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Development of clean-up values for soils polluted with petroleum hydrocarbons using an indicator-fraction approach

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Abstract

Clean-up standards for soil pollution were promulgated on the 27th of April 1996 by the implementing order of the Flemish soil remediation decree. The soil clean-up standards differentiate between five land-use categories: natural areas (I), agricultural areas (II), residential areas (III), recreational areas (IV) and industrial areas (V). Generally, the clean-up standards for the last four categories are obtained by human exposure modelling. Derivation of soil clean-up standards for petroleum hydrocarbons using the Total Petroleum Hydrocarbon Criteria Working Group approach (TPHCWG-approach) was carried out in this framework. Knowledge about the toxicity, the physico-chemical properties, the fate and transport of the different petroleum hydrocarbon fractions in the soil is of major importance. Cancer and non-cancer risks are considered separately using respectively an indicator- and fraction-approach. Soil clean-up standards were calculated using the human exposure model VLIER-HUMAAN for each aliphatic and aromatic fraction. The groundwater clean-up standards were calculated according to the WHO procedure for deriving drinking water guidelines and were adjusted on a solubility basis if needed. Whole product (e.g. gasoline, diesel, heating oil ...) soil and groundwater clean-up standards were developed for use in the exploratory site investigation whereas fraction specific soil clean-up standards were derived for application in the descriptive site investigation and the site remediation. The modelling results served as the basis for a final proposal for clean-up standards for soil pollution.

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1 Introduction

There are a significant number of petroleum hydrocarbon impacted sites across Flanders resulting from a wide range of industrial activities (e.g. petroleum refineries), distribution practices and storage tanks. Since petroleum releases to the environment can cause safety hazards, ecological damage, adverse human health effects, unpleasant appearance and odour of soil and water, there is a real need for appropriate soil clean-up standards. For the land-use categories agriculture, residence and recreation, the actual Flemish soil clean-up value for petroleum hydrocarbons is 1000 mg/kg dm. For industry a soil clean-up value of 1500 mg/kg dm is used. The clean-up value for groundwater is equal to 500 µg/l. However, these values are not based on a risk-assessment approach. Consequently, these soil clean-up values are presently under revision.

Several approaches are possible: the indicator approach, the surrogate approach and the hydrocarbon block method. The indicator approach is most generally applied and most appropriate for evaluation of the carcinogenic risks from Total Petroleum Hydrocarbons (TPH). This approach assumes that the estimated risks from TPH are characterized by a small number of indicator compounds (benzene, carcinogenic Polycyclic Aromatic Hydrocarbons (carcinogenic PAH)). The use of the indicator approach for determining non-carcinogenic risks has, however, not been fully developed.
The surrogate approach assumes that TPH can be characterized by a single surrogate compound. This methodology could overestimate toxicity and mobility because of the compounds typically available for use as surrogates (for example benzene). A variant of the surrogate approach is the whole product approach in which toxicity and mobility of TPH are based on a whole product with similar character. Weathering effects and variability of the mobility in the constituents of a typical hydrocarbon product are neglected.
More recently, approaches have been developed which are a compromise between the indicator and surrogate approach. Carcinogenic risks are estimated based on indicators (benzene and carcinogenic PAH) while the non-carcinogenic risks from TPH are estimated based on a relatively small number of groupings, fractions or blocks. Each of these so-called blocks is composed of TPH-constituents with similar toxicity and mobility. The TPHCWG-approach [1] and the methodology developed by the Massachusetts Department of Environmental Protection (MADEP) [2] are two examples of this compromise approach. The basic approach of TPHCWG is similar to that developed by MADEP in that TPH are split into a small number of blocks with similar properties. The main difference is that TPHCWG selects blocks based on potential mobility whereas MADEP defines blocks based on available toxicity data. In fact the TPHCWG-approach is an extension of the MADEP-approach based on a more complete database of physico-chemical and toxicological properties.

In the TPHCWG-methodology blocks or fractions are defined using their Equivalent Carbon Number (EC). The EC is related to the boiling point of a

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chemical normalized to the boiling point of n-alkanes or its retention time in boiling point gas chromatographic column. This relationship was empirically determined. Hence, for chemicals where the boiling points are known, an EC can easily be calculated [3].

