S-160: Environmental sampling: Soil and sediment

The analysis of phthalates in soil and sediment

Brief concept report of validation results

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Contents

1 Introduction

By order of the European Council for Plasticisers & Intermediates (ECPI), research has been carried out to develop an analytical procedure for the analysis of phthalates in soils and sediments.
This project is the first part of a larger project, that concerns development of analytical procedures for the analysis of phthalates in plants, milk, fish and cattle feed.
This report presents results for the analysis of phthalates in soils and sediments.

The first part of the project concerns the analysis of soil samples. Chapter 2 presents the analytical procedures. The validation of the analytical procedure for soil samples is presented in chapter 3 and, in chapter 4, the results of analysis of 35 soil samples from the Netherlands are presented.
Chapter 5 deals with a limited validation of a similar analytical procedure for sediment samples. Chapter 6 presents the results of 36 sediment samples, originating from Dutch and foreign waterways.

In chapter 7, finally, the results are discussed.

The compounds, analysed during this project are:

- dimethylphthalate (DMP),
- dimethylterephthalate (DMT),
- diethylphthalate (DEP),
- dipropylphthalate (DPP),
- di(iso)butylphthalate (DIBP),
- dibutylphthalate (DBP),
- di(2-ethylhexyl)phthalate (DEHP),
- butylbenzylphthalate (BBzP),
- dicyclohexylphthalate (DcHP),

and the iso mixtures:
- di(iso)heptylphthalate (DIHP),
- di(iso)octylphthalate (DIOP),
- di(iso)nonylphthalate (DINP),
- di(iso)decylphthalate (DIDP).

In this report, all compounds are referred to by their abbreviation.

2 Analytical procedures

2.1 Measures against contamination

As phthalate compounds are generally used as weakening agents in plastics, extreme care has been taken to avoid introduction of phthalates from the surroundings into the samples, which could lead to false positive results. For this purpose, the following measures were taken:

- All handling of samples and chemicals, including extraction and analysis, has been carried out by one and the same technician.
- All handling has been carried out in a fume hood, dedicated to the analysis of phthalates.
- All equipment parts used were made either from glass or from metal. The use of gloves has been avoided.
- During the period of analysis, all glass or metal equipment was stored in an oven at 400 °C.
- All measurements have been carried out on one and the same instrument.

During the first validation experiments, the solvent was distilled twice before it was used, in order to obtain a very pure solvent, as free from phthalates as possible. During the validation experiments however, tests showed that distillation was not effective in purifying the solvent regarding phthalate compounds. In fact, the handling involved in the distillation process sometimes even lead to higher concentrations of phthalates, compared with fresh solvent, taken from the original bottle.

2.2 Extraction procedure of soil and sediment samples

Extraction of soil and sediment samples has been carried out by means of Accelerated Solvent Extraction (ASE), according to EPA 3545. Dichloromethane was used as an extraction solvent. The extraction was performed at 2000 psi and 100 °C. The total extraction procedure required 15 min.
In earlier validation experiments on different organic compounds in soil samples, the mean recoveries have been found, as presented in table 1.

Table 1.
Recovery for ASE extraction, using dichloromethane.

	EC-3	SRM 1941A	HS-6
	recovery %*)	recovery %*)	recovery %*)
naphtalene	586	83	104
acenaphtene	152	105	
fluorene	92	79	55
phenantrene	130	104	106
anthracene	192	144	104
fluoranthene	106	91	94
pyrene	116	93	85
benzo(a)anthracene	110	100	83
chrysene	93	148	103
benzo(b)fluoranthene	108	107	80
benzo(k)fluoranthene	124	113	92
benzo(e)pyrene	112	100	
benzo(a)pyrene	115	102	69
indenopyrene	120	96	82
dibenzanthracene	198	119	179
benzo(ghi)perylene	133	103	94
PCB 28+31	142	104	
PCB 52	114	158	
PCB 101	91	135	
PCB 118	142	91	
PCB 138	97	77	
PCB 153	95	71	
PCB 170	115		
PCB 180	97	182	
1,2-dichlorobenzene	103		
1,3-dichlorobenzene	105		
1,4-dichlorobenzene	101		
1,2,3-trichlorobenzene	117		
1,2,4-trichlorobenzene	115		
1,3,5-trichlorobenzene	90		
1,2,3,4-tetrachlorobenzene	99		
1,2,3,5-tetrachlorobenzene	81		
1,2,4,5-tetrachlorobenzene	85		
pentachlorobenzene	108		
hexachlorobutadiene	90		
hexachlorobenzene	109		

*): recovery calculated with respect to the reference value.

Before extraction, about 15 g of sample was dried by mixing it with an equal amount of anhydrous Na_2SO_4, using a metal spoon, until a dry mixture was formed. Then the sample was completely transferred into the metal extraction cell. Then, an exact volume of a solution of Di(ring-D_4)butylphthalate

(DD_4BP) and Di(ring-D_4)(2-ethylhexyl)phthalate (DD_4EHP) in dichloromethane was added on top of the sample in the extraction cell (internal standards I), using a syringe. At first only DD_4BP was used as an internal standard, later (during the second half of the validation experiments) also DD_4EHP was introduced. These internal standards are used as a tracer for the extraction recovery. The results presented in this report are not corrected for the recovery of internal standards I.
After extraction, an exact quantity of internal standard II (i.e. diallylphthalate in dichloromethane) was added into the extract vial, using a syringe. This internal standard is used to correct measured concentrations for differences in extract volume of the samples. The extract volume, given by the ASE instrument, usually varies between 40 and 50 ml.

2.3 Analysis

Analysis of the extract was carried out on an LC-LVI-GCMS. Clean up of the extract takes place in the LC part of the instrument, which is used to separate matrix compounds from analytes. Analytes are transferred into the LVI-GCMS, preconcentrated on column and then measured. The concentrations of the phthalates were calculated based on an external calibration standard in dichloromethane. All concentrations are corrected for differences in extract volume, using internal standard II. Concentrations are nor corrected for the recovery of internal standards I.

2.4 Spiking procedure

During the validation experiments blank soil samples have been used that were spiked with phthalate compounds.
The spiking procedure has been as follows:
All phthalate compounds were solved in dichloromethane in exactly known concentrations (spiking solution). The blank samples to be spiked were of sandy clay soil, that was collected from an area next to the ALcontrol building.
The soil was dried at 105 °C and ground to particles <200 µm. Spiking was performed by weighing 15.0 g of soil into a glass vessel, and spraying an exact volume of the spiking solution on the soil surface by means of a glass syringe. Then the soil was placed in a fume hood for two hours, to allow the solvent to evaporate. The spiked soil sample was made homogeneous by stirring with a metal spoon and then stored in a refrigerator at 5 °C for a period of 48 hours (aging period).

2.5 Methods of other analyses

Other analysis that have been carried out in the scope of this project are dry matter (%DM) and total organic carbon (%TOC). The methods for these analysis have been as follows:

<u>Dry matter</u>
About 10 g of sample is weighed into a porcelain cup and dried at 105 °C during 8 h, or (when dried during a shorter period) until constant weight is obtained.

<u>Total organic carbon</u>
The sample material that has been used for the analyses of the dry matter is ground (HOE?????) until the particle size is smaller than 200 µm.
An amount of the ground sample (0,6 - 1 g) is weighed into a porcelain boat and placed in an oxygen fed tube oven at 1300 °C. The total carbon content (inorganic and organic) is measured as CO_2, by means of an infrared detector.
Next, about the same quantity of sample is weighed into a conical flask. The conical flask is gastight mounted inside the instrument, and a volume of phosphoric acid is added allowing all carbonate to react to CO_2. This amount of CO_2 is measured by means of the same detector. The amount of inorganic carbon is substracted from the total carbon content to give the %TOC.

Mineral particles < 2 µm
The sample is dried at 105 °C. About 20 g of dried soil sample is treated with chloric acid and hydrogenperoxide to remove carbonates and organic matter from the sample (mineralization). Sodiumpyrophosphate is added to prevent coagulation of particles. The content of particles < 2 µm then is measured from the settling speed of the solid particles in the liquid, using Stokes' law.

3 Validation of the analytical procedure for soil samples

In order to validate the analytical procedure, the following characteristics have been determined:

- The blank level of phthalates on 7 different days. The complete analytical procedure was followed, using Na_2SO_4 as sample material.
- The detection limit, by spiking a blank soil sample with the phthalate compounds.
- The reproducibility, by analysing a spiked soil sample on 11 different days.
- The precision of the method, as the standard deviation of the repeatability. The precision, by means of five series of duplicate analyses at different levels of spiking.
- The recovery of the method, using the results of the precision experiments.

3.1 Blank level

Because of the high risk of contaminating the samples with phthalates, blanks are a very important checkpoint within the analytical procedure.
During the validation process, concentrations of phthalates were measured in pure fresh solvents, in order to choose the best suitable solvent brand.
Besides, blanks of the analytical procedure were measured on seven different days. The analytical procedure blank is taken as the whole procedure, using only Na_2SO_4 as sample material ('everything but the sample itself').

Table A1 presents blank concentrations of phthalates in pure pentane and dichloromethane, taken from new, unopened bottles, and immediately injected into a GC vial. The concentrations of all phthalates in the three tested solvents are comparable. All validation experiments and all analyses have been carried out using DCM from Promochem as solvent.

