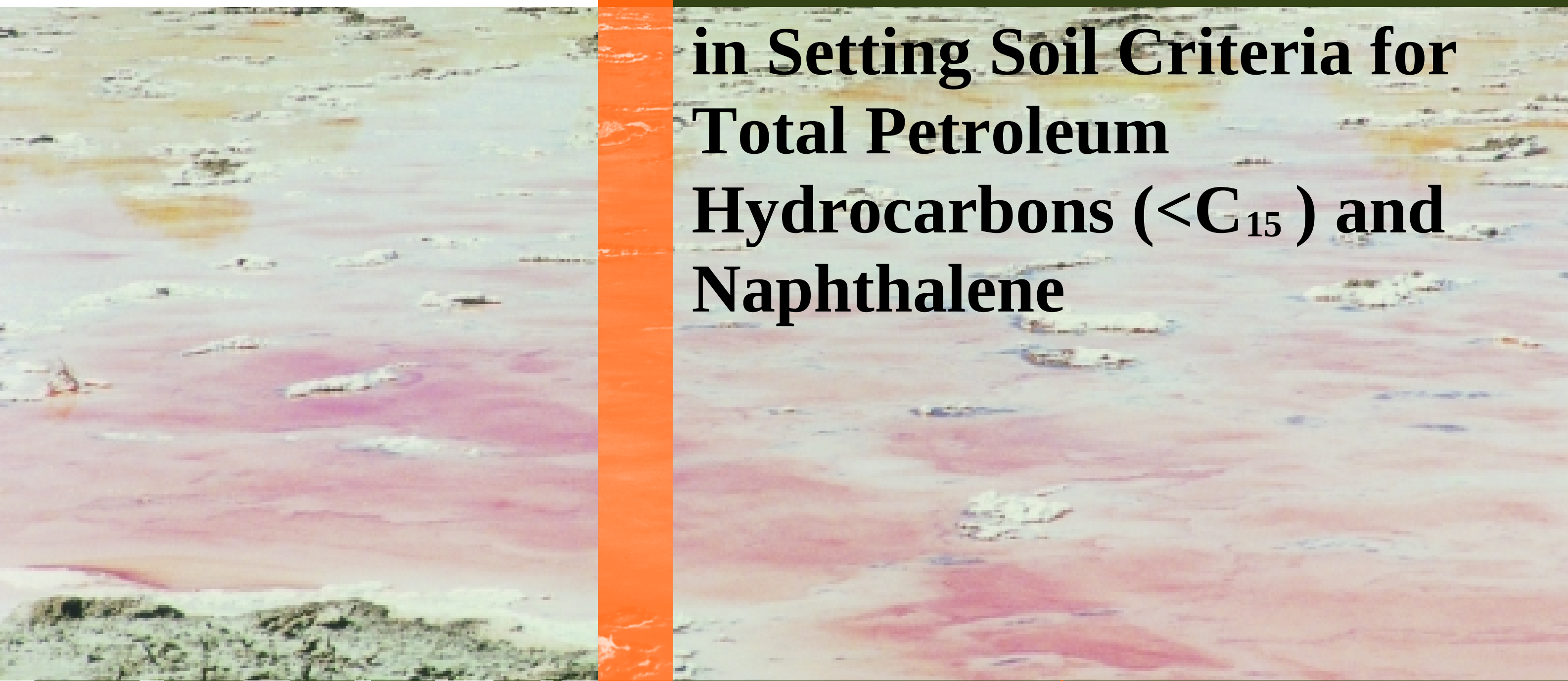
Ecological Considerations in Setting Soil Criteria for Total Petroleum Hydrocarbons (<C_{15}) and Naphthalene



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Ecological Considerations in Setting Soil Criteria for Total Petroleum Hydrocarbons (<C_{15}) and Naphthalene

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1 INTRODUCTION

Ecological considerations in setting soil criteria for total petroleum hydrocarbons (<C_{15}) and naphthalene are discussed from three perspectives:

- general approaches to setting soil criteria
- a review of published ecotoxicological and related information for TPH and naphthalene
- application of the general approaches in the context of TPH and naphthalene.

The purpose of this paper is to encourage discussion issues in establishing soil criteria. This paper does not purport to establish soil criteria for TPH or naphthalene. At present there is uncertainty as to whether there is sufficient reliable information to base such criteria, if delivered in a manner analogous to that used in the setting of soil investigation levels for other contaminants.

The general tiered or staged approach to the assessment of ecological risk has been adopted for the purpose of this paper. The approach set out in this paper may be best regarded as a risk-based assessment, rather than a rigorous quantitative ecological risk assessment. In a manner consistent with the recently published Australian and New Zealand Guidelines for Fresh and Marine Water Quality (Water Quality Guidelines) (ANZECC 2000), a risk-based decision framework is proposed as a means by which assessment of ecological impacts may be considered as part of a site-assessment.

Two key assumptions have been used to assist in narrowing the scope for this paper:

- Only ecological considerations relevant in the context of single-dwelling residential or parkland use are considered (ie more sensitive uses such as National Parks are excluded)
- TPH as an analytical measure has been confined to petroleum hydrocarbons associated with petrol and diesel only.

These assumptions reflect one of the most common problems faced in contaminated site assessment. The general approaches set out in the paper may be applicable to other petroleum mixtures supported by review of relevant toxicological data.

2 APPROACHES TO SETTING ECOLOGICALLY BASED CRITERIA

2.1 OVERVIEW OF EXISTING APPROACHES

2.1.1 Australian Approaches

National Environment Protection (Assessment of Site Contamination) Measure

The National Environment Protection (Assessment of Site Contamination) Measure 1999, Schedule B(5) Guideline on Ecological Risk Assessment (NEPC 1999), provides a broad framework for the assessment of ecological risk in Australia and identifies the key elements of a risk-based decision making framework.

