

Determination of phenols in landfill leachate-contaminated groundwaters by solid-phase extraction

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Abstract

A solid-phase extraction method for phenols in landfill leachates was developed and optimized in order to solve the expected and observed problems associated with an anaerobic matrix containing high concentrations of salts and organic matter. Isolute ENV+ cartridges exhibited the best retention of phenols of the four sorbents examined, and was the only cartridge which a 1 L leachate sample could pass through. With the other cartridges, clogging made this impossible. The final method, which included 27 different phenols, gave detection limits of $<0.1 \mu\text{g/L}$ (drinking water concentration limit for pesticides) for most phenols (25), and for 12 phenols $<0.01 \mu\text{g/L}$. Recovery rates (determined for four concentrations in the range 1–25 $\mu\text{g/L}$, two replicates of each) were in the range 79–104% (SD 1–12%), except for phenol ($26 \pm 1.3\%$) and 2-methoxyphenol ($62 \pm 4.2\%$). Up to 12 different phenols could be identified in leachates from three Danish landfills, ranging in concentration from 0.01 to 29 $\mu\text{g/L}$, which is at the lower end of the concentration range usually found for phenols in landfill leachates (sub- $\mu\text{g/L}$ to mg/L).

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1. Introduction

Leaching of pollutants from point sources such as landfills can result in contamination of the aquatic environment, including drinking water reservoirs. Several factors make the analysis of organic pollutants, such as phenols, in landfill leachate-contaminated groundwaters difficult compared with, for example, drinking water analysis. The matrix is very complex, consisting of high concentrations of humic and fulvic acids as well as salts, which can interfere

with detection or reduce analyte recovery [1–4]. Furthermore, the compounds of interest are typically present at very different concentrations, which makes it necessary to have a method that works within a broad concentration range. Therefore, preconcentration, sample clean up and selective detection are required [5–7]. Solid-phase extraction (SPE) offers a better extraction efficiency and selectivity than traditional liquid–liquid extraction, together with a reduction in the amount of organic solvent used and the degree of sample manipulation required to obtain the final extract [8].

Several studies have shown that phenols can be extracted by SPE with good recoveries and detection limits at the sub- $\mu\text{g/L}$ level [9,10]. The most

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commonly used sorbents are C₁₈-silica [11,12], graphitized carbon [13] and styrene–divinylbenzene copolymers [11,14–16].

The aim of this study was to apply SPE for the extraction of phenols, including methyl-, methoxy-, chloro- and nitrophenols, from landfill leachates and leachate-contaminated groundwaters. The method should solve the most pressing problems associated with the analysis of this complicated matrix.

2. Experimental

This study consisted of two parts: optimization of the SPE method, and application to real samples. The optimization part included a comparison of four cartridges at two pH values with spiked Milli-Q water, optimization of one cartridge with spiked Milli-Q water and testing/optimization of the procedure with spiked leachate.

2.1. Chemicals and samples

The investigated phenols were of analytical grade and purchased from Merck (Darmstadt, Germany), Fluka (Buchs, Switzerland), Aldrich (Milwaukee, WI, USA), Riedel-de Haën (Seelze, Germany) and Sigma (St. Louis, MO, USA). A stock solution containing 1000 mg/L of each phenol was prepared in acetonitrile. Dilutions of the stock solution in acetonitrile were used for spiking.

A solution of internal standards, D₆-phenol (CIL, Andover), 4-fluorophenol (TCI, Tokyo), 2-bromophenol and 2,4-dibromophenol (Aldrich), was prepared in milli-Q water–methanol (10:1) with 250 mg/L of each.

1-Bromonaphthalene (Lancaster, UK) was dissolved in acetonitrile (197 mg/L) and used as GC standard.

Methyl-*tert*.-butylsilyltrifluoroacetamide (MTBS-TFA) was obtained from Aldrich, HPLC-grade methanol and acetonitrile from Labscan (Dublin, Ireland), analytical-grade ethylacetate and toluene from Merck and concentrated hydrochloric acid from Riedel-de Haën. Analytical-grade KCl, KH₂PO₄ and KOH for pH 2 and pH 7 buffers were obtained from Merck.

Four different 6 mL SPE cartridges were used:

EnviCarb, 250 mg graphitized carbon (Supelco, Bellefonte, PA, USA); Isolute ENV+, 200 mg styrene–divinylbenzene copolymer (IST, Mid Glamorgan, UK); Isolute C₁₈/ENV+, 100 mg C₁₈ silica and 100 mg styrene–divinylbenzene (IST); Oasis HLB, 200 mg *N*-vinylpyrrolidone–divinylbenzene copolymer (Waters, Milford, MA, USA).

Four leachate samples were collected. Pyrex bottles (1 L) were filled with nitrogen in order to remove oxygen and then filled with leachate while flushing with nitrogen. The anaerobic samples were stored at 4 °C until analysis. Forlev landfill is an engineered landfill, while Soerup and Vejen are old landfills with no leachate-collecting systems. Samples from Vejen landfill were taken at the border (1) and 97 m down gradient (2) of the landfill [17,18]. The hydrochemical parameters characterizing the landfill leachates are listed in Table 1.