Table A2 presents results of blanks, obtained during a period of seven days. These series of blanks have been measured in the beginning of the validation experiments. From later experiments, it was concluded that the blank level increased to a higher but constant level. Table 2 presents typical blank concentrations, measured during the analyses of soil and sediment samples.

Table 2.
Mean blank level of the analytical procedure.

	Blank Na_2SO_4
	µg/kg
DMP	n.d.
DMT	n.d.
DEP	2.5
DPP	n.d.
DIBP	36
DBP	24
DEHP	35
BBzP	2.8
DCHP	4.3
DIHP	n.d.
DIOP	70
DINP	n.d.
DIDP	n.d.

n.d.: not detectable.

3.2 *Detection limit*

The detection limit has been determined by spiking the phthalate compounds on a low concentration level to anhydrous Na_2SO_4, and extracting the spiked material immediately afterwards. This experiment has been repeated 9 times, in one analytical series (conditions of repeatability). The level of spiking is ca. 5 µg/kg for the single compounds and ca. 10 µg/kg for the iso mixtures.
The detection limit for each phthalate has been calculated as the standard deviation of the mean concentration, multiplied by $3\sqrt{2}$. The factor 3 originates from the definition of the term detection limit. The factor $\sqrt{2}$ is introduced, because all concentrations measured are calculated, based on only one external standard.

The results of the detection limits are presented in table A3. The results are summarized in table 3.

Table 3.
Detection limits for phthalate compounds.

	Detection limit
	µg/kgdm
DMP	0.7
DMT	0.7
DEP	0.9
DPP	0.9
DIBP	2.0
DBP	3.8
DEHP	3.5
BBzP	1.2
DCHP	1.3
DIHP	4.1
DIOP	5.6
DINP	3.2
DIDP	2.9

3.3 *Reproducibility*

The reproducibility (RSDR) of the analytical procedure has been measured by analysing a soil sample on 11 different days. Before analysis the soil was spiked, allowing an aging period of 48 h. The level of spiking was circa 300 µg/kg for each phthalate compound.
According to the spiking procedure, 11 samples of blank soil were prepared and spiked on different days and stored at 5 °C. Each sample was then dried with Na_2SO_4, exactly 48 hours after spiking, extracted and measured.
The results are presented in table A4. The standard deviations of the reproducibilities are between 4.5 % and 9.6 % for each compound or iso mixture, except for DMP. The reproducibility of this compound is appr. 14 %. Except for DMP, the reproducibilities are satisfactory. It should be noted however, that the recoveries for all compounds, especially for DMP, are rather low.

3.4 *Precision*

The precision of the analytical procedure is expressed as standard deviation of the repeatability (RSDr). The precision has been determined by means of five series of duplicate analyses on different levels of spiking. The results are presented in table A5.

The standard deviation of the repeatability is calculated as pooled standard deviation according to:

$$RSDr(\%) = \sqrt{\left(\Sigma\, (sd_i\%)^2\right)/n}$$

where: sd_i is the standard deviation of each individual duplicate analysis',
n is the total number of duplicates (5)

The precision varies between 8.6 % and 23 %. It should be noted that the precision is determined on 5 different concentration levels.
The precision is influenced by the bad figures for level IV, where poor duplicate results were obtained. The reason for this is unknown. Eliminating these results from the calculation leads to the precision values in table 4.

Table 4.
Precision of the analytical procedure.

	Precision RSDr(%)
DMP	16
DMT	8.6
DEP	8.1
DPP	6.8
DIBP	6.8
DBP	6.0
DEHP	16
BBzP	7.1
DCHP	7.7
DIHP	15
DIOP	16
DINP	21
DIDP	15

The higher standard deviation for the repeatibility for the iso mixtures will mainly be caused by the fact that integration of the chromatogram is more difficult.

3.5 Recovery

The recovery of the method has been determined, using the precision data, at five levels of concentration. On each concentration level, the recovery is calculated as the mean concentration, measured for the spiked sample, divided by the mean concentration, measured for the spiked extract. The results are presented in table A6-A. For each compound, a mean recovery is calculated over the five recoveries (table A6-B).
The mean recovery varies between 80 % and 115 % for the single compounds, DMP excluded. The individual recoveries show higher and lower percentages. Close examination of the results suggests that these low recoveries were caused by the fact that the clean up in the LC part of the instrument was not optimal when matrix of the sample is present. The effect was made visible by an experiment in which an extract of the blank soil sample was diluted 5, 10, 50, 100 and 500 times, respectively. To these diluted extracts and to the undiluted extract, equal amounts of phthalates were added and the extracts were measured. The results are presented in table A7-1. They clearly show that diluting the extract, and thus the matrix, leads to an increase of the recovery.
After optimisation of the clean up, the experiment was repeated. These results are presented in table A7-2. It is obvious that the recoveries have improved after the optimisation and are satisfactory.

Recoveries for the iso mixtures vary between 81 and 91 %. The high recoveries at level I are caused by the phthalate present in the soil used, i.e. the 'blank' value.

After the clean up step was optimised the recovery was determined, using different matrices: dried soil (105 °C), wet soil and silversand. These results are presented in table A8. The analytical procedure was carried out immediately after the spiking of the samples.
The recoveries vary between 47 % and 102 % for dried soil. For DMP the recoveries are lower than 15 %, for DEHP the recovery is between 150 % and 180 %; this will probably be caused by the presence of DEHP in the dried sample. There is no explanation for the high recovery for DINP.
For silversand, the recoveries vary between 50 % for DIBP to 125 % for DIOP. For DINP the duplicates are −9 % and 72 %, caused by the presence of DINP in the blank sample.
The recoveries for wet soil are good: they vary between 61 % and 116 %, while most recoveries vary between 80 and 90 %.

Table A9 presents results of spiking experiments to silversand and wet soil. After spiking, the samples were aged during 48 h, after which the analytical procedure was carried out. The recoveries vary between 62 and 110 % for silversand and between 52 and 103 % for wet soil. The recovery for DMP is 6 % for wet soil, while it is circa 65 % for silversand. The cause for this is not known.

Generally, the recoveries were regarded as satisfactory. There is no explanation for low values for DMP in some experiments.

4 Results of the soil samples

35 Soil samples have been analyzed on phthalate compounds, using the analytical procedure, described in section 2 'Analytical procedure'. Apart from the phthalates, the percentages of dry matter and total organic carbon (TOC) have been measured.
The results are presented in table A10. The location of the samples is presented in figure 1, see annex 2.
Because of the critical problems with blank samples, the following method of calculation has been used:
For each phthalate the mean blank level has been determined. These values are presented in table 2. For soil samples a concentration has been considered, significantly higher than the blank concentration, when it exceeds this blank concentration with a quantity of t*RSDR:

Conc. [soil] > Conc. [blank] + t*RSDR.

For 5 measurements (4 degrees of freedom) and a probability of 99 %, t equals 3.747.

When a concentration in a soil sample exceeds this value, it is corrected by substracting the mean blank concentration, and then reported as final result.
When a concentration in a soil sample does not exceed this value, it is regarded as not significantly higher than the blank concentration. In this case the result is presented as 'less than reporting value'.

Table A10-B presents intermediate results; all measured concentrations, not significantly higher than the blank, have been removed.
Table A10-C presents the final results. All concentrations of table A10-B have been corrected for the mean blank concentration and checked for the reporting limit. All concentrations, removed from table A10-B, are reported as 'less than reporting limit'.
Because the blank concentrations in the different blanks are comparable, the measured concentrations have been corrected by substracting the mean blank concentration, in stead of the blank concentration, measured in the same series.

Some of the soil samples are duplicate, namely B06 being the duplicate of B01, and B23 being the duplicate of B19.
As duplicate samples have also been used in the analysis of sediment samples, the results of all duplicate samples have been presented separately in table A14. In this way it is possible to make some statements on the reproducibility of the analysis.

5 Validation of the analytical procedure for sediment samples

For the purpose of analyzing sediment samples, limited supplementary validation experiments have been carried out, measuring the recovery of phthalates, spiked on two sediment samples of different matrices. The results are presented in table A11.

Table A11-A presents concentrations in the sediment samples, not corrected for any blank, in µg/l extract and in g/kgdm ('Natural concentration'). The two columns on the right present the theoretical and the measured concentration of the spiking solution, spiked into 50 ml of dichloromethane. (Remark: all volumes of sample extracts are consequently corrected to 50 ml). The volume of spiking solution is equal to the volume used for the soil samples.
In order to determine the recovery for phthalates, the measured concentration is used as 100 % level.

Table A11-B presents results of the spikes, immediately followed by drying with Na_2SO_4 and ASE extraction. Deuterated standards have been added directly before extraction, to the dried sample in the ASE tube. The experiment is carried out in duplicate (columns I and II).
The left part of the table presents raw results in µg/l extract.
The center part presents results in µg/l extract, corrected for the 'natural concentration'. In order to do this, the measured concentration of the spiked sample is expressed as µg/kgds, then the 'natural concentration' substracted, after which the difference is recalculated as µg/l extract. The right part of the table presents the recovery, calculated from these corrected results and the measured 100 % concentration level.

Table A11-C presents the results of a similar experiment, in which, after addition of the spiking solution, all samples were allowed to stand during 48 h in a refrigerator (aging). The spiking solution is added to the wet sample. After the aging period, the spiked samples were dried with Na_2SO_4.

