WEATHERED PHC F2
AND THE ECO-CONTAT PATHWAY
PHASE I REPORT

Report Prepared for:
Petroleum Technology Alliance Canada (PTAC)
Suite 400, Chevron Plaza
500 – Fifth Avenue S.W.
Calgary, Alberta T2P 3L5

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Prepared by:

Miles J. Tindal, M.Sc.
Principal
Axiom Environmental Inc.
438 Eagle Heights
Canmore, Alberta T1W 3C9
Phone: (403) 678-4790
mtindal@axiomenvironmental.ca

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Introduction

The latest version of the CCME Petroleum Hydrocarbon Canada-Wide Standard revised the guideline for the ecological contact with plants and soil invertebrates pathway (the "eco-contact pathway") for PHC fraction F2 down from 900 mg/kg to 150 mg/kg (natural area, agricultural, and residential land use) and from 1,500 mg/kg to 260 mg/kg (commercial and industrial land use). This is the limiting pathway for F2 at the vast majority of sites. Current Alberta Environment Tier 1 Guidelines adopt this same value. This change to the F2 guideline value has significant implications for the management of hydrocarbons at upstream and downstream petroleum hydrocarbon sites.

Various work has indicated that plant and invertebrate toxicity in soil can be lower for aged/weathered than fresh F3 PHCs. However, very little corresponding work on the toxicity of aged/weathered F2 appears to have been conducted. The current project collects and analyzes data to support a possible future revision to the F2 eco-contact guideline. The current document reports on Phase I of this work, which considers the ecotoxicity of aged/weathered F2 in a fine grained soil. Funding is currently being sought for Phase II of this work which would expand the scope to consider a coarse-grained soil.

Methodology

In overview, the methodology used in this project included the following steps:

- Conduct a rangefinding test to investigate the kinetics of F2 degradation in soil.
- Based on the rangefinding test, spike an F2 distillate into an Orthic Black Chernozem soil to generate a concentration series.
- Age/weather these samples for six months.
- Conduct triplicate chemical analysis on each sample
- Conduct ecotoxicity testing using standard Environment Canada protocols and a standard battery of three plant and two invertebrate species.
- Derive soil remediation guidelines for the eco-contact pathway for weathered F2 in fine-grained soil using standard CCME protocols.

Results and Guideline Development

The soil remediation guidelines for aged/weathered F2 in fine soil are:

- 290 mg/kg – natural area, agricultural, and residential land use.
- 400 mg/kg – commercial and industrial land use.

Funding is currently being sought to expand the scope of this work to include coarse soils. If funded, a combined report will be submitted to AENV for their consideration.

1. INTRODUCTION

The latest version of the Canadian Council of Ministers of the Environment (CCME, 2008) Petroleum Hydrocarbon Canada-Wide Standard (PHC CWS) revised the guideline for the ecological contact with plants and soil invertebrates pathway (the "eco-contact pathway") for PHC fraction F2 down from 900 mg/kg to 150 mg/kg (natural area, agricultural, and residential land use) and from 1,500 mg/kg to 260 mg/kg (commercial and industrial land use). This is the limiting pathway for F2 at the vast majority of sites. Current Alberta Environment Tier 1 Guidelines (AENV, 2009) adopt this same value.

This change to the F2 guideline value has significant implications for the management of hydrocarbons at upstream and downstream petroleum hydrocarbon sites. Little of the difference between old and new F2 guideline values is due to new data that have become available since 2001. The two main reasons for the difference are i) a factor of 2 was erroneously applied to previous guideline values to extrapolate from coarse to fine soils, when in reality the test soils on which the guideline was based were fine, and ii) The CCME (2005) protocol uses a distribution of 25th percentile effect data, rather than the 50th percentile effect data used in the 2001 PHC CWS derivation. Based on current protocols and currently-available data, the revised F2 guideline of 150 mg/kg may be appropriate for fresh F2 hydrocarbon.

Various work, including several former PTAC studies (Axiom, 2005; Visser, 2005a,b) have indicated that plant and invertebrate toxicity in soil can be lower for aged/weathered than fresh F3 PHCs. It has been theorized (e.g., Alexander, 1997) that this lowering of toxicity may be due to sequestering of hydrocarbon by soil leading to lower bioavailability and/or the preferential removal of more toxic components of a hydrocarbon fraction. However, very little work on the toxicity of aged/weathered F2 appears to have been conducted.