The TPHCWG-approach was chosen since the advantages of the indicator approach and the surrogate approach are combined. Additionally its quality is generally recognised and the methodology is followed by various international organizations. This article focuses only on the estimation of non-cancer risks using TPH-fractions. Discussion with recognised soil remediation experts revealed that a detailed analysis as required by this methodology in the first phase of the soil investigation procedure is rather expensive. Consequently a scientific sound methodology in which soil and groundwater clean-up values for individual TPH-fractions and whole petroleum products are used, is proposed. In order to achieve this goal the multimedia model VLIER-HUMAAN was used for the derivation of soil-clean-up values for the different land-use categories. The groundwater clean-up standards were derived according to the drinking water guidelines of the World Health Organization. The modelling results served as the basis for the final proposal for petroleum hydrocarbon soil pollution clean-up standards and will be further discussed in this paper. A short overview of the legislative framework in which these clean-up values for soil contamination will be implemented, will also be discussed.

2 General legislative framework

On the 22nd of February 1995, the new soil remediation decree was ratified by the Flemish government. The Public Waste Agency of Flanders (OVAM) is the competent authority for this matter, and supervises all the soil and waste related activities. With regard to soil clean-up values three issues are of major importance:

- an inventory of contaminated sites;
- the difference between historical and new soil contamination;
- the soil remediation procedure;

Information on site pollution leads to the constitution of an inventory of contaminated sites. This information originates from a systematic examination of polluted sites at the time of property transfer, closure of well defined industrial activities or installations or in the framework of the execution of an obligatory periodical site investigation related to these industrial activities.

A distinction in policy is made regarding the time period the soil contamination is caused. Historical soil pollution dates from before the Decree came into force (the 29th of October 1995). New soil pollution originates from after the Decree went into effect. The clean-up of new pollution is compulsory as soon as the soil clean-up values are exceeded. Considering historical pollution, the decision to

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decontamination will depend on the actual or potential risk of the pollution to man and environment. So a risk-assessment approach is followed in accordance with the present legislation. The obligation for further investigation only arises after the clean-up order is issued by the government. Considering the limited financial resources available, the clean-up of historical pollution is subjected to priority setting by the government.

The soil decontamination procedure starts with an exploratory site investigation and is followed when needed by a descriptive site investigation, a site remediation project and site remediation operation.
The goal of the exploratory site investigation is to establish if there are indications that soil clean-up values are exceeded (new pollution) or if there are indications for a 'serious threat' (historical pollution). The aim of the descriptive site investigation is to establish the seriousness of the site contamination. The source, quantity, concentration and origin of the contaminating substances, the possibility that these might spread and the risks they pose for man and environment (vegetation, animals, surface water, groundwater) are described into detail. In case the descriptive site investigation points out that soil clean-up values are exceeded or that there is a risk for man and environment, a site remediation project is made and after approval by the OVAM the site remediation can start [4,5].

3 Modelling approach and data

A soil clean-up value is defined at a level of soil pollution above which serious harmful effects for humans or the environment might occur, taking into account the characteristics and functions of the soil. The exposure assessment multimedia model VLIER-HUMAAN [6] is used in the framework of the Flemish soil decree for the derivation of soil clean-up values for the TPH-blocks in the solid phase. It is based on the formulas used in the Dutch HESP-model with some additions and modifications [7]. These changes relate mainly to chemical-specific parameters and to land-use scenarios. An overview of the different fate and transport routes considered by VLIER-HUMAAN is given in figure 1.

Six land-use scenarios have been defined for four land-use classes [5]: agricultural areas (II), residential areas (III), recreational areas (IV) and industrial areas (V). Each land-use type has been characterised by typical exposure pathways and by typical human activity patterns. In an agricultural land-use all exposure pathways are active: exposure via dermal contact, ingestion and inhalation of soil particles and dust, inhalation of volatile chemicals, use of untreated groundwater as drinking and bathing water, consumption of vegetables, milk and meat. For industrial areas only exposure of adults during work activities (indoor or outdoor) via dermal contact, ingestion and inhalation of soil particles and dust, inhalation of volatile chemicals and use of tap water are considered. The other two land-use scenarios are intermediate. In case of the residential scenario consumption of milk, meat and groundwater as drinking

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water is not considered. Consumption of vegetables is limited. The remaining exposure pathways are the same as for agricultural land-use. For the recreational scenario the same exposure pathways as for industrial land-use are taken into account but now for children and adults during sporting activities or leisure activities a few hours a day or during the week-end.