The recoveries were regarded as satisfactory, although some low and extremely high recovery percentages were found for DEHP, DIOP, DINP and DIDP. These are probably caused by matrix effects, high blank signals and difficulties in the integration of the iso mixture peaks in the chromatogram.

6 Results of the sediment samples

36 Sediment samples have been analysed. The sediment samples have been taken on the sampling location and immediately been stored in two dark coloured glass containers. The sampling was carried out, using a metal can, connected to a large stick.
In the laboratory, the two glass containers was centrifuged twice (2 x 20 min at a rotation speed of 3500 min^{-1}). The water layer was decanted and removed. The solid fraction was stored in a deepfreezer (-18 °C) in a dark coloured glass container, closed with a teflon layered lid.

The location of the samples is presented in figure 2, see annex 2.
The results of the sediment samples are presented in table A12. For the calculation, the same method has been used as for the soil samples. Apart from phthalates, the percentages of dry matter, TOC and mineral particles <2 µm have been measured.
For the blank, the same concentrations of phthalates have been used as for the soil samples, because it was observed that the blank remained constant through all analyses.

Of all sediment samples, nine were taken in duplicate, i.e. numbers 3 and 4, 9 and 10, 11 and 12, 13 and 14, 23 and 24, 25 and 26, 27 and 28, 29 and 30, 35 and 36.

Apart from these duplicate samples, 6 out of 36 samples have been analysed in duplicate, from the same glass container. The results of these analyses are presented in table A13. The calculation has been carried out, following the procedure for the other sediment samples and the soil samples.

Table A14 presents the results of all the duplicate sediment and soil samples.

7 Discussion of the results

Along with the samples, blanks have been analyzed to check for any contamination in the analytical procedure. During the course of the project, blank concentrations slightly increased to a rather constant level, as is shown in table 2. However, in some cases, higher concentrations were found for DEHP and, to a smaller extent, DIBP and DBP. These compounds always show the highest concentrations in the blank samples.
This influences the recovery, calculated from analysis of spiked samples, as shown in tables A11-B and A11-C for DEHP in samples sed. 1, where the recovery of DEHP shows high percentages (200 – 300 %) or bad duplicate results (table A12-B, sed. 2).
The wide range of results for these compounds in the blanks, especially for DEHP, shows that the analytical procedure is very sensitive to contamination, and that care must be taken in judging recoveries or measured concentrations for these compounds.

Along with all analyses of phthalate compounds, deuterated internal standards have been used as a tracer for the extraction efficiency or compound loss during analysis. For spiked samples the recoveries vary between 80 and 100 %, depending on the matrix of the sample, especially expressed by TOC and mineral particles <2 μm.
For the soil samples, the recovery of the deuterated samples varies between 80 and 110 %. Here also, the recovery is lower when TOC increases (mineral particles are not measured in these samples). In general, the effect on the recovery of deuterated DEHP is somewhat higher than the effect on the recovery of deuterated DBP.
For the sediment samples, the recovery shows larger variation. The cause for this is the large variation in TOC and mineral particles <2 μm. Again, higher percentages of organic matter and mineral particles <2 μm lead to lower recoveries of the deuterated standards. The effect on the recovery of the deuterated DEHP is more pronounced than on the recovery of the deuterated DBP. In some cases, where the percentages of TOC or mineral particles <2 μm are relatively very high (TOC: >7 %, mineral particles <2 μm: >10 %), the recovery of the deuterated standards becomes lower than 80 %. Close examination of the percentages shows that the presence of mineral particles < 2μm is more important than that of TOC.
For this project the minimum recovery of the deuterated internal standards was set at 50 %. Of the soil samples, none of the samples showed recoveries lower than 50 %. Of the sediment samples five samples did not meet this criterion for one or both of the internal standards (samples S5, S9, S10, S17 and S23). After reanalysis, two samples still did not meet this criterion (samples S5 and S17). The results for these samples should be neglected. The cause for the low recovery must be the high content of mineral particles <2 μm and TOC in these samples, except for sample S23. For this sample, the recovery of deuterated DEHP does not meet the criterion, although the content of TOC and mineral particles <2 μm is not really high. However, duplicate samples 24 an 23, show about equal recoveries, while only deuterated DEHP meets the criterion.

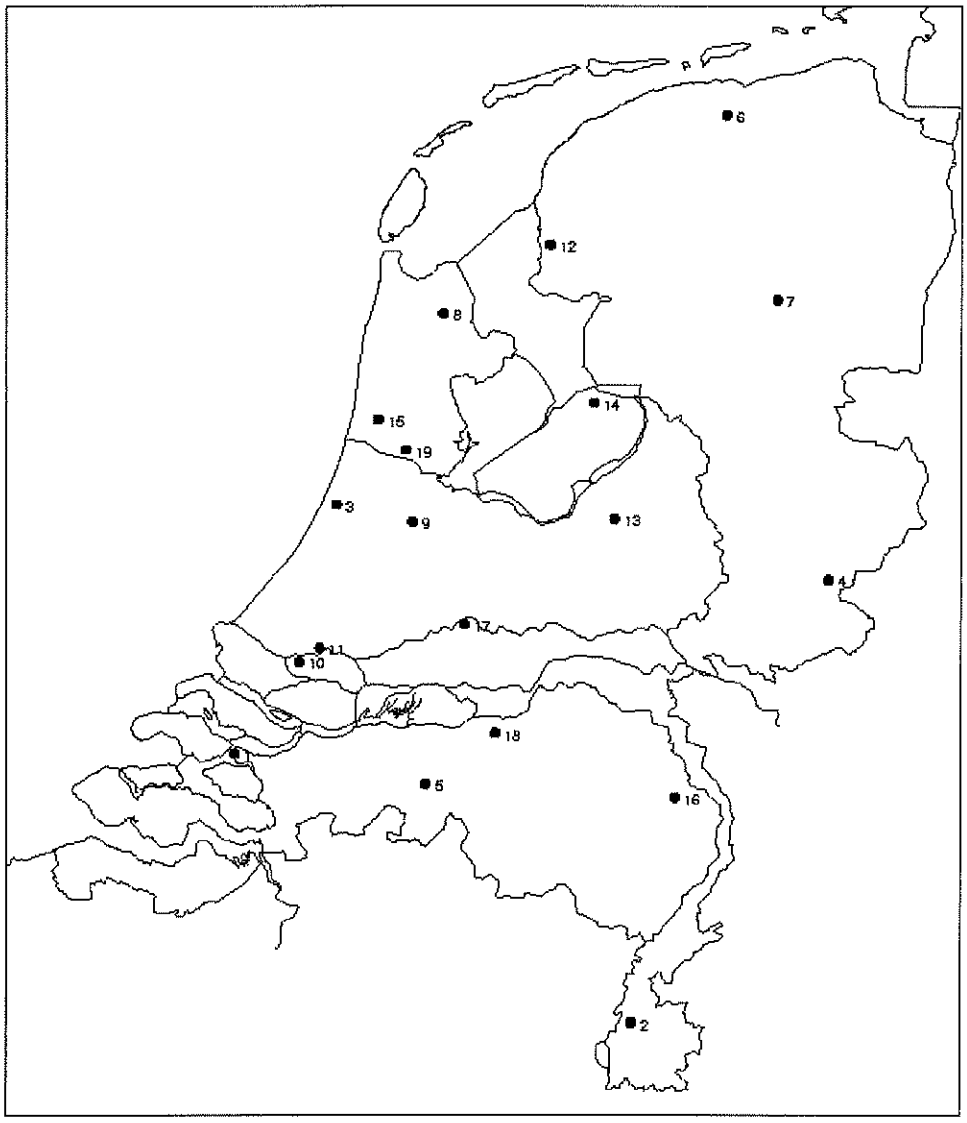
Table 14 shows results for all duplicate analyses. These duplicates concern both samples taken in duplicate and analysed independently, and samples analysed in duplicate.
Generally, the recoveries for the deuterated internal standards of the duplicate samples are comparable. Exceptions are the recoveries for samples S9 and S10 (first analysis, deuterated DBP) and the recoveries for the duplicates of sample 22. Generally, the reanalysis of samples shows better recoveries.

The duplicate results for the samples do not show a good consistancy. Most of the duplicate results match quite well, when they are considered as a concentration level (high level, medium level, low level). However, there are exceptions, e.g. DIHP in sample S9, DINP and DIDP in sample S10.
It should be noted, that the calculation of concentrations of the iso mixtures is a critical factor, especially the integration of the selected ion monitoring (SIM) chromatogram. The integration is carried out using the SIM of the 149 atomic mass ion, where the integration window is determined by an external standard of the same iso mixture. Most of the sediment samples, expecially the ones with a high TOC and mineral particles <2 µm values, show matrix effects in the chromatogram, complicating the integration of the iso mixtures. Here, matrix could be indentified as phthalates, resulting in false positive results, especially in samples with iso phthalates.

Annex 1
Tables 1 – 15.

Annex 2
Figures 1 – 2.