The former PTAC studies on the ecotoxicity of aged/weathered PHC F3 were some of the key data considered by the CCME in revising the eco-contact guideline for F3 upward from 800 mg/kg to 1,300 mg/kg in 2008. The current project is intended to collect data to support a possible future revision to the F2 eco-contact guideline. The current document reports on Phase I of this work, which considers the ecotoxicity of aged/weathered F2 in a fine grained Orthic Black Chernozem soil. Funding is currently being sought for Phase II of this work which would expand the scope to consider a coarse-grained soil.

1.1 Objective

The overall objective of the work presented in this report was to collect data to support a possible future revision to the F2 eco-contact guideline.

1.2 Scope of Work

The scope of work that was completed for Phase I of this project included the following tasks.

- Develop a workplan for the project.
- Conduct a rangefinding test with F2 spiked into Orthic Black Chernozem soil and regularly aerated and mixed, to investigate F2 degradation kinetics.
- Identify a series of initial F2 concentrations that are anticipated to generate an appropriate series of aged/weathered F2 concentrations for ecotoxicity testing.
- Spike F2 into Orthic Black Chernozem soil and simulate an aging/weathering process with regular aeration, mixing, and management of moisture content.
- Conduct sufficient analytical testing to determine when an appropriate level of aging/weathering has been achieved.
- At this point, initiate the following battery of ecotoxicity tests:
    - Definitive plant growth tests with northern wheatgrass, alfalfa and barley.
    - Invertebrate survival and reproduction tests with earthworm and springtail species.
- Use CCME (2008) methodology to calculate a draft guideline value for aged/weathered F2 in a fine-grained soil.
- Generate a report summarizing key elements of the program.

1.3 Acknowledgements

This project was a collaborative effort among many parties, and we would like to acknowledge the following:

Funding

- Environmental Research Advisory Council (ERAC)
- Bodycote Labs (in kind).

Coordination

- CAPP Project Manager: Steve Kullman, Husky Energy Inc.
- PTAC Coordinator: Tannis Such, PTAC coordinator.

Research Providers

- Axiom Environmental Inc. – Miles Tindal (project scope, data analysis, reporting).
- Exova Laboratories – Darlene Lintott (rangefinding testing).
- Stantec Consulting Ltd. – Gladys Stephenson et al. (ecotoxicological testing).

In addition, we are grateful to Stantec for making available F2 distillate and Orthic Black Chernozem soil that had been obtained for previous projects.

2. AGING AND WEATHERING

When hydrocarbon mixtures are added to soil, a number of processes can occur over time. Processes which reduce the bioavailability of the hydrocarbons to biota are referred to as aging. Processes which preferentially remove some components from the mixture and hence change its composition are collectively termed weathering processes. Both these processes can occur concurrently, and both can potentially contribute to a reduction of soil ecotoxicity over time, and hence the term “aged/weathered” is used in this report.

Aging of hydrocarbons in soil involves a number of processes including sorption and sequestration, all of which make hydrocarbons less available or unavailable to plants and soil invertebrates. ESG (2003b) includes a detailed review of these processes, and the details are not repeated in this summary report. Aggressive solvent extraction (such as the hexane/acetone Soxlet extraction used in the PHC CWS reference method, CCME, 2001) may still be able to recover much of the hydrocarbon sorbed or sequestered to soil components, and may not, therefore, be a particularly good indication of how much hydrocarbon is biologically available to plants or soil invertebrates.

Weathering refers to compositional changes in hydrocarbon mixtures over time as a result of various processes (e.g., biodegradation, volatilization, leaching) occurring at different rates for different hydrocarbon components. For instance, n-alkanes may be biodegraded more rapidly than aromatic hydrocarbons, resulting in compositional changes in the hydrocarbon mixture in the soil.

3. METHODOLOGY

Some of the previous work on aged/weathered F3 was based on aged/weathered soils that had previously been spiked with a whole hydrocarbon mixture (e.g., crude oil, diesel), or were the product of a spill (release) of a whole hydrocarbon mixture. Once thoroughly aged/weathered, these data could generally be interpreted in terms of the toxicity of the residual F3, since typically the F2 component had been degraded more rapidly than the F3. Such a method, however, cannot be used to determine the toxicity of aged/weathered F2, since any results would be confounded by residual F3 toxicity.

