits	Proposed Class 2 Limits	Proposed Class 3 Limits
Polycyclic aromatic hydrocarbons										
Naphthalene	<0.1	<0.05	<0.25	<0.1	<0.05	0.06	<0.05	0.1	50	50
Acenaphtylene		<0.05	<0.25	<0.05	<0.05	<0.05	<0.05	0.1	10	10
Acenaphtene		<0.05	<0.25	<0.05	<0.05	<0.05	<0.05	0.1	10	10
Fluorene		<0.05	<0.25	<0.05	<0.05	<0.05	<0.05	0.1	10	10
Anthracene		<0.05	<0.25	<0.05	<0.05	<0.05	<0.05	0.1	10	10
Benzo(g,h,i)perlyne		<0.05	<0.25	<0.05	<0.05	<0.05	<0.05	0.1	10	10
Chrysene		<0.05	<0.25	<0.05	<0.05	<0.05	<0.05	0.1	10	10
Fluoranthene		<0.05	<0.25	<0.05	<0.05	<0.05	<0.05	0.1	10	10
Benz(a)anthracene	<0.1	<0.05	<0.25	<0.05	<0.05	<0.05	<0.05	0.1	10	10
Benzo(b)fluoranthene	<0.1	<0.05	<0.25	<0.05	<0.05	<0.05	<0.05	0.1	10	10
Benzo(k)fluoranthene	<0.1	<0.05	<0.25	<0.05	<0.05	<0.05	<0.05	0.1	10	10
Benzo(a)pyrene	<0.1	<0.05	<0.25	<0.05	<0.05	<0.05	<0.05	0.1	10	10
Dibenz(a,h)anthracene	<0.1	<0.05	<0.25	<0.05	<0.05	<0.05	<0.05	0.1	10	10
Indeno(1,2,3-c,d)pyrene	<0.1	<0.05	<0.25	<0.05	<0.05	<0.05	<0.05	0.1	10	10
Phenanthrene	<0.1	<0.05	<0.25	<0.05	<0.05	0.27	<0.05	0.1	50	50
Pyrene	<0.1	<0.05	<0.25	<0.05	<0.05	<0.05	<0.05	0.1	100	100
Chlorinated hydrocarbons										
Chlorinated aliphatics										
Bromodichloromethane		<0.4	<0.1	<0.4	<0.01	<0.1	<0.1			
Bromoform		<0.4	<0.1	<0.4	<0.01	<0.1	<0.1			
Bromomethane		<1.6	<0.4	<1.6	<0.04	<0.4	<0.4			
2-Butanone		<20	<5	<20	<0.5	5	<5			
Carbon Tetrachloride		<0.4	<0.1	<0.4	<0.01	<0.1	<0.1			
Chloroethane		<0.8	<0.2	<0.8	<0.02	<0.2	<0.2			
Chloroform	<0.1	<0.4	<0.1	<0.4	<0.01	<0.1	<0.1	0.5	0.5	50
Chloromethane (methylchloride)	<0.1	<1.6	<0.4	<1.6	<0.04	<0.4	<0.4	0.5	0.5	50
Dibromochloromethane		<0.4	<0.1	<0.4	<0.01	<0.1	<0.1			
1,2-Dibromomethane		<0.4	<0.1	<0.4	<0.01	<0.1	<0.1			
Dibromomethane		<0.4	<0.1	<0.4	<0.01	<0.1	<0.1			
Dichlorodifluoromethane		<0.8	<0.2	<0.8	<0.01	<0.2	<0.1			
2-hexanone		<20	<5	<20	<0.5	<5	<5			
4-Methyl-2-pentanone		<8	<2	<8	<0.2	<2	<2			
Methylene Chloride		<12	<3	<12	<0.2	<3	<3			
Vinyl Chloride (chloroehtene)		<0.8	<0.2	<0.8	<0.02	<0.2	<0.2			
Dichloroethane (1,1-,)	<0.1	<0.4	<0.1	<0.4	<0.01	<0.1	<0.1	0.5	0.5	50
Dichloroethane (,1,2-)	<0.1	<0.8	<0.2	<0.8	<0.02	<0.1	<0.1	0.5	0.5	50
1,2-dichloropropane	<0.1	<0.4	<0.1	<0.4	<0.02	<0.1	<0.1	0.5	0.5	50