2.2. Instrumentation

SPE extractions were carried out with a FMI Lab pump (Model QSYX; Fluid Metering, New York, NY, USA), while elution and evaporation were carried out on a Supelco Visiprep and Visidry manifold.

GC–MS analysis was carried out with a HP 6890 gas chromatograph coupled to a HP 5973 mass spectrometer equipped with a Varian 8200 CX autosampler. The instrument was operated in the splitless mode (splitless time 0.16 min) and the injection port temperature was maintained at 280 °C. The injection volume was 1 µL. The column was a HP5MS 19091S-433, 30 m×0.25 mm I.D., 0.25 µm. The

Table 1
Hydrochemical data for three Danish landfills. Samples from Vejen landfill were taken at the border (1) and 97 m down gradient (2) of the landfill

	Forlev	Soerup	Vejen 1	Vejen 2
pH	7.0	7.1	6.3	6.1
Electrical				
conductivity (mS/cm)	54	4.7	5.3	1.2
Alkalinity (meq/L)	56	29	NM	NM
Oxygen (mg/L)	0.5	0.1	0.4	0.1
Chloride (g/L)	18	0.61	0.75	0.10
Ammonium (N-g/L)	0.86	0.21	0.21	0.005
NVOC (g/L)	0.36	0.21	0.25	0.02

temperature program was 50 °C for 0.5 min, 10 °C/min to 100 °C, hold 5 min, 2 °C/min to 175 °C, 7 °C/min to 240 °C, hold 3 min with a carrier gas flow (helium) of 0.9 mL/min. The transfer line, source and quadrupole temperatures were 250, 230 and 150 °C, respectively.

Generally, the mass spectra of the *tert*-butyl-dimethylsilyl derivatives were characterized by a small $[M]^+$ peak and a large $[M-57]^+$ peak [19]. The molecular ion was used for quantification while the $[M-57]^+$ peak and one to three other peaks, mostly isotope peaks, were used for identification.

2.3. Analytical procedures

2.3.1. Complete procedure

Standards (1 L) were buffered to pH 2 using KCl/HCl (or to pH 7 using $\text{KH}_2\text{PO}_4/\text{KOH}$) and spiked with appropriate dilutions of the stock solution. The internal standard solution (100 μL) was added to 1 L of sample or standard. Real samples were acidified to pH 2 with concentrated HCl and aerated for 10 min with bubbling air. The sample was left to equilibrate overnight at 4 °C. The SPE cartridges were conditioned with 2 mL ethylacetate, 2 mL methanol and 5 mL water buffered to pH 2 or 7. The sample was passed through the cartridge (constant pressure applied, initial flow-rate 8 mL/min), followed by drying of the sorbent with nitrogen for 30 min. Elution was performed with 4 mL ethylacetate [12,16,20], which was added manually, and allowed to soak for 5 min before it was passed through the cartridge at about 2 mL/min. Evaporation of the eluate to 200 μL was carried out under a gentle stream of nitrogen, and 50 μL GC standard was added. The derivatization was performed as described by Heberer and Stan [19], i.e. 50 μL MTBSTFA was added to an aliquot of 50 μL extract, which was then heated to 80 °C for 60 min.

2.3.2. Estimation of retention volumes

Each SPE cartridge (Envicarb, ENV+, $\text{C}_{18}/\text{ENV}+$ and Oasis HLB) was conditioned and equilibrated (recirculation for 20 h) with a 1 L buffered (pH 7 and 2) solution of phenol (100 $\mu\text{g}/\text{L}$, 10 mL/min). From measurements of the column hold-up volume (V_0), the mass of analyte adsorbed to the stationary phase (m_s) (elution as described above

with a small vacuum applied for all sorbents except ENV+), and the concentration in the mobile phase (c_m), the retention volume (corresponding to the inflection point of the breakthrough curve) was calculated using [21,22]

$$V_R = V_0 \cdot (1 + k') = V_0 \cdot (1 + m_s/c_m \cdot V_M)$$

2.3.3. Sample volume

Breakthrough curves for different concentrations (50 and 500 $\mu\text{g}/\text{L}$) of phenol on ENV+ at pH 2 and for different flow-rates (1.5, 4.5, 8 and 30 mL/min) were constructed from measurements of the effluent concentration at 50 mL intervals during the extraction. The volume that could be extracted without breakthrough (i.e. the breakthrough volume) was determined from the curves.

The retention/breakthrough experiments were carried out on single samples. For measurements of the water-phase concentration in these experiments, 10 mL water was extracted by 1 mL ether–pentane (85:15).

2.3.4. Detection limits and recoveries of the complete optimized procedure with ENV+

Detection limits and linear ranges were determined by application of the method to standards ranging in concentration from 0.001 to 200 $\mu\text{g}/\text{L}$ (single samples at 12 concentrations, and two blank samples). Recovery curves were established from measurements of spiked leachate from Soerup landfill (spiked with 1, 4, 10 and 25 $\mu\text{g}/\text{L}$ of each analyte, two replicates at each level, and two blank samples) by plotting recovered concentrations against expected concentrations (mean blank values subtracted), as described by Funk et al. [23]. Recovery rates were then found as the slope of a linear fit, i.e. one value is obtained for the entire range.