Thus, the work in this project to determine the toxicity of aged/weathered F2 was undertaken by spiking a distilled F2 cut into soil, conducting an accelerated aging/weathering treatment, and then performing eco-toxicity tests on the aged/weathered soils.

3.1 Overview

In overview, the methodology used in this project included the following steps:

- Identify a source of a PHC F2 cut and Orthic Black Chernozem soil.
- Conduct a rangefinding test to determine the rate of degradation of F2 under lab conditions.
- Based on the results of the rangefinding test, generate and weather/age a spiked F2 soil series to produce a suitable series of aged/weathered F2 concentrations for ecotoxicity testing.
- Conduct ecotoxicity testing with three plant and two invertebrate species
- Use the data from the ecotoxicity testing to calculate draft guideline values for aged/weathered F2 in fine-grained soil.

3.2 Source of Test Materials

Fraction F2 refers to hydrocarbons in the range equivalent to straight chain alkanes with a carbon chain length greater than 10 and up to 16. The F2 used for this work was originally generated for an earlier ecotoxicity project which was part of the process to develop the original PHC CWS guidelines (ESG, 2003a). The F2 used in the rangefinding test was from a distillation of Federated Crude Oil made by Environment Canada. The F2 used for the ecotoxicity tests was distilled in 2002 by Imperial Oil Ltd. at their Sarnia, Ontario facility from Federated Crude Oil. The F2 had been stored by Stantec (previously ESG), and was kindly made available by Stantec for the current work.

The Orthic Black Chernozem soil was also kindly made available by Stantec from supplies remaining from the original PHC CWS work. It was considered important to use the same soil and F2 distillate for this aged/weathered F2 project that were used to generate the fresh F2 ecotoxicity data for the original and current PHC CWS.

3.3 Rangefinding Test

Key parameters of the experimental design for the rangefinding test are summarized below.

- F2 was spiked into Orthic Black Chernozem at the following initial concentrations:
    - 3,000 mg/kg;
    - 10,000 mg/kg; and,
    - 30,000 mg/kg.
- An additional sample with 10,000 mg/kg F2 was brought to an electrical conductivity (EC) of 40 dS/m with the intention of sterilizing the sample.
- Once a week, soils were spread out, mixed and returned to the sample container. If necessary, water was added to maintain moisture content.
- Samples were analyzed for F2 to F4 hydrocarbons at the time of spiking (time zero) and after 1, 2, 3, 4, 5, 6, 7, 10, and 12 months. The time zero analysis was performed in triplicate for each concentration, subsequent analyses were single samples only.
- Four different analytical techniques were used to determine the F2 to F4 hydrocarbons. Either the standard Soxhlet extraction (CCME, 2001) or a shake flask procedure was used. Each extraction was used with and without the silica gel clean-up step.
- Other analyses performed included benzene, toluene, ethylbenzene and xylenes (BTEX) and F1, heterotrophic place count and hydrocarbon utilizing bacteria.

3.4 Ecotoxicity Tests

The results of the rangefinding test were used to estimate a series of initial F2 concentrations that would, after aging/weathering, become a suitable series of concentrations for conducting ecotoxicity tests. Details of the methodology for spiking and aging/weathering the soils, and of the ecotoxicity tests themselves are in Stantec (2009), which is available on the PTAC website. Key points are summarized here.

- F2 was spiked into Orthic Black Chernozem at the following initial concentrations:
    - 0 mg/kg (control)
    - 100 mg/kg;
    - 250 mg/kg;
    - 500 mg/kg;
    - 1,000 mg/kg;

    - 2,000 mg/kg;
    - 4,000 mg/kg;
    - 8,000 mg/kg;
    - 16,000 mg/kg;
    - 32,000 mg/kg; and,
    - 64,000 mg/kg.
- Each sample was hydrated to 35% water holding capacity, mixed vigorously and returned to its bucket on a biweekly basis for the first month, and weekly thereafter.
- After six months of this aging/weathering procedure, ecotoxicity testing was initiated.
- The battery of ecotoxicity tests conducted with the aged/weathered F2 soils was the same as that used with fresh F2 for the PHC CWS (ESG, 2003a), and included the following species/endpoints:
    - Earthworm (*Eisenia andrei)* – 35 day adult survival.
    - Earthworm (*Eisenia andrei)* – 63 day reproduction endpoints.
    - Springtail (*Folsomia candida)* – 28 day adult survival.
    - Springtail (*Folsomia candida)* – 28 day reproduction endpoints.
    - Northern wheatgrass (*Agropyron dasystachyum)* – 21 day growth endpoints.
    - Alfalfa (*Medicago sativa)* – 21 day growth endpoints.
    - Barley (*Hordeum vulgare)* – 14 day growth endpoints.
- Ecotoxicity tests were conducted according to Environment Canada protocols (EC 2004, 2005a,b).
- Chemical analysis for F2 was performed at various intervals throughout the aging/weathering process and in triplicate at the start of the ecotoxicity tests.