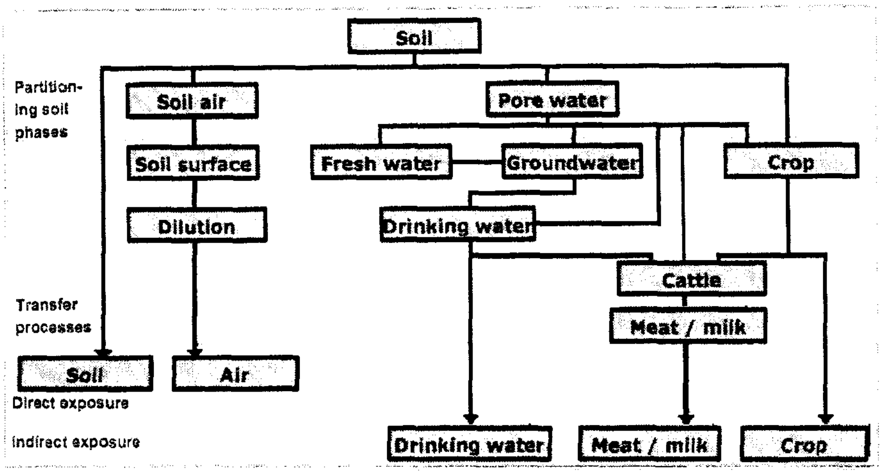

Figure 1: Overview of the different fate and transport routes considered by the model VLIER-HUMAAN.

The calculations of the exposure model are grouped in three layers. The first layer calculates the distribution of the contamination in the soil. The second layer covers the migration to other compartments (air, water, vegetables ...) and the concentrations in these compartments. In the third layer exposure of human beings (children, adults) in contact with the considered environmental compartments is calculated. Details can be found in [6].
Total exposure for non-carcinogenic compounds is divided by the Tolerable Daily Intake (TDI) resulting in a risk index (RI). This TDI is in fact equivalent with the Reference Dose (RfD) which is proposed by TPHCWG. Children and adults are considered separately. The soil clean-up value is primarily derived on the level in soil corresponding with a calculated RI equal to 1. Total exposure comprises exposure from the polluted site together with background exposure from undefined sources. Air quality guidelines, drinking water quality objectives, phytotoxicity (all land-use categories), toxicity for cattle and legal standards for vegetables and animal products (land-use agriculture) are used as additional limiting criteria.

Groundwater clean-up values are not based on risk-assessment modelling by means of VLIER-HUMAAN. Groundwater is a mobile medium which crosses areas with different land-use (e.g. from an industrial area to an agricultural area).

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For that reason there is no differentiation between different land-use categories. Groundwater clean-up values represent drinking water quality standards since in Flanders, groundwater serves as a source for drinking water production and, additionally, private wells for drinking water use still exist. For that reason groundwater has to meet drinking water quality objectives.

Human toxicological data (RfD), physico-chemical data were taken from the TPHCWG (solubility, vapour pressure, Henry coefficient, octanol-water partition coefficients, diffusion coefficients air and water) [1] and from the Dutch National Institute of Public Health and Environmental Protection (RIVM) (permeation through polyethylene and polyvinylchloride water pipeline) [8]. Background exposure via air was calculated based on the information of the Flemish Environment Agency VMM (1999) [9]. Data on background exposure via food in Flanders was not available. Information on biological block-specific data (uptake vegetables, absorption and excretion by cattle, limit values for meat, milk and vegetables) was also lacking. Consequently, the standard assumptions of the model VLIER-HUMAAN were used in the calculations. The upper limit for the protection of cattle was set on respectively 155 and 230 mg/kg dm for the aromatic blocks $EC_{>8-10}$ and $EC_{>10-12}$ in agricultural land-use [10].