1,3-dichloropropene (cis,)	<0.1	<0.4	<0.1	<0.4	<0.01	<0.1	<0.1	0.5	0.5	50
1,3-dichloropropene (trans)	<0.1	<0.4	<0.1	<0.4	<0.01	<0.1	<0.1	0.5	0.5	50
1,1,2,2-tetrachloroethane	<0.1	<0.4	<0.1	<0.4	<0.01	<0.1	<0.1	0.5	0.5	50
Tetrachloroethene		<0.4	<0.1	<0.4	<0.01	<0.1	<0.1			
Carbon tetrachloride	<0.1	<0.4	<0.1	<0.4	<0.01	<0.1		0.5	0.5	50
Trichloroethane (1,1,1-)	<0.1	<0.4	<0.1	<0.4	<0.01	<0.1	<0.1	0.5	0.5	50
Trichloroethane (1,1,2-)	<0.1	<0.4	<0.1	<0.4	<0.01	<0.1	<0.1	0.5	0.5	50
Trichloroethene		<0.4	<0.1	<0.4	<0.01	<0.1	<0.1			
Trichlorofluoromethane		<0.4	<0.1	<0.4	<0.01	<0.1	<0.1			
Other compounds										
Aniline		<1	<10	<1	<1	<15	<5			
Bis(2-chloroehtyl)ether		<0.06	<0.6	<0.06	<0.06	<0.9	<0.3			
Benzyl Alcohol		<0.3	<3	<0.3	<0.3	<4.5	<1.5			
Bis(2chloroisopropyl)ether		<0.05	<0.5	<0.05	<0.05	<0.75	<0.25			
Hexachloroethane		<0.05	<0.5	<0.05	<0.05	<0.75	<0.25			
N-nitorso-di-n-propylamine		<0.8	<8	<0.8	<0.8	<12	<4			
Nitrobenzene		<0.09	<0.9	<0.09	<0.09	<0.14	<0.45			
Isophorone		<0.1	<1	<0.1	<0.1	<1.5	<0.5			
Bis(2-chloroethoxy)methane		<0.06	<0.6	<0.06	<0.06	<0.9	<0.3			
4-Chloroaniline		<0.5	<5	<0.5	<0.5	<7.5	<2.5			
Hexachlorobutadiene		<0.2	<2	<0.2	<0.2	<3	<1			
2-Methylnaphtalene		<0.3	<3	<0.3	<0.3	<4.5	<1.5			
Hexachlorocyclopentadiene		<1	<10	<1	<1	<15	<5			
2-chloronaphtalene		<0.07	<0.7	<0.07	<0.07	<1	<0.35			
2-Nitroaniline		<0.4	<4	<0.4	<0.4	<6	<2			
2,6-Dinitrotoluene		<0.2	<2	<0.2	<0.2	<3	<1			
3-Nitroaniline		<0.3	<3	<0.3	<0.3	<4.5	<1.5			
2,4-Dinitrotoluene		<0.07	<0.7	<0.07	<0.07	<1	<0.35			
4-Chlorophenyl-phenylether		<0.04	<0.4	<0.04	<0.04	<0.6	<0.2			
4-Nitroaniline		<0.4	<4	<0.4	<0.4	<6	<2			
N-nitrosodiphenylamine		<0.8	<0.3	<0.8	<0.8	<0.45	<0.15			
Azobenzene		<0.06	<0.6	<0.06	<0.06	<0.9	<0.3			
4-Bromophenyl-phenylether		<0.03	<0.3	<0.03	<0.03	<0.45	<0.15			
Acridine		<0.03	<0.3	<0.03	<0.03	<0.45	<0.15			
Monochlorobenzens										
Chlorobenzene	<0.1	<0.4	<0.4	<0.4	<0.01	<0.1	<0.1	0.1	0.1	10
Dichlorobenzenes										
1,2-dichlorobenzenes	<0.1	<0.08	<0.8	<0.4	<0.4	<0.1	<0.1	0.1	0.1	10
1,3-dichlorobenzenes	<0.1	<0.09	<0.9	<0.4	<0.4	<0.1	<0.1	0.1	0.1	10
1,4-dichlorobenzenes	<0.1	<0.08	<0.9	<0.4	<0.4	<0.1	<0.1	0.1	0.1	10