Some of the key elements of the framework include:

1. A tiered-approach in which ecological investigation levels provide a trigger for more detailed site-specific consideration
2. Identification of ecological values or "what do we want to protect?" in relation to the ecosystem
3. Consideration of the potential of contamination to affect other media and cause ecological effects.

Draft National Ecological Risk Assessment Framework

The Draft National Framework for Ecological Risk Assessment of Contaminated Sites (EPA Victoria 1997) (Draft National Framework) is comprised of 3 parts; Part 1 – overall framework, Part 2 – detailed methodology for calculating ecological investigation levels (including site-specific levels) and Part 3 – background data.

Part 1 is incorporated in the NEPM to provide the overall direction and approach to ecological risk assessment, but does not specify a method for calculating ecological investigation levels.

Part 2 presents a detailed methodology for deriving ecological investigation levels from toxicological data, allowing users to modify any generic published EILs to account for site-specific conditions. The method involves:

1. a review of the relevance and strength of available toxicological data
2. consideration of direct toxicological effects through the use of either the lowest or median published no effect level, with relevant safety factors. Where necessary, equations are presented to assist in estimating exposure (eg through ingestion of contaminated soil or water)
3. consideration of secondary poisoning and similar food chain effects through application of equations to predict bioconcentration, and to estimate exposure through consumption of contaminated food sources.

The above method is applied in a generic manner to derive regional EILs, and the same methodology can be used to derive site-specific EILs by incorporating available site-specific information.

Australian and New Zealand Guidelines for Fresh and Marine Water Quality

The Water Quality Guidelines (ANZECC 2000) adopt a risk-based approach to the assessment of water quality. Key features include:

1. Definition of the management objectives for a particular surface water body;
2. A risk-based decision making framework, to assist in determining actions;
3. A tiered approach in which comparison with general trigger levels is part of the first step, but where there is scope to then:
   - consider the specific needs of the particular water body (eg what species are present requiring appropriate levels of protection)
   - apply more sophisticated assessment methods such as biological monitoring

In establishing trigger levels, consideration is given to the ecosystem type and the level of protection desirable. Where sufficient toxicological information is available the Water Quality Guidelines adopt a method for derivation of trigger levels based on a modification of the Aldenberg and Slob method used by The Netherlands in defining Intervention and Target Levels (Aldenberg and Slob 1993 in ANZECC 2000: Section 8.3.4). This method fits the available toxicological endpoint data to a log logistic distribution. The distribution is then used to define the concentration of the contaminant assumed to protect a certain proportion of the species with a particular level of confidence. The proportion of species and level of confidence are policy decisions – for different circumstances values of 50%, 75%, 90%, 95% and 99% have been used. This approach does not explicitly address issues of secondary poisoning or similar food chain effects although adoption of a guideline value based on protection of a higher proportion of species is recommended.

2.1.2 Other Approaches

Most jurisdictions use similar risk-based approaches when setting ecological soil criteria (see 1.1.3). However the data requirements and methods used to determine trigger or screening levels differ in each jurisdiction.

The Netherlands

The Dutch use NOEC[1] data for deriving their Ecological Serious Risk Concentrations (SRC_{eco}) and Maximum Permissible Concentrations (MPC). These are used together with SRC_{human} to derive Intervention and Target Values. As outlined for the Water Quality Guidelines, a statistical distribution is fitted to the available toxicological data. Various guideline values are then defined based on protection of certain proportions of species with a specified level of confidence.

Where insufficient terrestrial data exist, (less than 4 taxonomic groups), aquatic data is utilised by applying the equilibrium partitioning method. This method assumes that toxicity, bioavailability and bioaccumulation are closely correlated to the pore water concentrations and that the sensitivity of aquatic organisms is comparable to the sensitivity of terrestrial organisms. The Dutch have set SRC_{eco} and MPC for naphthalene but not for TPH. (See Verbruggen *et al* 2001, and Ecological Planning and Toxicology 1999 section 2.1).

USEPA

Ecological soil screening levels (EcoSSL) are derived through a review of the relevant literature. Studies are evaluated in terms of certain quality criteria, including soil organic

[1] No observed effect concentration

content (<10%) and the bioavailability of the contaminant. EC_{20}[2], EC_{10}, NOEC and LOEC[3] data are acceptable, however acute LC_{50}[4], EC_{50} and EC_5 data are not used. EcoSSLs are derived based on the geometric mean of toxicity values (preferably EC_{20}) for studies selected based on the quality of the study and the bioavailability of the contaminant in the matrix. EcoSSLs have not been established for TPH and naphthalene (Swindoll 2002, and Ecological Planning and Toxicology 1999: section 2.8).

Canada

Canada has developed tier 1 soil quality levels for TPH depending on soil type (coarse/fine) and landuse (Agriculture/Industrial etc). In the development of these values the Canadian Council of Ministers of the Environment (CCME) used ECx or LCx data, where available, in preference to NOEC or LOEC data. Similarly data generated from longer exposure periods, where available for the same test, were preferred over shorter periods.

The effects-endpoints were standardised at or near the 50% response level and data was weighted according to the conditions and methods used. New toxicity data for specific TPH fractions was preferred over surrogate data (which was standardised to whole fraction data). If neither new toxicity data for specific TPH fractions nor surrogate data is available, available whole product toxicity data from controlled laboratory studies may be used.