For each TPH-block and each land-use type, calculations of the exposure were performed to establish a total exposure that is equal to the RfD for non-cancer effects. Values for the RfD were taken from TPHCWG [1] for all TPH-blocks except the aliphatic block EC_{5-6} and $EC_{>6-8}$. In the latter case the RfD of RIVM was used [11] since according to our opinion a TDI of 2 mg/kg bw based on 6 studies using n-heptane and 7 studies based on hexane-isomers (cyclohexane, methylcyclohexane) is more reliable than the RfD of 5 mg/kg bw [1]. The latter was selected based on indistinct reasons although they derived a reference dose of 2 mg/kg bw based on a study using n-heptane. The toxicological and physico-chemical properties of the aromatic blocks $EC_{>5-7}$ and $EC_{>7-8}$ are based on benzene (non-carcinogenic effects) and toluene. Since soil clean-up values for these chemicals are already available in VLAREBO [5], no calculations were carried out for these blocks. Ethylbenzene, xylenes and styrene were misclassified by the TPHCWG and grouped together with toluene. However those chemicals should be put into block $EC_{>8-10}$. Nevertheless, the physico-chemical properties as defined by the TPHCWG were used because the above mentioned misclassification had no significant consequences on the physico-chemical properties of the different blocks.

4 Results and discussion

4.1. Individual TPH-blocks

4.1.1 Soil

The soil clean-up values for individual TPH-blocks are listed in table 1. In first instance these results were tested by the RfD. This resulted sometimes in very

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high soil clean-up values (>25000 mg/kg dm and most >100000 mg/kg dm). Certainly the results for the aliphatic blocks in land-use types IV and V are extremely high. The results for the aromatic blocks were significantly lower. Consequently, exact modelling of the distribution of the TPH-blocks is not longer possible since the maximum solubility of the petroleum hydrocarbons in pore water at the calculated clean-up value is exceeded many times.

Current detection limits for standard analytical techniques are of the same order of magnitude as the calculated values in table 1 for some of the aromatic blocks ($EC_{>8-10}$, EC_{10-12}) in destination type II. In order to avoid that those sites would be assessed as polluted and included in the inventory of contaminated sites, these values have to be increased. For that reason the requirement that the lowest soil clean-up values are at least 5 times the detection limit is used as general rule.

In first instance the modelling results were checked to additional toxicological criteria: tolerable concentration in air (TCA), drinking water guidelines, phytotoxicity and toxicity for cattle (land-use category II). Relating the calculations to air quality guidelines and drinking water standards lowered some of the extreme results in particular for the aliphatic block EC_5-EC_6 and the aromatic blocks $EC_{>8-10}$ and $EC_{>10-12}$. Toxicity information for cattle is very limited. For the aromatic blocks $EC_{>8-10}$ and $EC_{>10-12}$ in destination type II those standards were not exceeded [10]. Lowering of the remaining extremely high results according to phytotoxicity and legal standards for vegetables was not possible since data were only available for mixtures and not for individual TPH-blocks.

However, other than the toxicological criteria could be applied to limit soil clean-up values: correction for the formation of colloids, solubility, safety standards, visual aspects, odour and legislation. Some of the criteria have only a rather weak scientific basis and interpretation is subjective. For instance, visual and odour observations depend from one person to another, the soil type, concentration, petroleum product and vary according to our practical experience from 10000-20000 mg/kg dm. Colloidal accommodation occurs but scientific information is scarce and correction of the calculations is not straight-forward. Solubility corrections for TPH in pore water lead to unreasonable low soil clean-up values and are not a good indicator for mobility of the pure product formed. Consequently only safety standards and legislation were withdrawn. From the point of view of safety, the Canadian Council of Ministers of the Environment recommended that the sum of the concentration of the TPH-blocks $EC^{al}_{>6-8}$ $EC^{al}_{>8-10}$ $EC^{ar}_{>8-10}$ should not exceed 1000 mg/kg dm [10]. This criterion was developed in order to avoid crossing of the ignition limit by accumulation of gases in buildings and could be applied in a site-specific risk assessment but not for deriving generic soil clean-up values. Current Flemish legislation allows that contaminated soil, which is dumped on a landfill, may contain maximum 10 % organic material. In order to leave some buffer for other organic pollutants it was decided in agreement with representatives of the industry and OVAM to set the

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upper limit on 2 % for the sum of the soil clean-up values of all blocks. Additionally for those blocks for which the calculations resulted in extremely high soil clean-up values, it was decided not to propose soil clean-up values. The final result of all those adaptations and modifications is summarised in table 1.