4. RESULTS

4.1 Rangefinding Test

The measured F2 concentrations from the rangefinding test, as a function of time, are summarized in Table 1.

Table 1. Measured F2 Concentrations in Rangefinding Test

	Initial (Nominal) F2 Concentration (mg/kg)		
	3,000	**10,000**	**30,000**
Time, Months			
0	2,323	8,653	26,133
1	1,710	5,690	16,100
2	1,420	5,620	19,400
3	1,120	5,490	12,900
4	1,470	4,710	19,900
5	870	3,900	15,900
6	750	4,160	13,300
7	670	3,350	8,960
10	370	2,190	13,900
12	300	1,120	4,280

Notes:
Where multiple analyses are available, the mean value is shown.

These data are presented graphically in Figure 1, with the time on the x-axis and the natural log (ln) of the F2 concentration on the y-axis. As can be seen, the data for each time series is a reasonable fit to a linear trendline, and therefore the three time series data sets approximate first order decay. The slope of each time series (indicated on Figure 1) is therefore the negative of the first order decay constant (λ), and the half life is ln(2)/λ. Thus the estimated half life for F2 with each nominal (starting) concentration is as follows.

- Nominal 3,000 mg/kg: half life = 4.1 months.
- Nominal 10,000 mg/kg: half life = 4.8 months.
- Nominal 30,000 mg/kg: half life = 6.6 months.

This suggests that there may be increasing inhibition of biodegradation at increasing F2 concentrations.

No distillation of a mixture of chemicals can be perfect. Most of the other analyses performed were intended to determine whether the amounts of compounds heavier and lighter than F2 were present in high enough concentrations to be significant. BTEX analysis indicated that there were small amounts of BTEX in the F2 distillate, about 0.2% of total F2, but that the BTEX had essentially disappeared by month 7. F1 concentrations were 1% or less of total F2, and thus not expected to be significant. F3 was present at up to about 10% of the F2 concentration. F4 concentrations were negligible.

Analysis of bacterial parameters (heterotrophic plate counts and hydrocarbon utilizing bacteria) were quite variable from measurement to measurement and did not appear to add useful data. Bacterial counts did not seem to be significantly different for the “sterilized” and unsterilized 10,000 mg/kg tests, indicating that using a salinity of EC = 40 dS/m does not appear to have been effective for bacterial sterilization.

The different extraction and clean-up methodologies used for F2 to F4 analysis showed significant variability. In most case the relative percent difference between corresponding values was less than 50%, but in a few cases was as high a 100%. The “shake flask” analytical technique was generally the closest to the nominal value for time zero data, and accordingly, this is the value presented in Table 1 and Figure 1.

4.2 Ecotoxicity Tests

The results of the study to generate the concentration series of F2 spiked soils and the results of the ecotoxicity testing are provided in detail in Stantec (2009), available on the PTAC website. Key points are summarized here, and data analysis is provided in Section 5. Triplicate samples were collected from each concentration at the start of the tests, and Table 2 summarizes the mean measured concentration for each nominal (initial) concentration. The original laboratory reports for these data are available in Stantec (2009).

Figure 2 shows the relationship between measured and nominal concentrations graphically. As with the rangefinding test, degradation of F2 progressed more rapidly with lower initial concentrations relative to higher concentrations.

However, it is noted that the percentage of F3 (<1% of total F2) is much lower in this test than in the rangefinding test, presumably indicating a cleaner F2 distillate.