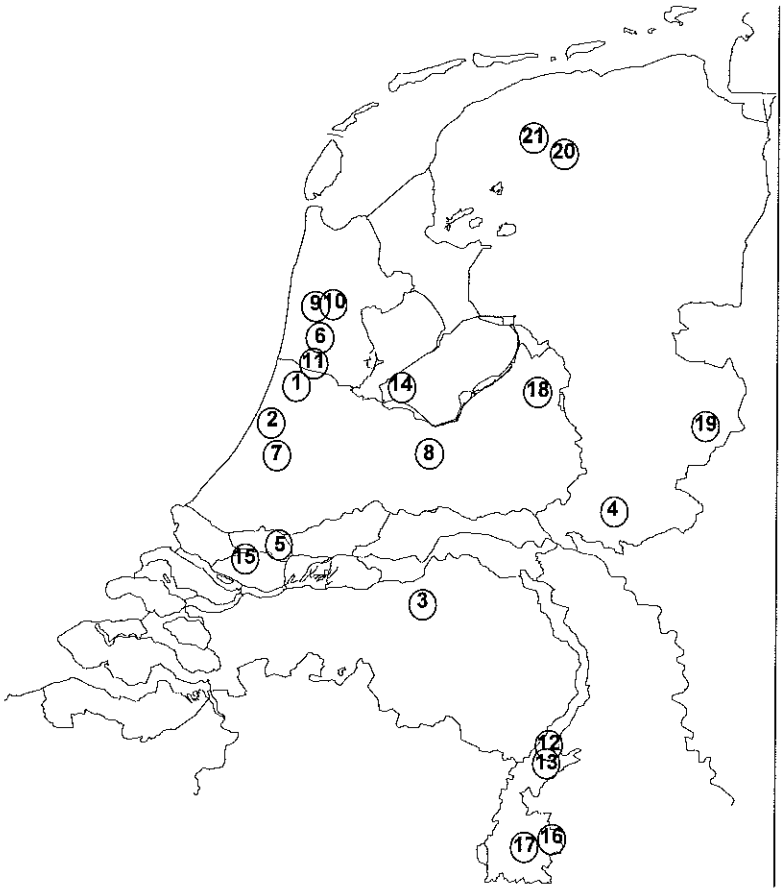
Figure 1. View on the sample locations for the analysis of phthalate esters in Dutch soil.



1. Anna Jacobapolder (17)
2. Beek (8)
3. De Zilk (12,33)
4. Eibergen (9, 32)
5. Gilze-Rijen (10)
6. Kollumerwaard (6)
7. Lheebroekerzand (31,34)
8. Middenmeer (27)
9. Nes aan de Amstel (25)
10. Pernis (15)
11. Rotterdam (16)
12. Schraard (26,30)
13. Speulderveld (13)
14. Swifterbant (28)
15. Uitgeest (1,2,3,5,6,7)
16. Venray (14)
17. Vianen (18,19,20,21,22,23)
18. Vlijmen (24)
19. Zaandam (4)

The number between brackets refer to sample codes of the soil samples.

Figure 2. View on the sample locations for the analysis of phthalate esters in dutch sediments.



1. Haarlem (13,14)
2. Noordwijk (15,16)
3. Rosmalen (22,23,24)
4. Doetinchem (19)
5. Hendrik Ido Ambacht (21)
6. Wormerveer (8)
7. Voorschoten (29,30)
8. Woudenberg (25,26)
9. Alkmaar (9,10)
10. Alkmaar (11,12)
11. Assendelft (7)
12. Herten (3)
13. Herten (4)
14. Almere (27,28)
15. Oud-Beijerland (20)
16. Landgraaf (5)
17. Heerlen (6)
18. Heerde (17)
19. Enschede (18)
20. Opeinde (1)
21. Leeuwarden (2)

The number between brackets refer to sample codes of the sediment samples.

Table A10-A
BBP in soil samples

µg/kgdm	Sample codes
<4	1: Uitgeest, 5 m from motorway
<4	2: Uitgeest, 400 m from motorway
<4	3: Uitgeest, 20 m from motorway
6	4: Zaandam, waste burning installation
<4	5: Uitgeest, 100 m from motorway
<4	6: Uitgeest, 5 m from motorway duplicate of sample 1
<4	7: Uitgeest, 800 m from motorway
<4	8: Beek, Airport
<4	9: Eibergen
10	10: Gilze-Rijen
<4	11: Kollumerwaarde
#	12: De Zilk
#	13: Speulderveld
#	14: Venray
<4	15: Pernis
<4	16: Rotterdam, waste burning installation (AVR)
<4	17: Anna Jacobapolder
<4	18: Vianen, motorway A27, 400 m from motorway
<4	19: Vianen, motorway A27, 10 m from motorway
<4	20: Vianen, motorway A27, 800 m from motorway
5	21: Vianen, motorway A27, 3 m from motorway
<4	22: Vianen, motorway A27, 100 m from motorway
9	23: Vianen, motorway A27, 10 m from motorway
<4	24: Vlijmen
<4	25: Nes aan de Amstel
<4	26: Schraard
<4	27: Middenmeer
<4	28: Swifterbant
	(29: Sample not analyzed)
<4	30: Schraard
<4	31: Lheebroekerzand
6	32: Eibergen
<4	33: De Zilk
<4	34: Lheebroekerzand
<4	35: Bilthoven, along road Utrecht-station Bilthoven

Table A10-A
Phthalates in soil samples
DIOP includes DEHP
Raw results

06_01_99	blank	1	2	3	4	5	6	7	8	9	35
Compound	µg/kgdm	µg/kgdm	µg/kgdm	µg/kgdm	µg/kgdm	µg/kgdm	µg/kgdm	µg/kgdm	µg/kgdm	µg/kgdm	µg/kgdm
DMP	0	0	0	0	0	0	0	0	0	0	0
DMT	0	0	0	0	0	0	0	0	0	0	0
DEP	2.12	3.34	4.92	3.5	3.08	4.77	3.39	6.62	3.77	3.91	4.36
DPP	0	0	0	0	0	0	0	0	0	0	0
DICBP	25.49	35.43	54.04	62.95	39.62	52.1	38.37	68.1	42.03	52.48	49.01
DBP	19.33	41.53	41.72	46.04	41.76	42.69	35.37	55.8	34.64	47.15	43.07
DEHP	25.09	93.47	110.26	66.93	67.58	97.58	144.52	68	69.16	71.59	100.49
BBzP	1.62	5.27	6.41	2.8	7.17	5.62	4.81	3.71	3.89	5.08	5.52
DCHP	4.27	2.21	0	0	0	0	1.03	0	1.48	0	0
DIHP	0	0	0	0	0	0	0	0	0	0	0
DIOP	164.08	150.57	250.81	282.01	124.24	240.12	336.71	319.92	233.62	230.33	170.51
DINP	0	0	0	0	0	0	0	0	0	0	0
DIDP	0	0	0	0	0	0	0	0	0	0	0
D(D4)BP (%)	112.6	106.2	101.8	84.1	107.7	102.2	108.8	87.5	110.6	112.3	106.3
D(D4)EHP (%)	109.2	106.2	103	76.6	102	105.7	105.7	90.8	106.3	115.9	90.5
Dry matter %		88.38	65.67	76.25	88.14	73.28	85.17	63.42	78.87	84.39	84.51
TOC %		2.4	6	12	4	9.7	2.7	11	3	3.3	1.8

07_01_99	blank	10	11	12	13	14	15	16	17	18	19
Compound	µg/kgdm	µg/kgdm	µg/kgdm	µg/kgdm	µg/kgdm	µg/kgdm	µg/kgdm	µg/kgdm	µg/kgdm	µg/kgdm	µg/kgdm
DMP	0	0	0	0	0	0	0	0	0	0	0
DMT	0	0	0	0	0	0	0	0	0	0	0
DEP	2.35	9.22	2.93	4.99	9.03	4.02	3.29	3.8	3.88	5.45	3.47
DPP	0	5.58	0	0	0	0	0	0	0	0	0
DICBP	35.06	89.8	34.58	46.16	71	10.14	51.89	45.6	58.16	76.05	52.43
DBP	24.3	82.76	50.09	49.15	78.2	22.73	37.8	40.24	43.82	59.19	38.05
DEHP	32.66	147.15	50.53	64.69	129.1	124.13	150.08	83.95	85.56	117.18	89.75
BBzP	1.87	11.87	2.49				4	5.84	4.34	5.72	4.3
DCHP	11.76	15.99	3.41	0	0	0	11.52	2.07	0	0	0
DIHP	0	0	0	0	0	0	0	0	0	0	0
DIOP	129.1	365.05	135.78	185.92			208.62	124.16	225.26	290.82	207.61
DINP	0	0	0	0	0	0	0	0	0	0	0
DIDP	0	0	0	0	0	0	0	0	0	0	0
D(D4)BP (%)	108.2	107.3	87.6	105.2	102.3	110.4	101.5	103.2	103.2	98.9	106.3
D(D4)EHP (%)	106.6	116.1	86.2	87.8	100.7	132.1	94.2	97.3	100.3	102.4	90.5
Dry matter %		92.14	83.52	90.95	72.57	96.29	70.3	87.49	81.94	66.69	74.64
TOC %		3.7	1.5	2.9	6.5	1.2	5	2	1.8	8.2	4.6