The Tier 1 level for sensitive landuse (Agricultural/Residential) was derived from the 25th percentile of ECx/LCx toxicity data from plants and soil invertebrates. For less sensitive landuse (Industrial/Commercial) it was derived from the 50th percentile of the plant toxicity data set. (See CCME 2000, and Ecological Planning and Toxicology 1999: section 2.2).

2.1.3 Summary of Approaches

Common elements of risk-based approaches to assessing ecological risk include:

- screening criteria or trigger levels that are based on principles of ecological risk assessment, but do not entail a detailed ecological risk assessment
- tiered approach to developing a response in the case of screening criteria being exceeded
- stepping through the tiers, where required, involving incorporation of more site-specific information and in some cases increasing complexity
- decision framework to guide users through the more detailed assessment
- use of site-specific information, including soil type and properties, local ecosystem characteristics, and the results of toxicity and other testing to assist in refining ecological investigation levels and response levels.

Summary:
In developing soil criteria in Australia, it may be appropriate to adopt an approach based on the risk-based assessment process set out in the Water Quality Guidelines (ANZECC 2000). Some refinement should be considered to ensure applicability to soils.

[2] Concentration that affects x%.
[3] Lowest observed effect concentration
[4] Concentration that is lethal to x%.

2.2 IDENTIFYING ECOLOGICAL VALUES

The identification of the ecological values to be protected is a key first step in a risk-based approach to the assessment of ecological impacts. This is critical in translating general objectives such as "maintenance of ecosystems" into more specific outcomes that can then be used as a focus for review of literature and site information.

For the purpose of this paper, a conventional single dwelling residential context has been considered. This represents the most common sensitive site use when considering the effects of contamination, particularly petroleum hydrocarbon contamination (as a result of the redevelopment of service stations and industrial facilities for residential purposes).

Ecological values to be considered for a single dwelling residential may include:

- establishment and maintenance of decorative gardens containing a range of exotic and native species;
- establishment and maintenance of vegetable and fruit producing plants;
- protection of domestic animals;
- protection of wildlife that may in part live at, or visit, the site;
- maintenance of supporting ecological processes (eg as provided by earthworms and nutrient cycling bacteria).

In other contexts, other ecological values may also need to be considered, eg subterranean ecosystems may be of value for some sensitive uses.

3 SUMMARY OF TOXICOLOGICAL AND FATE INFORMATION FOR PETROLEUM HYDROCARBONS (<C15)

A summary of published information on the ecotoxicological effects of the petroleum hydrocarbons is presented below. Due to limitations in the available data, some data for crude products is included alongside data for the refined products of interest (petrol and diesel).

A discussion of the composition and fate and transport properties of petroleum hydrocarbon products of relevance in assessing the ecological impacts of these compounds is also presented.

3.1 NATURE OF THE MIXTURE

TPH is a non-specific laboratory method that detects a broad range of organic compounds within certain boiling ranges, eg some chlorinated solvents can be measured in the C_6 to C_9 TPH range.

For this reason, attention is focused only on TPH contamination derived from petrol and diesel – the most commonly encountered sources of TPH.

While this is useful in partially defining the problem being considered, petrol and diesel are both complex mixtures, which vary with:

- source – eg different additives (see Heath *et al* 1993).
- time – production techniques have changed over the years and new and different additives have been used eg lead vs manganese, use of MTBE.

- location – differences in petroleum composition occur with climate.
- weathering – once released to the environment a range of weathering processes alter the nature of the product eg a fresh sample may contain significant proportions of aliphatics, while a highly weathered sample of petrol may have lost much of the aliphatic component leaving behind a significant proportion of the highly substituted aromatic fraction (see Salanitro *et al* 1997).

Summary:
- Given the importance of the composition of the mixture to the resulting toxicological effects, the remainder of the paper focuses on petroleum mixtures derived from petrol and/or diesel spills.
- The composition of the mixture will vary with source and with time due to weathering – therefore any generic criteria can only be very approximate
- Weathering will alter the proportion of aliphatic and aromatic components and therefore there is a need to consider this on a site-by-site basis.
- For the purpose of this paper it is assumed that other specific additives (both current and historical) eg lead, are considered separately.

3.2 FATE AND TRANSPORT

3.2.1 Weathering

Salanitro *et al* (1997) found that 70-90% of the C_{11}-C_{22} fraction, 40-60% of the C_{23}-C_{34} and 35-60% of the C_{35}-C_{44} fraction of fresh crude oil were lost after 3-4 months of bioremediation in optimal conditions (note these values depended on the organic content of the soil). Similarly, under favourable conditions most components of petrol and diesel ($<C_{15}$) are lost by weathering processes, including biodegradation, transformation, volatilisation and leaching. However, such biodegradation may alter the relative proportion of hydrocarbon components. For example, a water-leached soil may be comparatively lower in water-soluble aromatic components. Similarly, a soil subject to intense biodegradation may be comparatively higher in less readily degraded components eg higher aromatic and aliphatic components. Such changes in residual composition are not identified by conventional TPH analytical methods, but these may be important in determining the extent of ecological impact.

Bioavailable petroleum hydrocarbons are unlikely to persist for a significant period of time (>years) in exposed surface soils, including near surface soils in which there is significant biological activity eg garden soils. Therefore the degree of weathering is relevant to accessing assessing ecological impact, and thus weathered fresh and weathered contamination may need to be considered separately. While both petroleum contamination are relevant problems, weathered contamination is more commonly encountered in contaminated site work.

Less weathered TPH contamination may persist at depth (particularly beneath formerly paved service stations etc), remote from the most significant effects of weathering, and the concentrated root zone. While plants may establish in surface soils under these conditions, the potential for effects may remain when roots reach such residual TPH. While the root zone of most grasses, shrubs, vegetables and herbaceous plants is limited to the upper 1m of soil, the roots of trees and woody plants can penetrate deeper. Studies on the effects of contamination at this depth to mature plants was not identified in the literature.