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British Columbia Pulp and Paper Association
Organix File: 9457-02-06

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Chlorobenzenes										
1,2,4 Trichlorobenzene	<0.05	<0.05	<0.5	<0.05	<0.05	<0.75	<0.25	0.05	0.05	10
Tetrachlorobenzenes	<0.05	-	-	-	-	-				
Pentachlorobenzenes	<0.05	-	-	-	-	-				
Hexachlorobenzene (perchlorbenzene)	<0.05	<0.04	<0.4	<0.04	<0.04	<0.6	<0.2	0.05	0.05	10
hexachlorocyclohexane (Lindane	<0.01	-	-	-	-	-				
Total Polychlorinated biphenyls	<0.5									
Arochlor 1242		<0.03	<0.03	<0.03	<0.03	<0.03		0.5	0.5	50
Arochlor 1248		<0.03	<0.03	<0.03	<0.03	<0.03		0.5	0.5	50
Arochlor 1254		<0.03	<0.03	<0.03	<0.03	<0.03		0.5	0.5	50
Arochlor 1260		<0.03	<0.03	<0.03	<0.03	<0.03		0.5	0.5	50
PCDDs and PCDFs (TEQ, ng/kg)	10							10	10	250
PCDDs and PCDFs (TEQ, pg/g) @ 1/2 DL		1.6	0.184	0.438	0.372	0.249	0.92			
PCDDs and PCDFs (TEQ, pg/g) @ DL=0		1.48	0.002	0.282	0.218	0.07	0.01			
Selected cogeners										
2,3,7,8 TCDD		<0.1	<0.1	<0.1	<0.1	<0.1	<0.5			
1,2,3,7,8 PCDD		<0.1	<0.1	<0.1	<0.1	<0.1	<0.5			
1,2,3,4,7,8 H6CDD		<0.2	<0.2	<0.2	<0.2	<0.2	<1.0			
1,2,3,6,7,8 H6CDD		0.5	<0.2	0.5	0.3	<0.2	<1.0			
1,2,3,7,8,9 H6CDD		0.2	<0.2	0.3	0.3	<0.2	<1.0			
1,2,3,4,6,7,8 H7CDD		16	<0.3	2.9	3.8	2.4	<1.7			
O8CDD		200	1.9	15	37	37	6			
2,3,7,8 TCDF		7.8	<0.1	1.6	0.8	<0.1	<0.5			
1,2,3,7,8 P5CDF		0.4	<0.1	<0.1	<0.1	<0.1	<0.5			
2,3,4,7,8 P5CDF		0.4	<0.1	<0.1	<0.1	<0.1	<0.5			
1,2,3,4,7,8 H6CDF		<0.2	<0.2	<0.2	<0.2	<0.2	<1.0			
1,2,3,6,7,8 H6CDF		<0.2	<0.2	<0.2	<0.2	<0.2	<1.0			
2,3,4,6,7,8 H6CDF		0.3*	<0.2	<0.2	<0.2	<0.2	<1.0			
1,2,3,7,8,9 H6CDF		<0.2	<0.2	<0.2	<0.2	<0.2	<1.0			
1,2,3,4,6,7,8 H7CDF		2.5	<0.3	<0.3	0.5	0.6	<1.7			
1,2,3,4,7,8,9 H7CDF		0.4	<0.3	<0.3	<0.2	<0.3	<1.7			
O8CDF		25	<0.5	<0.5	5.6	3.2	<2.7			
		* = Peak detected but did not meet quantification criteria: indicates potential maximum value.								
Petroleum hydrocarbons										
Volatile petroleum hydrocarbons	<200	280	560	130	<100	290	<100	no test	no test	no test
Volatile Hydrocarbons (6-10)		280	560	130	<100	290	<100	no test	no test	no test
LEPH, corrected for PAH, silica gel clean	<1000	1400	4200	510	<250	3600	<250	no test	no test	no test