08_01_99	blank	20	21	22	23	24	25	26	27	28
Compound	µg/kgdm	µg/kgdm	µg/kgdm	µg/kgdm	µg/kgdm	µg/kgdm	µg/kgdm	µg/kgdm	µg/kgdm	µg/kgdm
DMP	0	0	0	0	0	0	0	0	0	0
DMT	0	0	0	0	0	0	0	0	0	0
DEP	2.72	3.46	3.17	5.25	4.5	3.28	3.47	3.62	2.87	2.78
DPP	0	0	0	0	0	0	0	0	0	0
DICBP	36.6	49.76	43.88	59.32	67.12	52.6	52.82	60.25	49.15	35.61
DBP	30.95	39.25	30.86	43.49	51.13	34.7	37.05	42.38	27.43	22.83
DEHP	44.83	62.45	62.58	73.86	108.3	58.93	79.94	50.94	43.32	41.93
BBzP	3.5	3.43	8.78	5.18	12.8	6.19	4.68	7.36	3	3
DCHP	1.24	1.7	1.13	1.53	1.57	2.25	4.62	0	0	0
DIHP	0	0	0	0	0	0	0	0	0	0
DIOP	151.43	192.04	146.02	195.14	235.15	146.96	183.31	199.39	225.13	141.89
DINP	0	0	0	0	0	0	0	0	0	0
DIDP	0	0	0	0	0	0	0	0	0	0
D(D4)BP (%)	105.6	103	99.5	93.7	92.8	102.8	98.8	92.7	99	90.7
D(D4)EHP (%)	104.3	106	99.1	97	95.1	105.4	104.6	79.5	99.4	91.6
Dry matter %		82.48	87.44	78.88	75.4	89.94	85.3	69.72	85.92	82.44
TOC %		2.9	1.6	4.6	4.7	2.5	2.1	5.9	1.2	2.2

12_01_99	blank	30	31	32	33	34
Compound	µg/kgdm	µg/kgdm	µg/kgdm	µg/kgdm	µg/kgdm	µg/kgdm
DMP	0	0	0	0	0	0
DMT	0	0	0	0	0	0
DEP	2.72	5.16	5.44	3.97	2.77	2.43
DPP	0	0	0	0	0	0
DICBP	37.83	50.56	95.56	56.23	38.67	30.92
DBP	25.31	34.11	60.18	38.38	20	19.37
DEHP	36.75	154.13	78.7	39.53	33.41	205.46
BBzP	4.12	6.13	7.63	10.42	3.57	4.15
DCHP	0	0	0	0	0	0
DIHP	0	0	0	0	0	0
DIOP	153.16	241.51	391.49	183.02	127.9	197.81
DINP	0	0	0	0	0	0
DIDP	0	0	0	0	0	0
D(D4)BP (%)	99.2	93.1	101.4	100.9	105.7	98
D(D4)EHP (%)	105	85.8	97.3	84.8	112.7	90.9
Dry matter %		72.61	64.65	87.99	95.69	96.55
TOC %		6.4	6.4	2.4	0.9	<0.5

Table A10-B
Phthalates in soil samples
DIOP includes DEHP
Remaining results, significantly higher than blank results, not corrected for blanks

Compound	1 µg/kgdm	2 µg/kgdm	3 µg/kgdm	4 µg/kgdm	5 µg/kgdm	6 µg/kgdm	7 µg/kgdm	8 µg/kgdm	9 µg/kgdm	35 µg/kgdm
DMP										
DMT										
DEP		4.92			4.77		6.62	3.77	3.91	4.36
DPP										
DICBP		54.04	62.95				68.1			
DBP			46.04				55.8		47.15	
DEHP	93.47	110.26			97.58	144.52		69.16	71.59	100.49
BBzP				7.17						
DCHP										
DIHP										
DIOP		250.81	282.01		240.12	336.71	319.92	233.62	230.33	
DINP										
DIDP										
D(D4)BP (%)	106.2	101.8	84.1	107.7	102.2	108.8	87.5	110.6	112.3	106.3
D(D4)EHP (%)	106.2	103	76.6	102	105.7	105.7	90.8	106.3	115.9	90.5
Dry matter %	88.38	65.67	76.25	88.14	73.28	85.17	63.42	78.87	84.39	84.51
TOC %	2.4	6	12	4	9.7	2.7	11	3	3.3	1.8

Compound	10 µg/kgdm	11 µg/kgdm	12 µg/kgdm	13 µg/kgdm	14 µg/kgdm	15 µg/kgdm	16 µg/kgdm	17 µg/kgdm	18 µg/kgdm	19 µg/kgdm
DMP										
DMT										
DEP	9.22		4.99	9.03	4.02		3.8	3.88	5.45	
DPP	5.58									
DICBP	89.8			71				58.16	76.05	
DBP	82.76	50.09	49.15	78.2					59.19	
DEHP	147.15			129.1	124.13	150.08	83.95	85.56	117.18	89.75
BBzP	11.87									
DCHP										
DIHP										
DIOP	365.05					208.62		225.26	290.82	207.61
DINP										
DIDP										
D(D4)BP (%)	107.3	87.6	105.2	102.3	110.4	101.5	103.2	103.2	98.9	106.3
D(D4)EHP (%)	116.1	86.2	87.8	100.7	132.1	94.2	97.3	100.3	102.4	90.5
Dry matter %	92.14	83.52	90.95	72.57	96.29	70.3	87.49	81.94	66.69	74.64
TOC %	3.7	1.5	2.9	6.5	1.2	5	2	1.8	8.2	4.6

Compound	20 µg/kgdm	21 µg/kgdm	22 µg/kgdm	23 µg/kgdm	24 µg/kgdm	25 µg/kgdm	26 µg/kgdm	27 µg/kgdm	28 µg/kgdm
DMP									
DMT									
DEP			5.25	4.5			3.62		
DPP									
DICBP			59.32	67.12			60.25		
DBP				51.13					
DEHP			73.86	108.3		79.94			
BBzP		8.78		12.8			7.36		
DCHP									
DIHP									
DIOP				235.15				225.13	
DINP									
DIDP									
D(D4)BP (%)	103	99.5	93.7	92.8	102.8	98.8	92.7	99	90.7
D(D4)EHP (%)	106	99.1	97	95.1	105.4	104.6	79.5	99.4	91.6
Dry matter %	82.48	87.44	78.88	75.4	89.94	85.3	69.72	85.92	82.44
TOC %	2.9	1.6	4.6	4.7	2.5	2.1	5.9	1.2	2.2

Compound	30 µg/kgdm	31 µg/kgdm	32 µg/kgdm	33 µg/kgdm	34 µg/kgdm
DMP					
DMT					
DEP	5.16	5.44	3.97		
DPP					
DICBP		95.56	56.23		
DBP		60.18			
DEHP	154.13	78.7			205.46
BBzP		7.63	10.42		
DCHP					
DIHP					
DIOP	241.51	391.49			197.81
DINP					
DIDP					
D(D4)BP (%)	93.1	101.4	100.9	105.7	98
D(D4)EHP (%)	85.8	97.3	84.8	112.7	90.9
Dry matter %	72.61	64.65	87.99	95.69	96.55
TOC %	6.4	6.4	2.4	0.9	<0.5

Compound	27 µg/kgdm	28 µg/kgdm	30 µg/kgdm	31 µg/kgdm	32 µg/kgdm	33 µg/kgdm	34 µg/kgdm	35 µg/kgdm
DMP	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5
DMT	<2	<2	<2	<2	<2	<2	<2	<2
DEP	<4	<4	<4	<4	<4	<4	<4	<4
DPP	<3	<3	<3	<3	<3	<3	<3	<3
DICBP	<15	<15	<15	58	18	<15	<15	<15
DBP	<15	<15	<15	35	<15	<15	<15	<15
DEHP	<25	<25	117	42	<25	<25	169	75
BBzP	<4	<4	<4	<4	6	<4	<4	<4
DCHP	<15	<15	<15	<15	<15	<15	<15	<15
DIHP	<6	<6	<6	<6	<6	<6	<6	<6
DIOP	74	<45	88	238	<45	<45	45	<45
DINP	<25	<25	<25	<25	<25	<25	<25	<25
DIDP	<15	<15	<15	<15	<15	<15	<15	<15
D(D4)BP (%)	99	90.7	93.1	101.4	100.9	105.7	98	106.3
D(D4)EHP (%)	99.4	91.6	85.8	97.3	84.8	112.7	90.9	90.5
Dry matter %	85.92	82.44	72.61	64.65	87.99	95.69	96.55	84.51
TOC %	1.2	2.2	6.4	6.4	2.4	0.9	<0.5	1.8

#: integration not possible, due to matrix interferences

from 33 sites
34 samples only 5 w/ BBP above detection limit
range ~~<0.004 to 0.009 mg/kg~~
<0.004 to 0.009 mg/kg

Compound	18 µg/kgdm	19 µg/kgdm	20 µg/kgdm	21 µg/kgdm	22 µg/kgdm	23 µg/kgdm	24 µg/kgdm	25 µg/kgdm	26 µg/kgdm
DMP	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5
DMT	<2	<2	<2	<2	<2	<2	<2	<2	<2
DEP	<4	<4	<4	<4	<4	<4	<4	<4	<4
DPP	<3	<3	<3	<3	<3	<3	<3	<3	<3
DICBP	41	<15	<15	<15	23	31	<15	<15	24
DBP	35	<15	<15	<15	<15	20	<15	<15	<15
DEHP	85	57	<25	<25	29	63	<25	35	<25
BBzP	<4	<4	<4	5	<4	9	<4	<4	<4
DCHP	<15	<15	<15	<15	<15	<15	<15	<15	<15
DIHP	<6	<6	<6	<6	<6	<6	<6	<6	<6
DIOP	162	79	<45	<45	<45	84	<45	<45	<45
DINP	<25	<25	<25	<25	<25	<25	<25	<25	<25
DIDP	<15	<15	<15	<15	<15	<15	<15	<15	<15
D(D4)BP (%)	98.9	106.3	103	99.5	93.7	92.8	102.8	98.8	92.7
D(D4)EHP (%)	102.4	90.5	106	99.1	97	95.1	105.4	104.6	79.5
Dry matter %	66.69	74.64	82.48	87.44	78.88	75.4	89.94	85.3	69.72
TOC %	8.2	4.6	2.9	1.6	4.6	4.7	2.5	2.1	5.9