Table 2. F2 Measured vs. Nominal Concentrations – Start of Ecotoxicity Test

Nominal F2 Concentration (mg/kg)	Measured F2 Concentration (mg/kg)
0	<10
100	<10
250	<10
500	<10
1,000	78
2,000	249
4,000	715
8,000	1,557
16,000	6,007
32,000	18,233
64,000	26,800

Notes:
Measured concentrations are the mean of three values

The results of the ecotoxicity testing program are summarized in Table 3. Stantec (2009) presented their results both in terms of nominal concentrations, and in terms of measured concentrations. Only the data based on measured concentrations are presented in Table 3. Emergence data are not included as this endpoint typically has a low sensitivity to contaminants.

Table 3. Ecotoxicity Testing Results – Based on Measured Concentrations

Species	Endpoint	IC25 (mg/kg)	IC50 (mg/kg)
Northern Wheatgrass	Shoot Length	647	1,183
Northern Wheatgrass	Shoot Dry Mass	199	437
Northern Wheatgrass	Root Length	295	767
Northern Wheatgrass	Root Dry Mass	290	942
Alfalfa	Shoot Length	355	800
Alfalfa	Shoot Dry Mass	265	492
Alfalfa	Root Length	258	550
Alfalfa	Root Dry Mass	403	659
Barley	Shoot Length	1,200	2,366
Barley	Shoot Dry Mass	418	1,047
Barley	Root Length	522	986
Barley	Root Dry Mass	493	1,035
E. andrei (earthworm)	Adult Survival	NA	1,202
E. andrei (earthworm)	Progeny Production	139	248
E. andrei (earthworm)	Progeny Dry Mass	293	395
F. candida (springtail)	Adult Survival	NA	1,230
F. candida (springtail)	Progeny Production	533	905

5. DATA ANALYSIS

Based on the latest protocol developed by CCME (2008) and adopted by AENV (2009), soil remediation guidelines for the protection of ecological direct contact are preferably developed from a species sensitivity distribution. Available data are standardized at, or as close as possible to the 25th percent effects level. A minimum of 10 independent data points are required.

Selected ecotoxicity data, ranked by their value, are summarized in Table 4, and the corresponding species sensitivity distribution is illustrated in Figure 3.

Table 4. Ranked Selected Ecotoxicity Data – Based on Measured Concentrations

Species	Endpoint	Selected Value * (mg/kg)
E. andrei (earthworm)	Progeny Production	139
Northern Wheatgrass	Shoot Dry Mass	199
Alfalfa	Root Length	258
Alfalfa	Shoot Dry Mass	265
Northern Wheatgrass	Root Dry Mass	290
E. andrei (earthworm)	Progeny Dry Mass	293
Northern Wheatgrass	Root Length	295
Alfalfa	Shoot Length	355
Alfalfa	Root Dry Mass	403
Barley	Shoot Dry Mass	418
Barley	Root Dry Mass	493
Barley	Root Length	522
F. candida (springtail)	Progeny Production	533
Northern Wheatgrass	Shoot Length	647
Barley	Shoot Length	1,200
E. andrei (earthworm)	Adult Survival	1,202
F. candida (springtail)	Adult Survival	1,230

Notes:

** Selected values is IC25 where available, IC50 used for the two invertebrate survival endpoints*

Following the convention in CCME (2008), shoot length, root length, shoot dry mass and root dry mass are considered independent endpoints for each species tested. Emergence data are not considered, since emergence is often a poor indicator of toxicity. Wet mass measurements are considered not considered to be independent from corresponding dry mass measurements. For earthworms, the number of progeny and the progeny mass are considered to be independent endpoints.

IC25 values, based on measured data, are used for all species/endpoint combinations except for the invertebrate survival endpoints. In these cases, IC25 values could not be calculated, and the IC50 values were used.

The CCME (2008) and AENV (2009) protocols, taken together, indicate that the soil remediation guidelines for ecological direct contact for natural areas, agricultural and residential land use are calculated as the 25th percentile of the species sensitivity distribution indicated above. The soil remediation guidelines for ecological direct contact for commercial and industrial land use are calculated as the 50th percentile of the species sensitivity distribution.

Thus the soil remediation guidelines for weathered F2 in fine soil are:

- 290 mg/kg – natural area, agricultural, and residential land use.
- 400 mg/kg – commercial and industrial land use.

6. CLOSURE

The information presented in this report was compiled and interpreted exclusively for the purposes stated in Section 1 of this document. Axiom Environmental Inc. (Axiom) provided this report for the Petroleum Technology Alliance of Canada (PTAC) solely for the purpose noted above.