HEPH corrected for PAH, silica gel clean	<1000	1300	8500	4200	1600	2000	<250	no test	no test	no test
Origin of components		biological	not petroleum based	biological	biological	biological				
Phthalic acid esters (µg/g)		CanTest/Axys	CanTest/Axys	CanTest/Axys	CanTest/Axys	CanTest/Axys	CanTest/Axys			
Di-n-butyl phthalate	<30	<3 / 0.110*	<30/4.500	<3 / 0.180*	<3 / 0.570	<45/ 2.500	<15/ 0.190	<30	<30	<30
Bis(2-ethylhexyl) phthalate (DEHP)	<30	<0.8 / 0.230	<8/3.700*	<0.8 / 1.500*	<0.8 / 0.740	<12/ 3.000*	<4/ 0.300*	<30	<30	<30
Dimethylphthalate		<0.06 / 0.960	<0.6/ <0.370	<0.06 / 0.0021*	<0.06 / 0.0027*	<0.9/ <0.160	<0.3/ 0.011			
Diethylphthalate		<0.03 / 0.029	<0.3/ <0.180	<0.03 / 0.033	<0.03 / 0.0012	<0.45/ 0.390*	<0.15/ 0.030*			
Butyl Benzyl phthalate		<0.9 / 0.210	<9/ 5.700	<0.9 / 0.230*	<0.9 / 0.140	<14/ 1.100*	<4.5/ 0.082			
Di-n-octylphthalate		<0.08 / 0.022*	<0.8/ <0.400	<0.08 / <0.051	<0.08 / 0.023	<1.2/ 0.360*	<0.4/ 0.026*			
		* = Peak detected but did not meet quantification criteria: indicates potential maximum value.								
Alkylphenols (ng/g)										
4-Nonylphenol		130	130	<22	180/170	<940	<5.8			
NP1EO		2200	<600	<28	280/260	<170	<53			
NP2EO		<66	<2600	<150	<38/<29	<1500	<180			
Octylphenol		<2.0	64*	<2.5	<1.5/<0.85	<16	<4.1			
		* = Peak detected but did not meet quantification criteria: indicates potential maximum value.								
Additional Parameters										
TKN Nitrogen %		0.06	0.67	1.6	0.14	2.89	4.18			
Total Nitrogen										
Ammonium		10	32	15	6.7	2600	307			
Nitrate		73	76	33	12	76	56			
[Available nitrogen]										
Sulfate-S							783			
Bulk Density, wet (g/cm3)		0.61	0.72	0.55	1.15	0.54	0.82			
Bulk Density, dry (g/cm3)		0.16	0.18	0.16	0.44	0.16	0.11			
Electric Conductivity (mS.cm)		1.3	2.96	0.76	0.8	4.2	3			
Organic Matter										
[TOC]		46.8	61.6	46.9	21.1	50.4	49.5			
PH		8.4	6.8	6.7	7.9	6.6	7			
Moisture content (%)		75.2	72	71.3	60.7	71.5	75.1			
Cation Exchange Capacity me/100g		56.3	90.8	25	13.6	83.9	136.6			
Echangeable Ca me/100g		64	25.8	22	113	24	12.5			
Excahngeable Mg me/100g		5.6	3.24	2.9	1	7.9	6.67			
Exchangeable Na me/100g		12	10	8.8	0.9	3	19.2			
Exchangeable K me/100g		0.7	1	0.8	0.3	4.6	9.99			
SAR		2.03	2.62	2.49	0.12	0.75	6.20			