Summary:
Residual petroleum hydrocarbons in well weathered surface soils are likely to pose a relatively low risk of ecological impact on the basis that bioavailable components are likely to have already been lost to biodegradation. However, complexities in the fate and transport of petroleum hydrocarbons (eg persistence of lighter, toxic components within less available, high molecular weight hydrocarbon) mean that assumptions about bioavailability should be accompanied with reliable site-specific data to support such assumptions. Consideration should also be given to the potential ecological impacts arising from:

- deeper contamination – beyond the typical zone of weathering.
- off-site transport of petroleum hydrocarbons (eg by runoff or groundwater transport).

3.2.2 Availability

Any residual, generally higher molecular weight, TPH remaining after "weathering" under conditions favourable to biodegradation are unlikely to be bioavailable (see Salanitro *et al* 1997, Riis *et al* 1996, Rippen *et al* 1994, Dorn *et al* 1998, Alexander 1997 and Loehr *et al* 1997).

Higher molecular weight hydrocarbons are less available to organisms because they are less soluble in water and are more likely to sorb onto the soil. This is reflected by the lower toxicity of petroleum hydrocarbons in high organic/fine soils, which promote sorption of hydrocarbons (Salanitro *et al* 1997, Dorn *et al*, 1998).

Summary:
Where there is a need to rely on the reduced bioavailability of a petroleum hydrocarbon mixture, in most circumstances such reduced availability should be demonstrated through use of ecotoxicological tests or other site-specific information.

3.2.3 Bioaccumulation

In general the key petroleum products of interest in the TPH measurement of petrol and diesel are not expected to significantly bioaccumulate under most conditions, whether that be in plants, invertebrates or mammals.

There are very few studies that have investigated the uptake of petroleum hydrocarbons by plants. Chaineau *et al* (1997) found that plants do not uptake TPH at concentrations compatible with plant growth. In practice the rhizosphere is a zone of enhanced biological activity, which increases the rate of degradation of a range of contaminants, including petroleum hydrocarbons.

While petroleum hydrocarbons may bioconcentrate to a limited extent in earthworms and other invertebrates, higher animals (eg mammals) readily metabolise petroleum hydrocarbons.

Summary
For the purposes of this paper, bioaccumulation of petroleum hydrocarbons has been effectively ignored in setting ecologically-based soil criteria and in developing a risk-based decision framework.

3.3 ECOTOXICITY

The mechanisms of TPH toxicity are not well studied. Although the inherent toxicity of some compounds is a probable cause, the lack of water and nutrients in oily soils has also been found to be a significant factor in an organism's response (see Chaineau *et al* 1997 and CCME 2000). In addition petroleum hydrocarbons may affect the lipid structures within plant and other cells if they are not metabolised quickly.

A broad range of biological assays has been established to determine the level of toxicity of chemical contaminants to terrestrial species (for example see guidelines and protocols set out by OECD and USEPA). However the relevance of these tests in assessing impacts on Australian native species may be low (O'Halloran *et al* 2000). Both exotic and native ecotoxicity tests need to be selected or developed for assessing the potential for ecological impact under Australian conditions, in the context of residential landuse.

The toxicity of petroleum hydrocarbons in soil has been studied using a range of species, including bacteria, algae, earthworms and plants, and a range of lethal and sub-lethal effects, such as seed germination, root elongation, and reproduction. No studies on native Australian species were identified in the literature.

Most of the published literature has focused on whole product toxicity, with the majority using crude oil. Toxic effects have appeared over a large range of TPH concentrations. However, most effect concentrations are >1000mg/kg (see Table 1). The lowest EC_{50}s for seed (typically lettuce) germination studies are in the range of 2000-3000mg/kg (Chaineau *et al* 1997 and Saterbak *et al* 1999).

The low molecular weight aromatic component is likely to be a significant contributor to the toxicity of these mixtures (see Henner *et al* 1999, Chaineau *et al* 1997 and CCME 2000). Chaineau *et al* (1997) found that partially de-aromatised fuel oil was significantly less toxic to all crops except for the most sensitive species. Similarly Chaineau *et al* (1997) found that napthas (the light fraction) were 20 times more toxic than the heavier fraction. Individual polycyclic aromatic hydrocarbons (PAHs) concentrations that are toxic to soil-dwelling species are in the range 20-100mg/kg (Mitchell *et al* 1998 quoted in WHO 1998:620, Maliszurska-Kordybach *et al*, and Sverdrup *et al* 2002). This is around 40 times more toxic than the whole product toxicity of TPH, which is consistent with the naphthalene component of diesel, which is in the order of 3% (see Potter *et al* 1998).

Very limited published terrestrial ecotoxicological data has been identified for the C_6-C_9 fraction including benzene (see CCME 2000). This is in part due to limitations in the application of the ecotoxicity tests for volatile and rapidly degradable compounds[5].

Although there is limited evidence in the literature for a reduction in the toxicity of aged (weathered) contamination[6] (see CCME 2000 and Table 1), bioremediated soils have been found to exhibit significantly higher effects concentrations (see Salanitro *et al* 1997, Riis *et al* 1996, Rippen *et al* 1994, Loehr *et al* 1997 and Henner *et al* 1999). Residual TPH

[5] The rapid loss by volatilisation and biodegradation also reduces the risk of persistent ecological effects.