Axiom has exercised reasonable skill, care and diligence to assess the information acquired during the preparation of this report, but makes no guarantees or warranties as to the accuracy or completeness of this information. The information contained in this report is based upon, and limited by, the circumstances and conditions acknowledged herein, and upon information available at the time of its preparation. Guidelines developed in this report are based on current regulatory protocols. The information provided by others is believed to be accurate but cannot be guaranteed.

Axiom does not accept any responsibility for the use of this report for any purpose other than that stated in Section 1 and does not accept responsibility to any third party for the use in whole or in part of the contents of this report. Any alternative use, including that by a third party, or any reliance on, or decisions based on this document, is the responsibility of the alternative user or third party.

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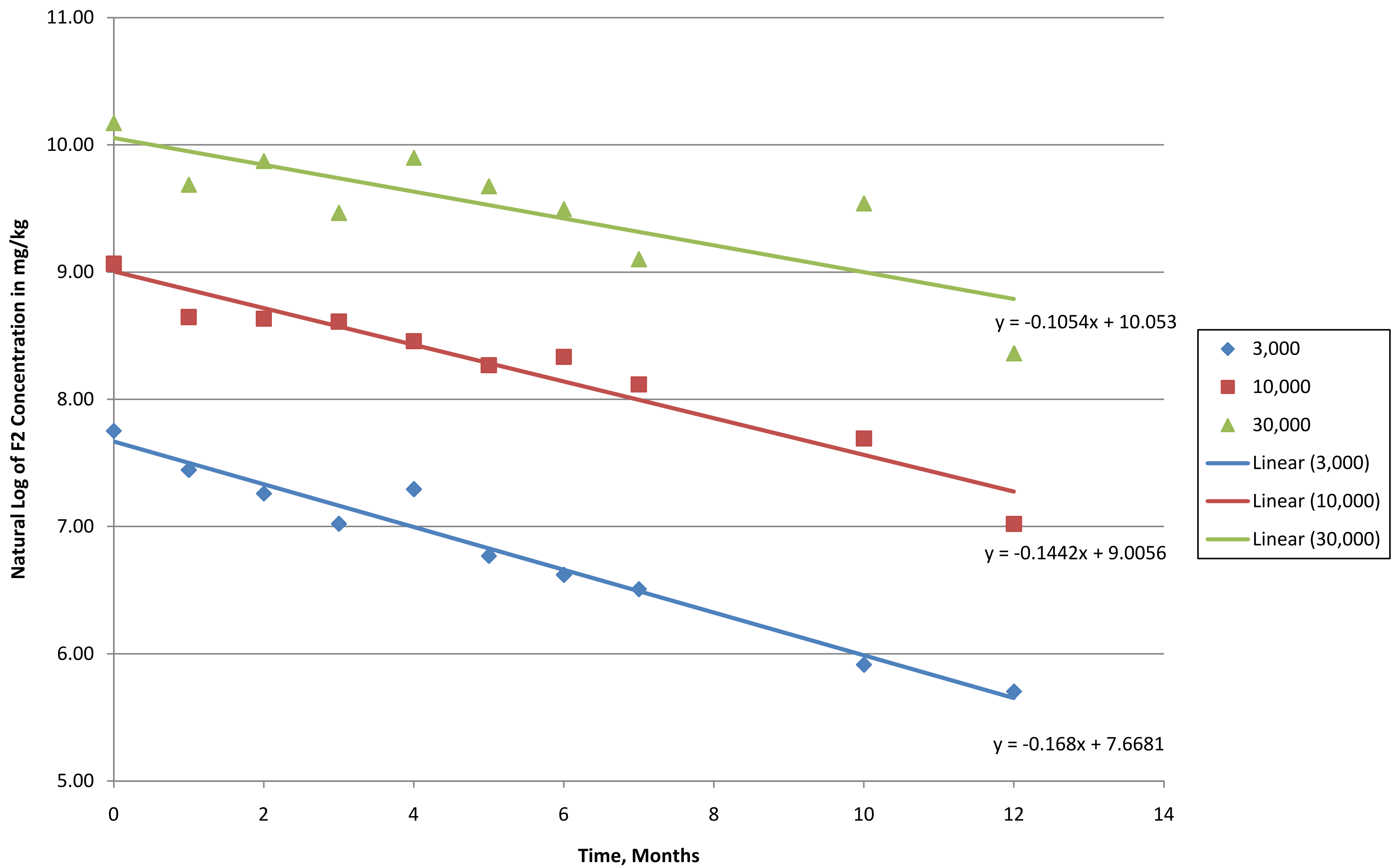
FIGURES