TABLE 1
Overview of Residual Quality for Six Pulp and Paper Mills in BC
British Columbia Pulp and Paper Association
Organix File: 9457-02-06

	Draft 3 - Class 1 Sludge	Mill A, March 2000	Mill B, March 2000	Mill D, March 2000	Mill E, March 2000	Mill F, March 2000	Mill G, March 2000, Aspen	Proposed Class 1 Limits	Proposed Class 2 Limits	Proposed Class 3 Limits
Available AL		725	45	325	3	180	130			
Availlable Calcium		8667	3500	3500	10750	2200	2333			
Available Copper		5.3	1.4	8.5	1	8.4	4			
Available Iron		450	24	400	<1	460	23			
Available Magnesium		33	363	350	90	740	967			
Available Manganese		550	21	538	11	880	9.9			
Available Sodium		2133	2375	2075	475	900	5833			
Available Zn		63	14	70	0.5	44	13			
Total P, %		0.08	0.16	0.16	0.06	0.59	0.47			
Available P		244	119	293	35	1610	943			
total K, %		0.14	0.05	0.05	0.04	0.29	0.26			
Available K		123	263	218	45	1560	4167			
Total Colifrom MPN/100 g								no test	no test	no test
Fecal Coliform MPN/100g (as is)		negative	17,000,000	540,000	2500	9,200,000				
Fecal Coliform MPN/g (dry)		negative	607143	18815	64	322807		no test	no test	no test
Salmonella MPN/g		negative	negative	negative	negative	negative	negative	No sewage	negative	no test
E Coli MPN/100g (as is)		negative	2400000	35000	230	700000	350000			
E Coli MPN/g (dry)		negative	85714	1220	6	24561	14056	No sewage	<1000	<2,000,000

APPENDIX 1

SAMPLING PROTOCOL

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SAMPLING PROTOCOL

Introduction

The BC Pulp and Paper Association (BCPPA) has retained Organix Waste Solutions Inc. (Organix) to assist them with gathering test data on the quality of pulp mill residual (also known as sludge or Short Paper Fibre waste or SPF). BCPPA members decided that data should be gathered to characterize the sludge from participating mills and that new and existing data should be presented to the BC Ministry of Environment, Lands & Parks (BCE) as a basis to write a workable and cost-effective Pulp Mill Sludge Regulation. The wide range of processes and furnishes will provide a sense for the variability in residual quality.

This document outlines step by step the sampling protocol to be followed for the data analysis.

Protocol

Samples taken from fresh residual will be analyzed for the parameters listed in Draft 3 of the Pulp Mill Sludge Regulation. As this is a screening of the residual from six participating mills, the consistency of sampling methods and sampling handling is extremely important. Therefore, to properly prepare this protocol, we have interviewed each mill to evaluate factors that may affect analyses results and to select the proper sampling location. This protocol includes:

- A list of analyses.
- Containers.
- Sample location customized for each participating mill.
- Description of the expected sludge product for this mill.
- Timing of sampling.
- Required sampling tools.
- Sample handling.
- Return of samples to Organix.

Analyses

The Draft 3 of the Pulp Mill Sludge Regulation includes testing requirements for the following parameters:

- Metals
- Cyanide
- Fluoride
- Sulphur
- PCB
- PAH
- Chlorinated and non-chlorinated phenols
- Light and heavy extractable petroleum hydrocarbons
- Volatile organic compounds
- Base neutral extractables (gasoline compounds)
- Dioxins/furans
- Nonylphenol ethoxylates (NPE) and derivatives
- Phthalates
- Available micronutrients/metals + SAR
- Macronutrients (N, P,K)
- CEC
- pH
- Ammonium and nitrate

Containers

The myriad of analyses requires sending samples to three laboratories. Further, some of the analyses require specific containers to prevent contamination. Therefore, we have provided you with one bag (to be double bagged) and five containers. This will provide the laboratories with ample sample for their analyses.