[6] Note this may be due to the methods by which 'fresh' soils are tested leading to artificial weathering anyway. The Canadian values are lower because their values are based on the initial or expected initial exposure concentrations not the concentration with which the soil was spiked as in the other 'fresh' results.

concentrations in the range of 10,000mg/kg have been found to be non-toxic (see Salanitro *et al* 1997 and Loehr *et al* 1997). In summary Alexander (1997:23) writes,

> "The results have shown that: (1) hydrocarbons are degraded by indigenous soil microorganisms to a concentration which no longer decreases, or which decreases very slowly, with continued treatment, (2) reductions below this concentration are limited by the availability of the hydrocarbons to the microorganisms, (3) the residual hydrocarbons that remain after biological treatment, regardless of the extent of treatment, are significantly less leachable (in water) and significantly less available to other organisms as measured by simple indicator toxicity tests such as earthworm mortality and Microtox®, and (4) the aged hydrocarbons in soil are less available to many organisms, resulting in less exposure and reduced toxicological effects, and are less prone to leaching compared to hydrocarbons that are freshly added to soils."

Soil properties may also affect the concentration at which certain ecotoxicological effects may be noted. Conditions conducive to biological activity are likely to result in a lower proportion of bioavailable components in the residual hydrocarbons present in the soil – such soils are then likely to report ecotoxicological endpoints at correspondingly higher concentrations.

Similarly, high organic carbon content will increase the sorption of most hydrocarbon compounds, reducing the proportion available to plants etc. in soil water. For example Salanitro *et al* (1997) and Dorn *et al* (1998) found soils with high organic content reported toxic endpoints at concentrations at least double, and up to eight times, that of soils with low organic content. Environment Canada found hydrocarbon contamination in fine soils is 2-6 times less toxic (higher endpoint concentrations) than contamination in coarse soils for similar reasons (CCME 2000).

Table 1: Selected range of effect concentrations of TPH

Product	Age	Endpoint	Value	Comment	Source
Plants					
Fuel oil – light fraction	F[7]	Seed germination LC_{50}[8]	3000mg/kg fuel oil in sand[9]	Maize	Chaineau *et al* (1997)
Fuel oil heavy fraction	F	Seed germination LC_{50}	60,000mg/kg fuel oil in sand	Maize – compare to above.	Chaineau *et al* (1997)
Fuel oil	F	Seed germination LC_{50}	3000mg/kg – 70,000mg/kg fuel oil in sand	7 crops (most sensitive lettuce, least sunflower)	Chaineau *et al* (1997)
Diesel Fuel	F	Seed germination LC_{50}	25,000mg/kg fuel in sand	9 species of grasses	Adam and Duncan (1999)
Diesel Fuel	F	Seed germination LC_{50} for 50% of the species	50,000mg/kg fuel in sand	22 species of grasses legumes, and commercial crops	Adam and Duncan (1999)

[7] Fresh contamination.
[8] 50% Reduction in germination compared to the control.
[9] Note that this is not a concentration of TPH as measured by any of the laboratory methods but is the amount of fuel oil added.

Product	Age	Endpoint	Value	Comment	Source
Light Crude oil	F	Significantly reduced growth – (20-70%)	4200-26,600mg/kg TPH-GC[10]	Wheat and oats. More sensitive but more variable than seed germination.	Salanitro *et al* (1997)
Fraction C_{10}-C_{16} (F2) Fresh		25TH percentile (LC)EC_{50}	600mg/kg (1,800mg/kg) [11]	Based on numerous plants and varying endpoints (see sample results below).	Stephenson *et al* (2000) in CCME (2000)
		21 day Dry weight[12] EC_{50}	450mg/kg (1,370mg/kg)	Alfalfa shoot – one of the lowest EC_{50}s	
		14 day EC_{50}	450mg/kg (1,370mg/kg)	Northern Wheatgrass – one of the lowest EC_{50}s	
Fraction C_6-C_{10} (F1) (Mogas) Fresh		25th percentile (LC)EC_{50}	165mg/kg (1,700mg/kg)	Based on numerous plants and varying endpoints	Stephenson *et al* (2000) in CCME (2000)
		21 day Dry weight EC_{50} (artificial soil)	270mg/kg (2,520mg/kg)	Alfalfa shoot – one of the lowest EC_{50}s	
		Wet mass 13 day[13] EC_{50} (sandy loam soil)	72mg/kg (870mg/kg)	Barley root – one of the lowest EC50s	
Microalgae					
Diesel oil	W[14]	Biomass and enzyme activity NOEC	<2,120mg/kg TPH-GC	The C_9-C_{14} fraction had <31mg/kg and <C_9 was not present	Megharaj *et al* (1999)
Soil Invertebrates (Earthworms)					
Light Crude oil	AW[15]	Survival LC_{50}	42-96mg/kg TPHGC	Depended on the organic content of the soil.	Salanitro *et al* (1997)
Crude oil	W	Survival NOEC	1000mg/kg TPH-GC	Generally non-toxic above 4000mg/kg	Wong *et al* (1999), Saterbak *et al* (1999)

[10] TPH measured by Gas Chromatography.
[11] The nominal concentration, in the brackets, is the concentration added to the soil however the concentrations used by Canada are the "initial" concentrations. Initial concentrations are either measured or predicted concentrations of the contaminants at the beginning of the test.
[12] 50% reduction in dry weight.
[13] 50% reduction in wet mass.
[14] Weathered.
[15] Artificially weathered (contamination added and mixed into soils).