Figure 1. Degradation Kinetics - Rangefinding Test (Log-Linear)



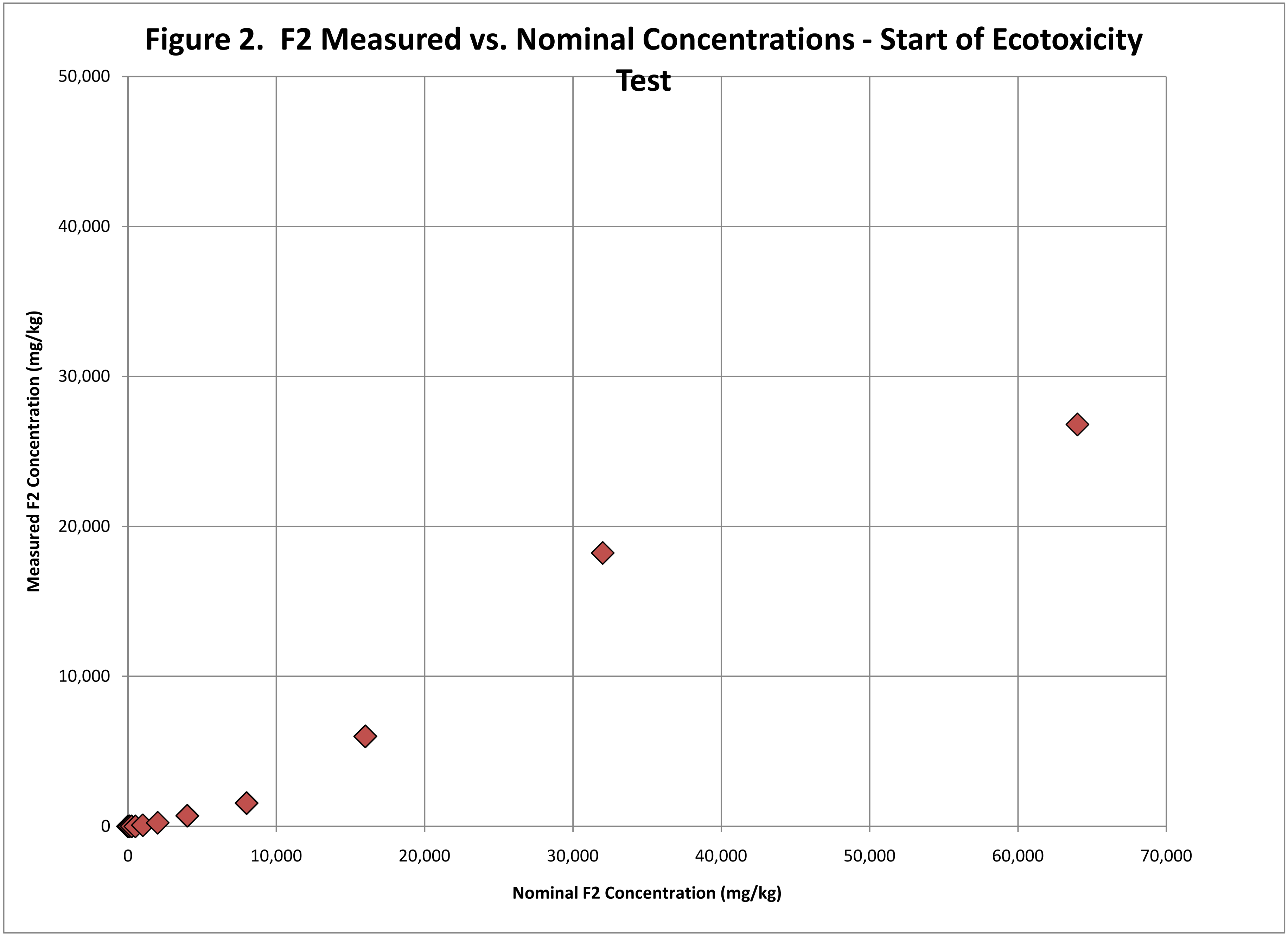


Figure 2. F2 Measured vs. Nominal Concentrations - Start of Ecotoxicity Test
Measured F2 Concentration (mg/kg)
50,000
40,000
30,000
20,000
10,000
0
0
10,000
20,000
30,000
40,000
50,000
60,000
70,000
Nominal F2 Concentration (mg/kg)