Compound	10 µg/kgdm	11 µg/kgdm	12 µg/kgdm	13 µg/kgdm	14 µg/kgdm	15 µg/kgdm	16 µg/kgdm	17 µg/kgdm
DMP	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5
DMT	<2	<2	<2	<2	<2	<2	<2	<2
DEP	7	<4	<4	7	<4	<4	<4	<4
DPP	6	<3	<3	<3	<3	<3	<3	<3
DICBP	55	<15	<15	36	<15	<15	<15	23
DBP	58	26	25	54	<15	<15	<15	<15
DEHP	114	<25	32	96	91	117	51	53
BBzP	10	<4	#	#	#	<4	<4	<4
DCHP	<15	<15	<15	<15	<15	<15	<15	<15
DIHP	<6	<6	<6	<6	<6	<6	<6	<6
DIOP	236	<45	<45	#	#	80	<45	96
DINP	<25	<25	<25	<25	<25	<25	<25	<25
DIDP	<15	<15	<15	<15	<15	<15	<15	<15
D(D4)BP (%)	107.3	87.6	105.2	102.3	110.4	101.5	103.2	103.2
D(D4)EHP (%)	116.1	86.2	87.8	100.7	132.1	94.2	97.3	100.3
Dry matter %	92.14	83.52	90.95	72.57	96.29	70.3	87.49	81.94
TOC %	3.7	1.5	2.9	6.5	1.2	5	2	1.8

Table A10-C
Phthalates in soil samples
DIOP includes DEHP
Net results, corrected for blanks

Compound	1 µg/kgdm	2 µg/kgdm	3 µg/kgdm	4 µg/kgdm	5 µg/kgdm	6 µg/kgdm	7 µg/kgdm	8 µg/kgdm	9 µg/kgdm
DMP	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5
DMT	<2	<2	<2	<2	<2	<2	<2	<2	<2
DEP	<4	<4	<4	<4	<4	<4	5	<4	<4
DPP	<3	<3	<3	<3	<3	<3	<3	<3	<3
DICBP	<15	29	37	<15	<15	<15	43	<15	<15
DBP	<15	<15	27	<15	<15	<15	36	<15	28
DEHP	68	85	<25	<25	72	119	<25	44	47
BBzP	<4	<4	<4	6	<4	<4	<4	<4	<4
DCHP	<15	<15	<15	<15	<15	<15	<15	<15	<15
DIHP	<6	<6	<6	<6	<6	<6	<6	<6	<6
DIOP	<45	87	118	<45	76	173	156	70	66
DINP	<25	<25	<25	<25	<25	<25	<25	<25	<25
DIDP	<15	<15	<15	<15	<15	<15	<15	<15	<15
D(D4)BP (%)	106.2	101.8	84.1	107.7	102.2	108.8	87.5	110.6	112.3
D(D4)EHP (%)	106.2	103	76.6	102	105.7	105.7	90.8	106.3	115.9
Dry matter %	88.38	65.67	76.25	88.14	73.28	85.17	63.42	78.87	84.39
TOC %	2.4	6	12	4	9.7	2.7	11	3	3.3

Sample codes:

Q1: Quartz 1
Q2: Quartz 2
1. Opeinder Kanaal (canal). Sampled 18/1/99.
2. Hantummervaart (canal). Sampled 18/1/99.
3. River Maas near Ool. Sampled 21/1/99. Duplicate of sediment sample 4. Sandy sediment.
4. River Maas near Ool. Sampled 21/1/99. Duplicate of sediment sample 3. Sandy sediment.
5. Castle Strijthagen in Landgraaf. Sampled 21/1/99. Sample contains leaf material.
6. Fishing club N.O. Hoek in Heerlen/Landgraaf. Sampled 21/1/99. Sandy sediment.
7. Assendelft. Sampled 22/1/99.
8. Wormerveer. Sampled 22/1/99.
9. Alkmaar - Hoornse/Hoevevaart (canal). Sampled 22/1/99. Duplicate of sediment sample 10.
10. Alkmaar - Hoornse/Hoevevaart (canal). Sampled 22/1/99. Duplicate of sediment sample 9.
11. Alkmaar - Noord-Hollands kanaal (canal). Sampled 22/1/99. Duplicate of sediment sample 12.
12. Alkmaar - Noord-Hollands kanaal (canal). Sampled 22/1/99. Duplicate of sediment sample 11.
13. Haarlem - Ringvaart Vijfhuizen (canal). Sampled 22/1/99. Duplicate of sediment sample 14.
14. Haarlem - Ringvaart Vijfhuizen (canal). Sampled 22/1/99. Duplicate of sediment sample 13.
15. Noordzijderpolder in Noordwijk. Sampled 22/1/99.
16. Leidse trekvaart (canal). Sampled 27/1/99.
17. Apeldoorns Kanaal (canal), Hoorn neighbourhood in the Heerde district. Sampled 28/1/99.
18. Fishing club VIOS in Enschede. City lake Enschede. Sampled 28/1/99.
19. Yacht-basin Oude IJssel te Doetinchem. Sampled 28/1/99.
20. De Zoomwijckvijver (a more than 10 year old pond in Oud-Beijerland). Sampled 29/1/99.
21. Fishing club ERHV de Waal in Ridderkerk. Location: Het Waaltje, Hendrik Ido Ambacht. Sampled 29/1/99.
22. River Dommel , Vught, near motorway A2. Sampled 30/1/99.
23. River AA , near Rosmalen. Sampled 30/1/99. Duplicate of sediment sample 24.
24. River AA , near Rosmalen. Sampled 30/1/99. Duplicate of sediment sample 23.
25. Pond Wipperveld located Kennedylaan in Woudenberg. Sampled 5/2/99. Duplicate of sediment sample 26.
26. Pond Wipperveld located Kennedylaan in Woudenberg. Sampled 5/2/99. Duplicate of sediment sample 25.
27. Canal around Almere, Harlingensingel 31, Almere. Sampled 5/2/99. Duplicate of sediment sample 28.
28. Canal around Almere, Harlingensingel 31, Almere. Sampled 5/2/99. Duplicate of sediment sample 27.
29. Fishing club De Vliet . Sixlaan 33, Voorschoten. Sampled 8/2/99. Duplicate of sediment sample 30.
30. Fishing club De Vliet . Sixlaan 33, Voorschoten. Sampled 8/2/99. Duplicate of sediment sample 29.
31. Production site A (Regret). Sampled 23/2/99.
32. Production site A (Village). Sampled 23/2/99.
33. Production site B. Sample location: Terra Nova Bridge crossing 2 km above plant. Sampled 4/3/99.
34. Production site B. Sample location: Terra Nova Bridge crossing 2 km down stream from the plant. Sampled 4/3/99.
35. Production site A (Regret). Waste Water Treatment Plant. Sampled 25/3/99. Duplicate of sediment sample 36.
36. Production site A (Regret). Waste Water Treatment Plant. Sampled 25/3/99. Sample is also being analysed by LOC. Duplicate of sediment sample 35.

Table sediments A12-A

Raw results, concentrations in sediment samples

DIOP includes DEHP

	blank +t.RSDR	Q1 µg/kgdm	Q2 µg/kgdm	S1 µg/kgdm	S2 µg/kgdm	S3 µg/kgdm	S4 µg/kgdm	S5 µg/kgdm	S6 µg/kgdm	S7 µg/kgdm
DMP	0	0	0	0	0	0	0	0	0	0
DMT	0	0	0	0	0	0	0	0	0	46.81
DEP	3.6	4.24	5.58	6.57	8.3	13.05	12.31	9.7	7.36	13.33
DPP	0	0	0	0	0	0	0	0	0	0
DIBP	53.2	43.92	53.06	58.97	69.86	84.75	99.65	69.73	72.34	138
DBP	44.6	35.55	48.94	64.72	93.21	89.26	142.59	56.62	70.2	123.55
DEHP	68.4	23.18	19.16	99.34	50.46	1460.54	1946.56	34.81	69.85	137.08
BBzP	7.1	10.71	5.27	4.88	4.5	10.79	17.1	0	0	0
DCHP	21.6	0	0	0	0	0	11.77	0	0	0
DIHP	0	0	0	0	0	0	0	0	0	0
DIOP	200	41	26.15	174.04	87.27	1339.39	2672.12	179.57	102.83	723.79
DINP	0	0	0	150.27	93.16	1135.32	1152.35	0	233.73	0
DIDP	19.6	0	0	0	0	709.2	664.72	0	0	0
D(ring-D4)BP %		98	96.8	81.5	73.9	78	82.3	84.1	99.3	91
D(ring-D4)EHP %		100.8	96.2	79.3	66.4	64.7	68.6	38.5	86.6	53.4