Sample Location

Samples will be taken at the discharge of the residual press.

Expected Residual Quality

The residual originates from the following processes:

ClO_2 bleached Kraft

- BCTMP process
- TMP process
- Paper mill

The pulp/paper furnishes included:

- Market pulp
- Soft wood
- Aspen

Time of Sampling

Sampling is scheduled for Monday, March 27 or Tuesday, March 28, 2000, unless other arrangements have been made.

Sampling Tools

Sampling tools required are:

- A stainless steel bowl or bucket capable of holding 3L of sample.
- A stainless steel or teflon spoon or spatula.

Sampling tools must be washed with isopropanol and air dried.

Sampling

Sampling includes one **discrete** sample. Please collect the sample in the bowl or bucket, mix, and spoon the collected material in the containers and bag provided. Please note the sequence for filling the containers: please fill the 1L amber bottle first and the plastic bag last. Please fill the plastic bag with about 1L of material.

It is critical that sampled material to go into the 1L amber bottle does not come in contact with any plastic materials such as sample bags, drop sheets, packing materials, plastic gloves, or plastic utensils.

Please close the plastic bag with the provided twist tie and double bag.

Sample Handling and Storage

Some parameters have limited holding time. Sample handling and storage is designed to provide sufficient preservation. Please store the filled containers in a refrigerator (~+4°C) for several hours or overnight to cool, and freeze the supplied ice pack.

Repack the cooled samples and the frozen ice pack in the shipping cooler immediately prior to shipment

Return of Samples

Please return samples via (overnight) courier to:

Organix Waste Solutions Inc.

Attention: Hubert Timmenga

#1200 – 1185 West Georgia Street

Vancouver, BC, V6E 4E6

Timing of Return of Samples

Please forward the samples to our office to arrive here on Wednesday, April 5, before Noon. This provides us with enough time to collect samples from all participating mills, repack them, and forward them to the laboratories in order to start analyses before the weekend. By sending the samples from all mills as a single batch we are able to reduce the analytical costs.

APPENDIX 2

METHODS FOR ANALYSES

APPENDIX 2

METHODS FOR ANALYSES

Samples were analyzed for parameters included in Draft 3 of the Pulp Mill Sludge Regulation by CanTest Ltd. (Burnaby, BC), Axys Analytical Services Ltd. (Sidney, BC), and Pacific Soils Analyses Inc. (Richmond, BC). The following methods of analyses were used:

CanTest Ltd.

Elemental Sulphur: analysis was performed based on the method "Extraction and Colorimetric Determination of Elemental Sulfur in Organix Horizons of Forest Soils" in Canadian Journal of Soil Science, Vol 65 no.4 November 1985, pages 811-813.

Moisture in soil: analysis was performed gravimetrically by heating a separate sample portion at 105°C and measuring the weight loss.

pH in soil or solid: analysis was performed based on procedures described in the Manual on Soil Sampling and Methods of Analysis, published by the Canadian Sociaent of Soil Science, 1993. The test was perfumed using a dionized water leach with measurement by pH meter.

Conventional Parameters: analyses were performed using procedures based on those described in "British Columbia Environmental Laboratory Manual for the Analysis of Water, Waste water, Sediment and Biological Materials" (1994 Edition), Province of British Columbia, and "Standard Methods for the Examination of Water and Wastewater" 19th Edition (1995) and 17th Edition (1989, published by the American Public Health Association.

Extractable Petroleum Hydrocarbons in Water/Soil (LEPH/HEPH-GNS): analysis was performed using a draft DCM extraction-GC/FID procedure specified by the BC Ministry of Environment, Lands & Parks (BCE).

Polynuclear aromatic hydrocarbons: analysis was performed using procedures based on US EPA Methods 625/8270, involving extraction, clean-up steps, and analysis using GC/MS.

