

# **Weathered PHC F2 and the Eco-Contact Pathway – Phase I – Quantifying the Effects of Weathering**

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Canada-wide Standards (CWS) for Petroleum Hydrocarbons (PHCs) have developed in soil. Stantec developed the terrestrial toxicity test methods used to generate the data from which, in part, the standards were derived. The fraction-specific testing was completed by Stantec using these draft standard methods (Stantec, 2003). The Tier 1 standards (first issued June of 2001 and revised March 2007 and re-issued January 2008; CCME 2008a) serve as soil quality standards to assist with the management of site soils contaminated with petroleum hydrocarbons. The concentrations of the CCME fractions (F1 to F4) are measured in soil from a site and screened against these Tier 1 standards. If the concentrations measured in the site soils are higher than the Tier 1 fraction-specific values for the soil contact exposure pathway, there is an option to proceed to Tier 2. At this time, there is no provision at Tier 2 for adjustment of the Tier 1 standards associated with this soil contact

exposure pathway. There is, however, a provision for the potential elimination of the exposure pathway (CCME, 2008b).

In the course of the re-evaluation of the Tier 1 standards, the F2 values were lowered significantly from 900 mg/kg to 150 mg/kg and from 1,500 mg/kg to 260 mg/kg soil dry weight for soils associated with agricultural/residential and commercial/industrial land use classes, respectively. These values apply equally to both fine and coarse soils. The basis for the lowering of the values cited in the CCME (2008a) Supporting Technical Document was: 1) the test soils, that were used to generate the data from which the initial Tier 1 standards were derived, were fine textured and not coarse textured so the application of an adjustment factor of 2 was an error; 2) the effects endpoints used in the revised derivation were EC/IC/LC25s as opposed to EC/IC/LC50s; and, 3) the revised derivation included effects data for plant and soil invertebrate species as opposed to plant species only. The result was a significant lowering of the Tier 1 F2 standards.

It is generally acknowledged that weathering and aging of petroleum hydrocarbons in soil decreases the toxicity of these soils to ecological receptors. It is hypothesized that the decrease in toxicity could be attributed to a decrease in the bioavailability of the constituents due to sequestration and/or a preferential degradation of the more toxic

constituents. However, the magnitude of this decrease depends on a number of factors including the concentration and composition of the hydrocarbons in soil, the nature of the soil, and the chemical, physical, and biological conditions under which the weathering and aging occurs.

Weathering by definition refers to the relative change in the composition of contamination due to the preferential loss of constituents with time. Note that weathering has been used in various contexts within the literature. Aging refers to the time-dependent change in bioavailability of a compound(s) in soil (Stantec, 2004). In this definition, aging includes all sorption and sequestration processes, including the formation of non-extractable residues. Sequestration describes the time-dependent movement of contaminant molecules into remote, inaccessible areas of soil particles and/or organic matter. Sequestration does not involve the formation of covalent bonds. Non-extractable residues result from the time-dependent formation of residues that cannot be solvent extracted from soils, and which can only be removed upon hydrolysis with a strong alkali or acid (Alexander, 1999). These residues might involve the formation of covalent bonds between the parent compound or a metabolite with the organic matter. Residual fraction is the contaminant(s) remaining in the soil following weathering and aging (Stantec, 2004). This fraction includes those compounds that are resistant to degradation and other loss mechanisms, as well as

those that are unavailable to organisms for degradation. Therefore, it is the fraction that will remain in the soil with relatively little change in both composition and concentration over an indefinite period of time.

Phase 1 testing of fine-grained soil spiked with F2 and “weathered” in the laboratory was completed by Stantec Consulting Ltd. in 2009 (Angell et al., 2012; Stantec, 2009). The species sensitivity distribution constructed from the Phase 1 data indicates that weathered F2 is half as toxic as fresh F2 to ecological receptors in soil (Angell et al., 2012; Stantec, 2009). A “Request for Proposals” was issued by PTAC for a toxicity assessment of coarse-grained soils spiked with F2 and “weathered” in the laboratory to a stable endpoint where the F2 concentration is no longer decreasing significantly. The objective of Phase 2 ecotoxicity testing was to generate LC/EC/IC25s and LC/EC/IC50s for multiple endpoints and for a test species battery exposed to weathered PHC F2 in a coarse-grained soil.

This project contains the test reports and analytical reports and summaries for Phase 2 of a project facilitated by PTAC and in collaboration with Steve Kullman (Husky Energy) and Miles Tindal (Axiom Environmental Consulting Ltd.) whereby a toxicity assessment was conducted on coarse-grained artificial soils spiked with F2 and “weathered” in the laboratory to a stable endpoint. A stable endpoint is achieved

when the F2 concentrations are no longer decreasing significantly. Weathering cannot occur realistically under laboratory conditions but the soils can be spiked with F2 and aged and a simulation of weathering can take place. The aim of the testing in this project was to generate LC/EC/IC25s and LC/EC/IC50s for multiple endpoints and for a test species battery using coarse-grained soils that were contaminated with “weathered and aged” F2. Specific objectives were to:

1. Amend a coarse-grained soil with a range of F2 concentrations and “age” and “weather” these soils under laboratory conditions until F2 residuals are chemically stable.
2. Expose a battery of test species (plant and soil invertebrate species) to these soils with a gradient of stable residuals to quantify the exposure concentration-response relationships for each endpoint and each species.

The test species are representative of two major groups of soil organisms, plants and soil invertebrates. The monocotyledonous plant species were northern wheatgrass (*Elymus lanceolatus*) and barley (*Hordeum vulgare*), and the dicotyledonous plant species was alfalfa (*Medicago sativa*). The earthworm species is commonly referred to as the red wiggler or compost worm (*Eisenia andrei*) and soil arthropods were represented by the springtail (*Collembola* – *Folsomia candida*). The test species used are consistent with the earlier work completed in support of the

development of Tier 1 PHC standards and Phase 1 of the present PTAC project; however, *Folsomia candida* replaced *Orthonychiurus folsomi* because, although the sensitivities to petroleum hydrocarbons are comparable, the former has less variability associated with test results. The test methods and procedures were those of Environment Canada (EC, 2007, 2005a, 2004).

Reference toxicity tests with boric acid and each test species were also conducted concurrently to comply with the test protocols of Environment Canada; they are also a mandatory requirement for QA/QC purposes for CALA-accredited laboratories. The results of the reference testing have been included in each test report.