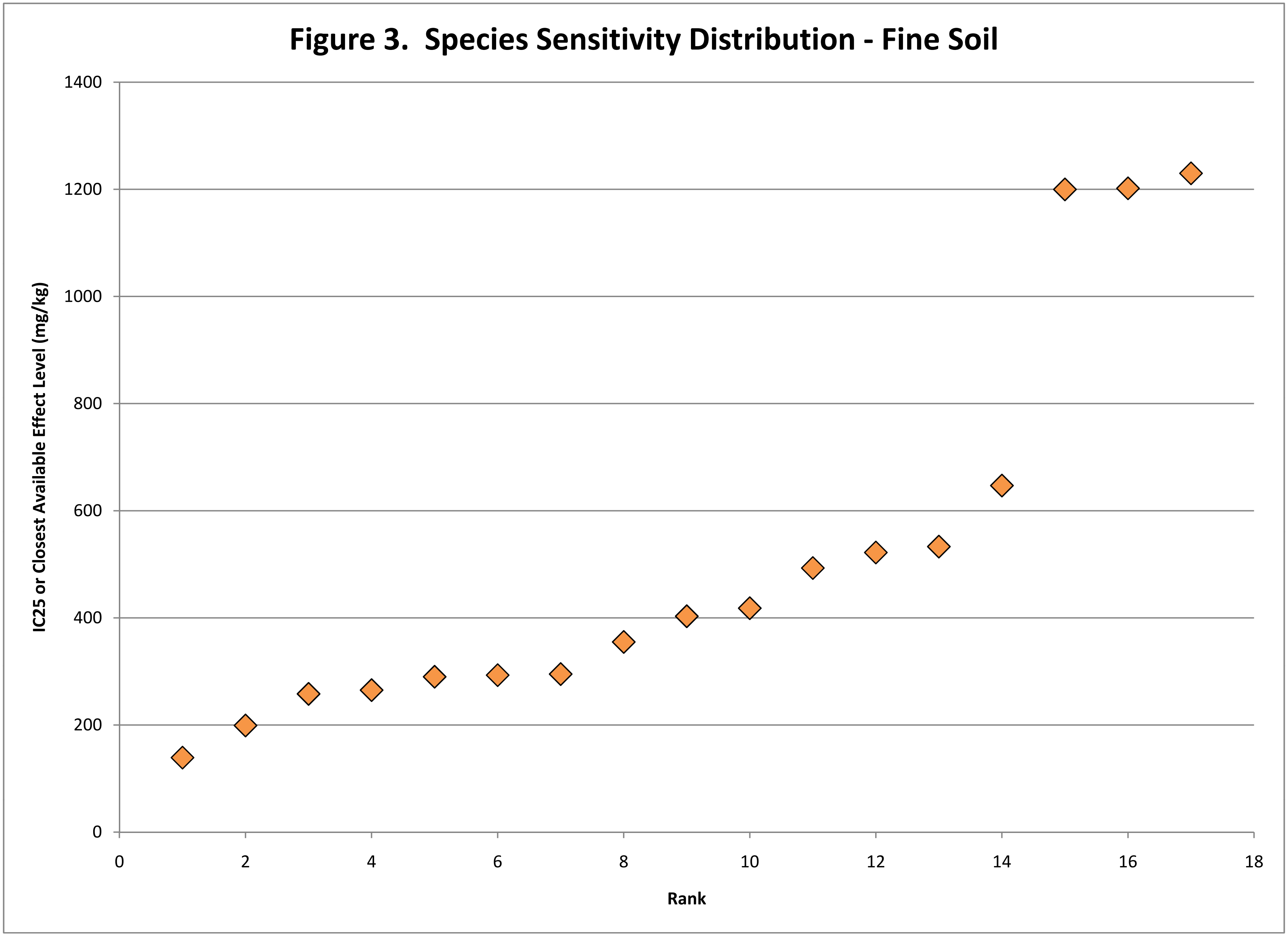


Figure 3. Species Sensitivity Distribution - Fine Soil
IC25 or Closest Available Effect Level (mg/kg)
0
200
400
600
800
1000
1200
1400
0
2
4
6
8
10
12
14
16
18
Rank