According to table 1 two requirements have to be fulfilled. Firstly, the soil concentration of the analysed sample may not exceed any of the individual soil clean-up values in table 1 for those blocks for which soil clean-up values were proposed. Secondly, the sum of the measured soil concentrations of all the mentioned blocks in table 1 and for one land-use category may not exceed 20000 mg/kg dm.

Table 1: Soil clean-up values for TPH-blocks.

Block	Land-use category				
	Soil (mg/kg dm)				Groundwater (μg/l)
	II	III	IV	V	II,III,IV, V
Aliphatics					
EC_{5-6}	21	28	250*	253*	6000
$EC_{>6-8}$	61	76	-	-	6000
$EC_{>8-10}$	16	19	-	-	300
$EC_{>10-12}$	87	96	-	-	300
$EC_{>12-16}$	12839	-	-	-	300
$EC_{>16-21}$	-	-	-	-	6000
Aromatics					
$EC_{>8-10}$	10***	11	47	233**	120
$EC_{>10-12}$	7***	22	85	367**	120
$EC_{>12-16}$	13	49	220	-	120
$EC_{>16-21}$	38	1174	7956	-	90
$EC_{>21-35}$	4335	7382	7957	-	90
Total					
Σ aliphatics + aromatics	20000	20000	20000	20000	-

* reduced based on Tolerable Concentration in Air (TCA)
** reduced based on drinking water guideline
*** elevated based on detection limits

4.1.2 Groundwater

Groundwater standards are given in table 1. These are deduced from the toxicological reference doses (RfD) of the TPHCWG [1] in accordance with the procedures of the WHO to derive drinking water quality objectives [12]. It is assumed that 10 % of the RfD might be attributed to consumption of drinking water for an adult of 60 kg drinking 2 litres of water a day. Additionally, the presence of light non-aqueous phase liquids is considered as unacceptable. For that reason check of the solubility has to be carried out. Since different blocks

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influence each others solubility and since under normal circumstances those blocks occur in polluted groundwater in combination with other blocks solubility criteria for individual fractions were not used for reduction of the groundwater clean-up values.

4.2 Whole petroleum products

4.2.1 Soil

Based on the calculated soil clean-up values for the different individual blocks before adjustment and the average composition of commercial petroleum products as listed in table 2, soil clean-up values for whole petroleum products can be calculated.

Table 2: TPH-Block-composition (weight %) of commercial petroleum products.

Block	gasoline	diesel	fuel oil 2	JP-4	crude oil	motor oil
aliphatics						
EC_{5-6}	28,82			7,89	3,58	
$EC_{>6-8}$	36,76	0,10	0,24	25,10	10,48	8,75
$EC_{>8-10}$	9,48	0,90	0,72	10,80	5,30	12,50
$EC_{>10-12}$	0,31	8,40	6,26	5,67	3,40	8,27
$EC_{>12-16}$		48,10	26,52	2,25		11,58
$EC_{>16-21}$		22,80	20,25			2,89
aromatics						
$EC_{>8-10}$	13,78	1,61	0,35	5,27	5,16	4,54
$EC_{>10-12}$	5,81	1,40	3,52	4,34	0,49	3,20
$EC_{>12-16}$		11,40	13,76	1,59	0,21	7,66
$EC_{>16-21}$		7,00	9,22		0,11	27,01
$EC_{>21-35}$		0,028	0,15			6,41

The values in table 2 are approximations for several reasons. Detailed analyses are not available for all products. Different sources give different compositions for the same products. Equivalent Carbon numbers (EC) are not available for all chemicals. Consequently the EC was estimated based on the effective carbon number and the EC of homologues. The sum of weight percent is not always 100 % due to the presence of insoluble fractions and other chemicals.