Product	Age	Endpoint	Value	Comment	Source
Fraction C_{10}-C_{16} (F2) -Fresh		25[th] percentile (LC)EC_{50}	200mg/kg (600mg/kg)	Based on a number of soil invertebrates (sample results below).	Stephenson *et al* (2000) in CCME(2000)
		No. of juveniles 63 day EC_{50}	160mg/kg (490mg/kg)	E. foetida *(earthworm)*	
		LC_{50}	170mg/kg (530mg/kg)	E. foetida	
Fraction C_6-C_{10} (F1) (Mogas) Fresh		25[th] percentile (LC)EC_{50}	75mg/kg	Based on a number of soil invertebrates (sample results below).	Stephenson *et al* (2000) in CCME(2000)
		14 day LC_{50}	56mg/kg (710mg/kg)	E. foetida	
		No. of Juveniles EC_{50} (artificial soil)	320mg/kg (2,890mg/kg)	O. folsomi (Springtail)	
Microtox®					
Light Crude oil	F	LC_{50}	10,000mg/kg-27,000mg/kg oil in sand	Depended on the organic content of soil.	Dorn *et al* (1998)
Light Crude oil	F	LC_{50}	>2000mg/kg TPH-GC	Converted from % contaminated soil	Salanitro *et al* (1997)

Limitations of published data

The applicability of reported toxicological data is limited for many reasons:

1. TPH is not an identified chemical, but is defined by and varies with the analytical technique used to measure it. Furthermore there is limited correlation between the concentrations measured by different techniques. For instance, although Saterbak (*et al* 1999) and Wong (*et al* 1999) found reasonable correlation between TPH determined by GC (C_6-C_{25}) and earthworm survival, freon-extractable TPH and oil and grease concentrations did not correlate strongly with earthworm survival.
4. The toxicity of TPH depends on its composition, which in turn depends on its source and environmental transformations. Without knowledge of the source or composition, TPH does not indicate the ecological risk of toxicity. As a result, this paper focuses on TPH associated with petrol and/or diesel.
5. Non-weathered petroleum products have little predictability for the majority of weathered "aged" spill sites (see 2.2.1).
6. Very limited data is available for the more volatile components. This reflects both limitations in the nature of ecotoxicological tests when applied to volatiles and in assumptions about the long-term significance of the volatile component. The soil toxicity tests cannot expose the plants continuously because the volatile and degradable fraction is lost, however in the field plants can be exposed continuously because pollutants will be supplied from the air and/or deeper soil layers (Hulzebos *et al* 1993). Stephenson *et al* (in CCME 2000) modified their data to take this into account. The initial concentration of TPH was measured during the test and is used in preference over the nominal or spiked concentrations. The initial concentration for C_6-

C_{10} fraction was <10% of the nominal concentration whilst for C_{16}-C_{34} it was up to 65% (CCME 2000:Section 4.2.5).

7. A small number of laboratories have contributed the majority of the published ecotoxicological data. Some unpublished data is available from other laboratories however this is more variable in quality.
8. Much of the ecotoxicological data published presents EC_{50} data and similar endpoints, rather than no effect levels.
9. As with most other published ecotoxicological data, there is little or no information on possible synergistic or antagonistic effects with other contaminants. However, to the extent that published data are available for TPH mixtures, synergistic and/or antagonistic effects between components of the TPH mixture are accounted for.

The large number of variables means a simple application of a generic criterion is likely to be very uncertain. Ecotoxicological tests can provide assistance in providing a more realistic assessment of the potential for ecological impact (see Bispo *et al* 1999, Dorn *et al* 1998, Marwood *et al* 1998, Baud-Grasset *et al* 1993, Salanitro *et al* 1997, Megharaj *et al* 2000, and Liptak *et al* 1996).

Summary:
Notwithstanding the above limitations, based on the collected ecotoxicological data, it is likely that screening or trigger levels of the following order:

- C_6 to C_9: 100-500 mg/kg
- C_{10} to C_{15}: 500-2000 mg/kg

could be expected to be protective of most ecosystem values in the context of normal urban residential use. However, in many circumstances much higher petroleum hydrocarbon concentrations may pose little or no risk of ecological impact.

Where petroleum hydrocarbons have been subject to significant weathering (eg in biologically active zones, such as garden topsoils), it is expected that the threshold for the onset of ecotoxicological effects may be significantly higher.

4 SUMMARY OF TOXICOLOGICAL AND FATE INFORMATION FOR NAPHTHALENE

4.1 FATE AND TRANSPORT

Much of the discussion of the fate and transport of petroleum hydrocarbons presented above also applies to naphthalene – a component of many petroleum hydrocarbon mixtures in the C_{10} range.

Naphthalene is soluble in water and volatile and therefore, although bioavailable, is lost quickly in the topsoil environment through leaching, volatilisation and biodegradation (see Megharaj *et al* 2000, Haeseler *et al* 1999, Bispo *et al* 1999). At greater depths however naphthalene can persist for greater periods of time (Madsen *et al* 1996).

Ma *et al* (1998) and Suprayogi *et al* (1999) found that at all PAH contaminated sites earthworms had below detection limit concentrations of naphthalene and that low molecular weight PAHs had very low bioaccumulation factors.

4.2 TOXICITY

There is very limited data on the specific toxicity of naphthalene in soils (see Table 2). The toxicity of naphthalene in the aquatic environment has been more widely studied. Some have predicted terrestrial ecotoxicity thresholds based on extrapolation from thresholds derived from the aquatic environment which has been subject to more intensive study (see Verbruggen *et al* 2001).

Environment Canada has completed a range of studies on earthworms and plants and found that LC_{50} and EC_{50}'s were in the range 56-86mg/kg initial concentrations[16]. Total naphthalenes (naphthalene and substituted naphthalenes) are present in diesel at around 3% (see Potter *et al* 1998). At this level, naphthalenes and substituted naphthalenes may be an important factor in the toxicity of the C_{10}-C_{16} fraction of TPH.