Sediments

	S8 µg/kgdm	S9 µg/kgdm	S10 µg/kgdm	S11 µg/kgdm	S12 µg/kgdm	S13 µg/kgdm	S14 µg/kgdm	S15 µg/kgdm	S16 µg/kgdm	S17 µg/kgdm
DMP	0	0	0	0	0	0	0	0	0	0
DMT	0	0	0	0	0	0	1.61	3.43	1.75	0
DEP	68.06	9	14.02	8.27	3.53	4.73	12.16	5.25	5.78	*
DPP	0	0	0	0	0	0	0	0	0	0
DIBP	294.28	92.4	89.56	56.01	50.59	44.06	86.27	50.43	61.46	332.35
DBP	324.6	75.81	85.44	64.32	43.9	62.43	103.12	41.01	47.86	190.29
DEHP	1123.81	221.28	542.35	52.44	48.16	55.59	85.42	86.15	158.63	229.55
BBzP	13.57	0	0	0	8.19	10.35	49.26	3.88	4.07	0
DCHP	0	0	0	0	0	0	0	0	0	0
DIHP	0	0	0	0	0	0	0	0	0	0
DIOP	890.06	264.4	477.93	257.4	360.01	106.4	173.57	149.04	347.03	424.75
DINP	6160.56	1046	0	73.04	0	0	237.31	0	106.86	2566.24
DIDP	1109.39	321.03	0	0	0	0	87.66	0	0	0
D(ring-D4)BP %	89.4	40.1	82.42	90.1	94.7	87.9	91.5	73.5	77.8	21.1
D(ring-D4)EHP %	95.9	31.4	33.1	83.6	84.6	60	77.5	64.4	65.9	28.2

Sediments

	S18 µg/kgdm	S19 µg/kgdm	S20 µg/kgdm	S21 µg/kgdm	S22 µg/kgdm	S23 µg/kgdm	S24 µg/kgdm	S25 µg/kgdm	S26 µg/kgdm	S27 µg/kgdm
DMP	0	0	0	0	0	0	0	0	0	0
DMT	0	1.27	0	5.36	9.28	0	0	0	6.75	6.36
DEP	7.21	12.44	17.84	16.29	9.56	3.83	4.08	9.8	8.97	6.52
DPP	0	0	0	0	0	0	0	0	0	0
DIBP	63.33	66.94	42.95	93.72	77.49	57.3	41.15	130.91	96.8	70.8
DBP	54.91	57.63	32.39	82.13	56.45	36.04	24.49	64.56	198.31	66.69
DEHP	170	608.05	150.02	218.61	664.24	51.36	56.95	639.02	532.89	258.82
BBzP	7.21	5.44	3.04	13.23	15.37	3.02	1.42	10.74	8.2	8.11
DCHP	10.99	0	0	0	0	0	0	5.56	0	113.66
DIHP	0	0	0	0	0	0	0	0	0	0
DIOP	455.73	889.66	287.94	618.98	633.36	374.93	194.97	834.49	981.36	507.06
DINP	0	0	0	214.35	427.88	0	0	331.1	273.15	173.33
DIDP	0	0	0	0	546.45	0	0	0	0	0
D(ring-D4)BP %	84.7	77.4	79	75.6	67.4	82.8	81.9	83.3	88	81.9
D(ring-D4)EHP %	90.9	51.6	89.5	73.8	54.9	43	53.7	68.8	81.7	68.1

Sediments

	S28 µg/kgdm	S29 µg/kgdm	S30 µg/kgdm	S31 µg/kgdm	S32 µg/kgdm	S33 µg/kgdm	S34 µg/kgdm	S35 µg/kgdm	S36 µg/kgdm
DMP	0	0	0	0	0	2.97	2.72	2.33	2.63
DMT	0	0	5.96	7.48	17.93	7.42	5.98	9.64	10.13
DEP	15.97	5.78	3.85	46.94	54.57	36.1	36.44	48.2	51
DPP	0	0	0	0	0	0	0	0	0
DIBP	134.08	49.94	49.67	354.8	423.55	299.68	320.93	371.27	420.02
DBP	112.14	48.51	44.65	279.28	731.86	240.58	232.81	746.54	571.16
DEHP	338.86	95.35	110.5	1992.04	7681.86	497.73	289.38	9792.07	7210.54
BBzP	4.39	6.72	4.36	39.46	80.51	34.37	28.01	50.85	37.88
DCHP	0	0	0	0	0	0	0	0	0
DIHP	0	0	0	0	492.61	102.36	0	863.54	587.28
DIOP	829.28	108.76	401.75	1175.97	17410.49	538.53	300.8	18451.71	15699.53
DINP	263.41	43.4	0	869.14	11556.75	237.12	48.14	9842.93	10220.09
DIDP	210.18	0	0	1074.94	42280.01	143.9	0	27261.58	28923.54
D(ring-D4)BP %	92.6	77.8	80.1	74.7	74.6	96.3	85.5	74.8	75
D(ring-D4)EHP %	51.6	62	64.3	74.1	78	94.2	83	73.5	73.8

*: matrix interference

Table sediments A12-B

Results, significantly higher than blank

DIOP includes DEHP

	blank +t.RSDR	Q1 µg/kgdm	Q2 µg/kgdm	S1 µg/kgdm	S2 µg/kgdm	S3 µg/kgdm	S4 µg/kgdm	S5 µg/kgdm	S6 µg/kgdm	S7 µg/kgdm
DMP	0									
DMT	0									46.81
DEP	3.6	4.24	5.58	6.57	8.3	13.05	12.31	9.7	7.36	13.33
DPP	0									
DIBP	53.2			58.97	69.86	84.75	99.65	69.73	72.34	138
DBP	44.6		48.94	64.72	93.21	89.26	142.59	56.62	70.2	123.55
DEHP	68.4	23.18	19.16	99.34		1460.54	1946.56		69.85	137.08
BBzP	7.1	10.71				10.79	17.1			
DCHP	21.6									
DIHP	0									
DIOP	200					1339.39	2672.12			723.79
DINP	0			150.27	93.16	1135.32	1152.35		233.73	
DIDP	19.6					709.2	664.72			
D(ring-D4)BP %		98	96.8	81.5	73.9	78	82.3	84.1	99.3	91
D(ring-D4)EHP %		100.8	96.2	79.3	66.4	64.7	68.6	38.5	86.6	53.4

Table sediments A12-B

Results, significantly higher than blank

DIOP includes DEHP

	blank +t.RSDR	Q1 µg/kgdm	Q2 µg/kgdm	S1 µg/kgdm	S2 µg/kgdm	S3 µg/kgdm	S4 µg/kgdm	S5 µg/kgdm	S6 µg/kgdm	S7 µg/kgdm
DMP	0									
DMT	0									46.81
DEP	3.6	4.24	5.58	6.57	8.3	13.05	12.31	9.7	7.36	13.33
DPP	0									
DIBP	53.2			58.97	69.86	84.75	99.65	69.73	72.34	138
DBP	44.6		48.94	64.72	93.21	89.26	142.59	56.62	70.2	123.55
DEHP	68.4	23.18	19.16	99.34		1460.54	1946.56		69.85	137.08
BBzP	7.1	10.71				10.79	17.1			
DCHP	21.6									
DIHP	0									
DIOP	200					1339.39	2672.12			723.79
DINP	0			150.27	93.16	1135.32	1152.35		233.73	
DIDP	19.6					709.2	664.72			
D(ring-D4)BP %		98	96.8	81.5	73.9	78	82.3	84.1	99.3	91
D(ring-D4)EHP %		100.8	96.2	79.3	66.4	64.7	68.6	38.5	86.6	53.4

Sediments

	S8 µg/kgdm	S9 µg/kgdm	S10 µg/kgdm	S11 µg/kgdm	S12 µg/kgdm	S13 µg/kgdm	S14 µg/kgdm	S15 µg/kgdm	S16 µg/kgdm	S17 µg/kgdm
DMP										
DMT							1.61	3.43	1.75	
DEP	68.06	9	14.02	8.27		4.73	12.16	5.25	5.78	
DPP										
DIBP	294.28	92.4	89.56	56.01			86.27		61.46	332.35
DBP	324.6	75.81	85.44	64.32		62.43	103.12		47.86	190.29
DEHP	1123.81	221.28	542.35				85.42	86.15	158.63	229.55
BBzP	13.57				8.19	10.35	49.26			
DCHP										
DIHP										
DIOP	890.06	264.4	477.93	257.4	360.01				347.03	424.75
DINP	6160.56	1046		73.04			237.31		106.86	2566.24
DIDP	1109.39	321.03					87.66			
D(ring-D4)BP %	89.4	40.1	82.42	90.1	94.7	87.9	91.5	73.5	77.8	21.1
D(ring-D4)EHP %	95.9	31.4	33.1	83.6	84.6	60	77.5	64.4	65.9	28.2