Soil clean-up values for commercial petroleum products can be calculated by neglecting or taking into account additivity of effects. We opted to ignore additivity of effects. On the one hand it is reasonable to assume that aliphatic and aromatic TPH-blocks have a different mode of action supporting the conclusion that the toxicology of the complete petroleum product cannot be obtained by simple summation of the effects of the blocks. On the other hand it is also reasonable to assume additivity of effects for fractions that affect the same source system or target organ. Additive effects between aliphatic fractions and

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between aromatic blocks cannot be excluded, but information is scarce and interpretation is not straight-forward.
If one ignores additivity of effects and specific interactions are neglected the risk index of the mixture can be calculated according to the following equations:

$$RI_i = D_i/RfD_i$$
$$RI_i = F_i\,(f_i * C_{mixture})/RfD_i \quad 1$$

where i stands for block i of the petroleum mixture (diesel, gasoline, jet fuel,...). Dose D_i of each block i is a function of the concentration in the soil C_i. The function F_i can be different for each block i, since each block i is characterised by different physicochemical properties. C_i is linked to $C_{mixture}$ via the fraction f_i in the mixture. $C_{mixture}$ is the concentration of the commercial petroleum product in the soil. The function F_i is in fact our model VLIER-HUMAAN.
This equation is used in the first step for the calculation of a soil clean-up value for each individual block of a well defined petroleum product (diesel, gasoline,...). In the second step the calculated soil clean-up values for all blocks of the considered whole petroleum product (for example gasoline) are compared to each other and the most stringent one is taken as the soil clean-up value for the considered petroleum product. This results in table 3.

Table 3: Calculated soil clean-up values for commercial petroleum products.

	Land-use	gasoline	diesel	fuel oil 2	JP-4	crude oil	motor oil
Soil	II	22*	114	94*	57*	58*	66*
(mg/kg dm)	III	80*	430	356	176	213	152
	IV	341	1930	1599	892	910	1035
	V	878	14472	10415	3206	4516	5132
Groundwater (µg/l)	II,III,IV,V	871	624	872	2277	2326	333

* These values need to be elevated to 100 mg/kg dm due to detection limits

1.1.2 Groundwater

Groundwater clean-up values for whole petroleum products are calculated based on the groundwater clean-up values ($GWCV_i$) for the individual blocks. Since additivity of toxicological effects is ignored for soil, it is for reasons of consistency also ignored for groundwater:

$$RI_i = (f_i * C_{mixture})/GWCV_i \quad 1$$

This equation is used in the first step for the calculation of each individual block of a well defined petroleum product (diesel, gasoline,...). In the second step the calculated values for all blocks of the considered petroleum product (for example gasoline) are compared to each other and the most stringent is taken as the groundwater clean-up value for the considered petroleum product resulting in the values reported in table 3. Inspection of the solubility data from the literature

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revealed that there was no reason to reduce the calculated groundwater clean-up values [13].

5 Conclusions

The results show that the TPHCWG-approach offers a very valuable and flexible basis for deriving soil pollution clean-up values for TPH-blocks and whole petroleum products. Some of the initially derived soil clean-up values are rather low or extremely high. This can partly be attributed to the conservative assumptions made in the data (RfD, bioconcentration factors for vegetables calculated using VLIER-HUMAAN) but probably also due to the modelling framework which is not able to take into account all detailed processes (for example those processes related to the solubility of the different blocks and the solubility interactions between different blocks). Consequently some modifications on the modelling results have to be made. Some of the low modelling results have to be increased since they are of the same order of magnitude as the current detection limits of the standard analytical techniques and inclusion in the inventory of contaminated sites should be avoided. A few of high modelling results were lowered based on toxicological criteria (TCA and drinking water guidelines) whereas others were limited based on legislation. Groundwater clean-up values were derived using the World Health Organization approach for drinking water guidelines. Additivity of toxicological effects was ignored for the calculation of soil and groundwater clean-up values for whole petroleum products. The present proposal is still under discussion with OVAM and external experts. The practical applicability is evaluated based on practical case-studies. The decision on the methodology and modifications to be applied in order to arrive at a feasible proposal is a policy decision.

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