The toxicities of other low molecular weight (three ring) PAHs have been studied on various terrestrial species with similar results. Sverdrup *et al* (2002) found earthworm reproduction EC_{50}s for phenanthrene, fluorine and acridine respectively were 87mg/kg, 55mg/kg and 1500mg/kg. LC_{50} results were all above 1600mg/kg. However some studies have reported LC_{50} values for various earthworm species of between 17 and 750mg/kg for fluorene and phenanthrene (Neuhauser 1986 and Bowmer 1993 in WHO 1998:620-21). Seed germination tests on native Australian plant species reported EC_{50} values for anthracene of between 30mg/kg and 720mg/kg (Mitchell 1988 quoted in WHO 1998:620). All other PAH ecotoxicity studies involved mixtures so it is hard to determine effect concentrations for specific compounds.

Table 2: Naphthalene Toxicity

Test/s	Value	Comments	Source
Plant and Soil invertebrate LC_{50} and EC_{50}'s	56-86mg/kg initial exposure concentration.	Based on Environment Canada studies [17](sample results shown below).	CCME (2000)
Lettuce seedling emergence EC_{50}	64mg/kg (144mg/kg)	5 day test. Nominal concentration. in brackets	
Radish seed germination EC_{50}	86mg/kg (90mg/kg)	3 day test	
Earthworm 7 day LC_{50}	77mg/kg (137mg/kg)	Note 14 day studies found nominal concentrations up to 362mg/kg	
Lettuce growth EC_{50}	100+mg/kg in soil 4.8-34mg/L in nutrient solution	Difficult due to the volatility/degradability of naphthalene in soil.	Hulzebos *et al* 1993.
Aquatic $L(E)C_{50}$ tests converted via the equilibrium co-efficient method to represent a soil MPC.	0.12mg/kg	Dutch screening level. Low reliability because no terrestrial tests used.	Verbruggen *et al* (2001)

[16] Nominal or spiked concentrations were converted to initial or expected initial concentrations..
[17] Note the Canadian Soil Quality Guideline values for Naphthalene are between 0.6-22mg/kg depending on landuse. However these values are based on human health. The ecotoxicity tests shown here were used to help verify C_{10}-C_{16} TPH trigger levels.

Based on the summary of toxicological data presented above there is insufficient identified data on which to base a trigger or screening level for naphthalene in soil.

5 TOWARDS ASSESSING ECOLOGICAL EFFECTS OF PETROLEUM HYDROCARBONS AND NAPHTHALENE IN SOIL

Given the range of issues affecting the ecological impact of soil contaminants, and petroleum hydrocarbons in particular, there is a need for a practicable decision framework to guide decision-making. It is anticipated that ecological investigation levels for petroleum hydrocarbons and naphthalene would form part of that decision framework.

In establishing ecological investigation levels for petroleum hydrocarbons and naphthalene there is a danger that such levels will be used inappropriately to require clean-up of low level contamination that does not pose a substantial risk. It is important that any investigation level within the context of a broader decision framework.

Note that in Victoria a range of beneficial uses of land are to be protected. These include:

- Human health
- Maintenance of ecosystems
- Aesthetics
- Buildings and structures
- Production of food, fibre and flora.

A similar range of beneficial uses may be protected in each State or Territory.

In addition contamination of land must be managed to protect the quality of other media including groundwater.

The following decision framework aims to provide an indication as to whether the beneficial use "maintenance of ecosystems" is protected. It is not a comprehensive and rigorous assessment of all possible impacts of contamination on ecosystem composition and function. Further, separate assessment of possible impact on other beneficial uses is required.

5.1 SUGGESTED DECISION FRAMEWORK

A possible decision-framework to assist in making a practical assessment of ecological risk is presented below. The framework has been prepared in the context of:

- Consideration of ecological impacts in a typical urban context – eg a former service station being redeveloped for residential use;
- A conventional site-assessment has been completed in which the nature and extent of contamination has been identified.

Tier 1:

1. Critically observe site for evidence of impact and contamination eg patterns of impacted vegetation, areas of soils that are non-wetting.
2. Confirm the nature of the TPH – is it petrol- or diesel-derived?
3. Review chromatograms for consistency with petrol or diesel product
4. Confirm that the applicable scenario is consistent with the intended use of the investigation level (ie no more sensitive than domestic residential garden).

- ie ecological values for the site being considered are consistent with those used to derive criteria.

5. Compare measured TPH concentrations with investigation level.

Tier 2:

1. Critically observe site for evidence of impact and contamination eg patterns of impacted vegetation, areas of soils that are non-wetting.
2. Confirm ecological values to be protected.
3. Identify any critical local species etc.
4. Identify and assess exposure pathways.
5. Review the distribution of contamination and the potential impact given the specific pattern of landuse (eg do the areas of contamination coincide with proposed buildings or gardens).
6. Apply range of physico-chemical tests to provide improved understanding of the nature of the contamination.
7. Consider the nature of the soils and the impact on ecological risk.
8. If necessary select and apply a range of ecotoxicological tests consistent with the ecological values of the site to be protected. Normally this would include phytotoxicity, invertebrate and bacterial tests.
9. Make a risk-based assessment of ecological impact based on There is also a need to specifically consider the potential for off-site transport and impact of contamination on other media such as groundwater and surface water.
10. Review assessment to ensure consistency with site observations.

Tier 3:

1. Will not ordinarily be necessary unless there is significant off-site transport of contaminants with impact on other media eg surface water, groundwater.

5.2 ADEQUACY OF DATA TO SUPPORT ECOLOGICAL INVESTIGATION LEVELS FOR TPH AND NAPHTHALENE

The available published data regarding ecological effects and end points for petroleum hydrocarbons (<C_{15}) and naphthalene are limited. The criteria for assessing the adequacy of toxicological data for setting ecological trigger levels or screening criteria set out in the ANZECC (2000) Water Quality Guidelines have been applied for the purposes of the following discussion.