Sediments

	S18 µg/kgdm	S19 µg/kgdm	S20 µg/kgdm	S21 µg/kgdm	S22 µg/kgdm	S23 µg/kgdm	S24 µg/kgdm	S25 µg/kgdm	S26 µg/kgdm	S27 µg/kgdm
DMP										
DMT		1.27		5.36	9.28				6.75	6.36
DEP	7.21	12.44	17.84	16.29	9.56	3.83	4.08	9.8	8.97	6.52
DPP										
DIBP	63.33	66.94		93.72	77.49	57.3		130.91	96.8	70.8
DBP	54.91	57.63		82.13	56.45			64.56	198.31	66.69
DEHP	170	608.05	150.02	218.61	664.24			639.02	532.89	258.82
BBzP	7.21			13.23	15.37			10.74	8.2	8.11
DCHP										113.66
DIHP										
DIOP	455.73	889.66	287.94	618.98	633.36	374.93		834.49	981.36	507.06
DINP				214.35	427.88			331.1	273.15	173.33
DIDP					546.45					
D(ring-D4)BP %	84.7	77.4	79	75.6	67.4	82.8	81.9	83.3	88	81.9
D(ring-D4)EHP %	90.9	51.6	89.5	73.8	54.9	43	53.7	68.8	81.7	68.1

Sediments

	S28 µg/kgdm	S29 µg/kgdm	S30 µg/kgdm	S31 µg/kgdm	S32 µg/kgdm	S33 µg/kgdm	S34 µg/kgdm	S35 µg/kgdm	S36 µg/kgdm
DMP						2.97	2.72	2.33	2.63
DMT			5.96	7.48	17.93	7.42	5.98	9.64	10.13
DEP	15.97	5.78	3.85	46.94	54.57	36.1	36.44	48.2	51
DPP									
DIBP	134.08			354.8	423.55	299.68	320.93	371.27	420.02
DBP	112.14	48.51		279.28	731.86	240.58	232.81	746.54	571.16
DEHP	338.86	95.35	110.5	1992.04	7681.86	497.73	289.38	9792.07	7210.54
BBzP				39.46	80.51	34.37	28.01	50.85	37.88
DCHP									
DIHP					492.61	102.36		863.54	587.28
DIOP	829.28		401.75	1175.97	17410.49	538.53	300.8	18451.71	15699.53
DINP	263.41	43.4		869.14	11556.75	237.12	48.14	9842.93	10220.09
DIDP	210.18			1074.94	42280.01	143.9		27261.58	28923.54
D(ring-D4)BP %	92.6	77.8	80.1	74.7	74.6	96.3	85.5	74.8	75
D(ring-D4)EHP %	51.6	62	64.3	74.1	78	94.2	83	73.5	73.8

Table sediments A12-C DIOP includes DEHP

Final results, corrected for blank

	Rep. limit µg/kgdm	Q1 µg/kgdm	Q2 µg/kgdm	S1 µg/kgdm	S2 µg/kgdm	S3 µg/kgdm	S4 µg/kgdm	S5 µg/kgdm	S6 µg/kgdm	S7 µg/kgdm
DMP	0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5
DMT	2	<2	<2	<2	<2	<2	<2	<2	<2	47
DEP	4	<4	<4	4	6	11	10	7	5	11
DPP	3	<3	<3	<3	<3	<3	<3	<3	<3	<3
DIBP	15	<15	<15	23	34	49	64	34	36	102
DBP	15	<15	25	41	69	65	119	33	46	100
DEHP	25	<25	<25	64	<25	1426	1912	<25	35	102
BBzP	4	8	<4	<4	<4	8	14	<4	<4	<4
DCHP	15	<15	<15	<15	<15	<15	<15	<15	<15	<15
DIHP	6	<6	<6	<6	<6	<6	<6	<6	<6	<6
DIOP	45	<45	<45	<45	<45	1339	2672	<45	<45	724
DINP	25	<25	<25	150	93	1135	1152	<25	234	<25
DIDP	15	<15	<15	<15	<15	709	665	<15	<15	<15
D(ring-D4)BP %		98	96.8	81.5	73.9	78	82.3	84.1	99.3	91
D(ring-D4)EHP %		100.8	96.2	79.3	66.4	64.7	68.6	38.5	86.6	53.4
dry matter %		100	79.4	77.6	64.6	56.4	58.7	62.6	73.3	41.7
TOC %		<0.5	<0.5	1.9	2	5.1	5.4	4.3	2.5	8.3
mineral particles <µm %		-	-	7.5	19	21	19	12	6.5	26

BBP 36 samples from 27 sites
Range <0.004 mg/kg to 0.078 mg/kg
w/ 17 of 36 samples having detectable quantities of BBP.

Sediments

	Rep. limit µg/kgdm	S8 µg/kgdm	S9 µg/kgdm	S10 µg/kgdm	S11 µg/kgdm	S12 µg/kgdm	S13 µg/kgdm	S14 µg/kgdm	S15 µg/kgdm	S16 µg/kgdm
DMP	0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5
DMT	2	<2	<2	<2	<2	<2	<2	<2	3	<2
DEP	4	66	7	12	6	<4	<4	10	<4	<4
DPP	3	<3	<3	<3	<3	<3	<3	<3	<3	<3
DIBP	15	258	56	54	20	<15	<15	50	<15	25
DBP	15	301	52	61	40	<15	38	79	<15	24
DEHP	25	1089	186	507	<25	<25	<25	50	51	124
BBzP	4	11	<4	<4	<4	5	8	46	<4	<4
DCHP	15	<15	<15	<15	<15	<15	<15	<15	<15	<15
DIHP	6	<6	<6	<6	<6	<6	<6	<6	<6	<6
DIOP	45	890	264	478	257	360	<45	<45	<45	347
DINP	25	6161	1046	<25	73	<25	<25	237	<25	107
DIDP	15	1109	321	<15	<15	<15	<15	88	<15	<15
D(ring-D4)BP %		89.4	40.1	82.42	90.1	94.7	87.9	91.5	73.5	77.8
D(ring-D4)EHP %		95.9	31.4	33.1	83.6	84.6	60	77.5	64.4	65.9
dry matter %		42.2	39.8	58.3	78.5	79.8	73.2	75.4	74.1	78.4
TOC %		14	6.1	5	0.5	0.5	1.9	1.4	0.8	0.9
mineral particles <µm %		8.8	25	26	2.2	2	2.9	3.1	1.7	2.7

Sediments

	Rep. limit µg/kgdm	S18 µg/kgdm	S19 µg/kgdm	S20 µg/kgdm	S21 µg/kgdm	S22 µg/kgdm	S23 µg/kgdm	S24 µg/kgdm	S25 µg/kgdm	S26 µg/kgdm
DMP	0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5
DMT	2	<2	<2	<2	5	9	<2	<2	<2	7
DEP	4	5	10	15	14	7	<4	<4	7	6
DPP	3	<3	<3	<3	<3	<3	<3	<3	<3	<3
DIBP	15	27	31	<15	58	41	21	<15	95	61
DBP	15	31	34	<15	58	32	<15	<15	41	174
DEHP	25	135	573	115	184	629	-35	<25	604	498
BBzP	4	4	<4	<4	10	13	<4	<4	8	5
DCHP	15	<15	<15	<15	<15	<15	<15	<15	<15	<15
DIHP	6	<6	<6	<6	<6	<6	<6	<6	<6	<6
DIOP	45	456	890	288	619	633	375	<45	834	981
DINP	25	<25	<25	<25	214	428	<25	<25	331	273
DIDP	15	<15	<15	<15	<15	546	<15	<15	<15	<15
D(ring-D4)BP %		84.7	77.4	79	75.6	67.4	82.8	81.9	83.3	88
D(ring-D4)EHP %		90.9	51.6	89.5	73.8	54.9	43	53.7	68.8	81.7
dry matter %		66.2	62.3	75.5	73.7	49.2	73.9	74.9	41	48.2
TOC %		9.1	5.3	<0.5	6.9	7	0.8	1	5.2	5.4
mineral particles <µm %		7.6	10	3.3	11	12	4.5	3	16	17

	Rep. limit µg/kgdm	S28 µg/kgdm	S29 µg/kgdm	S30 µg/kgdm	S31 µg/kgdm	S32 µg/kgdm	S33 µg/kgdm	S34 µg/kgdm	S35 µg/kgdm	S36 µg/kgdm
DMP	0.5	<0.5	<0.5	<0.5	<0.5	<0.5	3	3	2	3
DMT	2	<2	<2	6	7	18	7	6	10	10
DEP	4	13	<4	<4	44	52	34	34	46	49
DPP	3	<3	<3	<3	<3	<3	<3	<3	<3	<3
DIBP	15	98	<15	<15	319	388	264	285	335	384
DBP	15	88	25	<15	255	708	217	209	723	547
DEHP	25	304	60	75	1957	7647	463	254	9757	7176
BBzP	4	<4	<4	<4	37	78	32	25	48	35
DCHP	15	<15	<15	<15	<15	<15	<15	<15	<15	<15
DIHP	6	<6	<6	<6	<6	493	102	<6	864	587
DIOP	45	829	<45	402	1031	17265	394	156	18307	15555
DINP	25	263	43	<25	869	11557	237	48	9843	10220
DIDP	15	210	<15	<15	1075	42280	144	<15	27262	28924
D(ring-D4)BP %		92.6	77.8	80.1	74.7	74.6	96.3	85.5	74.8	75
D(ring-D4)EHP %		51.6	62	64.3	74.1	78	94.2	83	73.5	73.8
dry matter %		51.7	77.9	78.3	63.9	61.3	94.6	79.3	66.7	64.1
TOC %		4.6	0.8	0.6	5.1	9.9	0.8	<0.5	4.4	4.4
mineral particles <µm %		14	3.8	3.5	24	17	13	3	27	21