Polychlorinated biphenyls: analysis was performed using procedures based on US EPA Methods 604/8040, involving extraction, clean-up steps and analysis using GC/ECD.

Chlorinated phenols: analysis was performed using procedures based on US EPA methods 604/8040, involving extraction, derivatization, clean-up steps and analysis using GC/ECD.

Arsenic in soil: analysis was performed using Zeeman background-corrected Graphite Furnace Atomix Absorption Spectrophotometry.

Cadmium in Soil: analysis was performed using background corrected Flame Automic Absorption Spectrophotometry.

Mercury in Soil or Solids: analysis was performed using Cold Vapour Atomic Absorption Spectrophotometry.

Lead in Soil: analysis was performed using background-corrected Flame Atomic Absorption Spectrophotometry.

Metals in Soil or Solids: non-dried representative samples were digested with a mixture of nitric acid and hydrochloric acid – "Aqua Regia." Analysis was performed using Inductively Coupled Argon Plasma Spectroscopy (ICAP) or by specific techniques as described.

Selenium in soil or solids: analysis was performed using Zeeman background-corrected Graphite Furnace Atomic Absorption Spectrophotometry.

Thallium in soil: analysis was performed using an acid digestion followed by determination using Inductively Coupled Plasma Mass Spectrometry (ICP/MS).

Volatile organic compounds in water and soil: analysis was performed using procedures based on US EPA Methods 624/8240/8260, involving sparging with a Purge and Trap apparatus and analysis using GC/MS.

Axys Analytical Services Ltd.

Polychlorinated Dibenzodioxins and Dibenzofurans

All samples were spiked with ^{13}C-labelled surrogate standards prior to analysis. Samples were soxhlet extracted. All extracts were subject to a series of washing and chromatographic clean-up steps prior to analysis for polychlorinated dibenzodioxins and dibenzofurans by high resolution gas chromatography with high resolution mass spectrometric detection (HRGC/HRMS).

Phathalate Esters

All samples were spiked with an aliquot of surrogate standard solution containing perdeuterated analogues of phthalate esters prior to analysis. Samples were soxhlet extracted. Each extract was cleaned up on silica gel prior to analysis of phthalate esters by high resolution gas chromatography with low resolution (quadrupole) mass spectrometric detection (HRGC/MS).

Nonylphenols

Samples were spiked with ^{13}C-labelled nonylphenol and soxhlet extracted for three hours in dichloromethane. The extract was reduced to a small volume and reacted with acetic anhydride to convert nolylphenol to its acetate derivative. The extract was cleaned up by adsorption chromatography on silica, concentrated to 500µL and analyzed by gas chromatography/mass spectrometry (GC/MS) operated in the multiple ion detection mode (MID). The nonylphenol concentration was determined by Isotope dilution internal quantification.

Pacific Soils Analyses Inc.

All analyses were conducted according to the:

- Manual of Soil Sampling and Methods of Analysis, 1987, J.A. McKeague.
- Methods Manual, Pedology Laboratory, 1977. L.M. Lavkulich, Department of Soil Science, UBC.

pH and EC were determined in a 1:1 residual to water slurry.

Total carbon is determined using a LECO Carbon Analyzer.

Available phosphorus is determined colorimetrically using the ascorbic acid method on a 1: 10 residual to Bray Olsen extractant.

Available Ca, Mg, K are determined by Perkin-Elmer Atomic Absorption Sectrophotometer on a 1:5 residual to ammonium acetate extractant.

Available boron is determined colorimetrically on a hot water extract using the azomethine-H method.

Cation-exchange-capacity and exchangeable cations are determined by the 1N Ammonium Acetate method at pH 7.

Available NH4-N and NO3-N are determined colorimetrically on a K2SO4 extract.

Samples were digested by the Parkinsen and Allen method to analyse for total N, P, K, Ca, Mg, and Al. Dry ashed samples were used to determine total Cu, Zn, Fe, Mn and B.