For a highly reliable trigger value the Water Quality Guideline requires either:

- NOEC data from more than three studies that are well conducted (sufficient duration, adequate controls, statistical power and exposure/effect data), and have multiple species (representing all the basic properties of the ecosystems), or
- Chronic NOEC data for at least 5 species from 4 different taxonomic groups.

The Water Quality Guidelines provide for moderate reliability trigger values to be derived from acute EC/LC_{50}s from greater than 5 species.

Low reliability trigger values can only be used as interim guidance. The lowest value available is divided by 20 to 1000, depending on the amount and the type of data (see ANZECC 2000 section 8.3.4).

Specific comments regarding the adequacy of data to set soil criteria for petroleum hydrocarbons (<C_{15}, ie petrol or diesel) are as follows (limited to published data):

- 4 studies reported end points for diesel and motor oil (Giddens 1976, Marwood *et al* 1998, Adam *et al* 1999, and Megharaj *et al* 2000).
- No studies reporting end points for petrol were identified in the peer-reviewed literature.
- Data collected on behalf of Environment Canada provides a major component of the data available for petroleum hydrocarbons in the petrol and diesel ranges (CCME 2000).
- Data is available for algae, bacteria, soil invertebrates (earthworms), and plants (seed germination and growth tests).
- However most individual studies only examined one or two taxonomic groups.
- Majority of results are effect-concentrations (not NOEC).

Specific comments regarding the adequacy of data to set soil criteria for naphthalene are as follows (limited to published data):

- The Canadian data is the only identified data specific to naphthalene in soil.
- Studies looking at total PAHs used creosote- or gaswork-contaminated sites and hence it is frequently not possible to distinguish the effects of naphthalene from other components in the waste.
- Data is only available for earthworms and plants.

Note that in setting Maximum Permissible Concentrations (MPC) RIVM, on behalf of the Dutch Ministry for Public Health, Welfare and Sport, noted that there is insufficient information regarding the ecological effects of naphthalene in soils to set a guideline value. Instead, RIVM established a MPC based on a partitioning relationship applied to a MPC for naphthalene in water (Verbruggen *et al* 2001).

Summary:
Insufficient data is available to establish ecological investigation for soil using the statistical extrapolation approach established in the Water Quality Guidelines for either petroleum hydrocarbons or naphthalene.

Sufficient data is available to establish interim values for petroleum hydrocarbons (<C_{15}) for use as a trigger for further investigation, until such time that data or information is able to be generated.

Insufficient data is available to establish an investigation level for naphthalene.

6 CONCLUSIONS AND RECOMMENDATIONS

6.1 CONCLUSIONS

1. Contamination of soils by petroleum hydrocarbons and naphthalene is a significant environmental issue and a significant economic issue given the number of petroleum storage and retailing sites and former gasworks being redeveloped for more sensitive uses.
2. Given the complexities in predicting ecological effects, particularly associated with petroleum hydrocarbons, it is necessary to place any “criteria” that may be proposed

within a decision decision-making framework to minimise the risk of improper application.

3. TPH is not a compound but the result of a non-specific analytical method used to broadly characterise petroleum contamination. It is therefore necessary to confirm the nature of the contaminant identified by the TPH analysis. The ecological investigation Factors affecting ecological impact include the nature of the hydrocarbon itself (source, composition, extent of weathering), the soils environment, and the nature of the proposed use and therefore the nature of the ecosystem that is desired to be protected.
4. Light fraction TPH is unlikely to significantly persist in surface soils, particularly those cultivated for garden use. Residual petroleum hydrocarbons in surface and other environments conducive to degradation and other weathering processes are likely to pose only low ecological risk. However, deeper contamination or more recent releases may include available petroleum hydrocarbons increasing the risk of ecological impact.
5. Ecological impacts at a site depend on the depth of contamination. The depth at which petroleum impacts become of less ecological concern (in a residential context) depends on volatilisation processes and the rooting depth of desired plants. Notwithstanding this, petroleum contamination at depth may pose a risk to groundwater that must be evaluated.
6. While the available toxicological data is not adequate for rigorous derivation of an ecological investigation level for total petroleum hydrocarbons, it is likely that values in the following order would be appropriate for maintenance of modified ecosystems (such as normal residential gardens):
   C_6-C_9: 100-500 mg/kg
   C_{10}-C_{15}: 500-2000 mg/kg

6.2 RECOMMENDATIONS

1. A risk-based decision framework be adopted for the assessment of ecological impact of TPH and naphthalene contamination of soils – consistent with the approach set out in the NEPM (Assessment of Site Contamination) (NEPC 2000) and the Australian and New Zealand Water Quality Guidelines (ANZECC 2000).
2. As part of the risk-based decision framework a numerical screening criteria for diesel or and petrol derived TPHs be adopted. To support this:
   - An approach consistent with that adopted for the Water Quality Guidelines (ANZECC 2000) should be adopted.
   - An appropriate group should be established to review the available data and establish such an investigation level.
3. That relatively simple ecotoxicological tests form part of the second tier of the decision framework.
4. That an appropriate group be established to make recommendations regarding the selection of ecotoxicological tests (and development of relevant Australian standard tests) for use in contaminated site assessment.
5. That consideration be given to selected use of analytical techniques, which separately quantify the aliphatic and aromatic components of TPH to assist with qualitative assessment of ecological impact.

DISCLAIMER:

This paper represents the view of the authors and does not necessarily reflect the view of EPA Victoria and will not necessarily be incorporated into future policy.

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