2.3.5. Evaluation of chromatograms

Relative peak areas instead of absolute peak areas were used in all calculations, i.e. area of analyte peak/area of internal standard peak (D_6 -phenol was used for phenol, 4-fluorophenol for cresols, 2-bromophenol for dimethyl-, methoxy-, chloro- and dichlorophenols, and 2,4-dibromophenol for nitro- and higher chlorinated phenols). For the recovery de-

Table 2
Retention volumes (L) for the extraction of phenol (100 µg/L, 10 mL/min) using different sorbents

	Oasis HLB	Isolute ENV+	Isolute C ₁₈ /ENV+	EnviCarb
pH 7	^a	0.8	0.4	0.2
pH 2	0.8	1.1	0.5	0.4

^a Phenol was completely retained, but not recovered on elution of the cartridge.

terminations, the GC standard was used instead of the internal standard.

3. Results and discussion

3.1. Optimization of the method

The choice of SPE sorbent was based on retention experiments and practical considerations. Phenol was used as the test compound because of its relatively low retention on reversed-phase sorbents [1,20]. For all four sorbents, phenol was better retained when the pH was reduced from 7 to 2, and the largest retention was observed for ENV+ (see Table 2). An important advantage of ENV+ is that water can be passed through the ENV+ cartridge by gravity, while pumping is necessary for the other sorbents. When extracting landfill leachate this property means that a larger sample volume can be extracted before the cartridge becomes clogged. Based on the retention behavior of phenol on ENV+/pH 2 (see Table 3) it is expected that a sample volume of 1 L can be extracted without breakthrough when the concentration and flow-rate are low (<50 µg/L, 1.5 mL/min). Of the four cartridges, only ENV+ was capable of extracting such a large volume of landfill leachate, because of its low resistance to sample throughput. One liter is a relatively large break-

through volume compared with other styrene–divinylbenzene sorbents, for which the breakthrough volumes for phenol on smaller columns (10×2 mm I.D./10×3 mm I.D.) has been shown to be <10 mL (PLRP-S), 30 mL (Isolute ENV) and 35 mL (LiChrolut EN) (4 µg/L, 4 mL/min) [2], and 1 mL (PLRP-S) and 5 mL (ENVI-Chrom P) (25 µg/L, 3 mL/min) [15].

Another advantage was that ENV+ changes color when it becomes wet. This property made optimization of the drying step very simple.

Landfill leachates are often anaerobic because oxygen and other electron acceptors are consumed in the degradation of readily degradable organic matter deposited in the landfill [24]. Precipitation of metal (hydr)oxides can therefore be expected due to exposure of the samples to air. This is very difficult to avoid during sample handling, and it is the reason why the samples were actively aerated in order to ensure a reproducible procedure. The samples were also acidified to pH 2, which would cause the humic acids to precipitate. The precipitates, including particulate matter already present in the sample, were not actively removed, but allowed to settle on top of the cartridge during extraction. This means that any precipitate is also included in the elution step, since both sorbent and precipitates were extracted by the solvent.

The extractions were performed at constant pressure and a gradual decrease in flow-rate was observed for field samples due to clogging of the cartridge. Therefore, it was necessary to start the extraction at a relatively high flow-rate. An initial flow-rate of 8 mL/min was determined to be high enough to extract all the leachates in this study without the cartridge running dry.

The final extracts still contained a relatively large amount of matrix compounds. During GC–MS analysis, contamination of the injection liner was unavoidable, and the tailing of free phenols increased.

Table 3
Breakthrough volumes (L) for phenol on Isolute ENV+ at pH 2

	30 mL/min	8 mL/min	4.5 mL/min	1.5 mL/min
500 µg/L	0.01	0.1	0.2	0.3
50 µg/L				1

No tailing was observed when the leachate extracts were derivatized, and more than 50 extracts could be analysed without deactivation of the liner.

It was not possible to separate the analytes from matrix compounds chromatographically, as the matrix compounds were distributed all over the chromatogram and different extracts contained different matrix compounds. Therefore, EI-MS was used for detection, which, with its higher degree of fragmentation compared with other ionization methods, gives most information about the analyte.

Detection limits and recoveries are shown in Table 4. Linear ranges were from the quantification limit ($10/3 \cdot \text{LOD}$) to $50 \mu\text{g/L}$. The recoveries of phenol (26%) and 2-methoxyphenol (62%) suggest a signifi-

cant breakthrough, which can be explained by the flow-rate for leachates being larger than the optimal flow-rate. However, the high recoveries (79–104%) and low detection limits ($0.002\text{--}0.2 \mu\text{g/L}$) for the other analytes justify the large sample volume (1 L) compared with the observed phenol breakthrough. A low recovery of phenol on styrene–divinylbenzene sorbents has also been observed in other studies [20]. The detection limits are comparable to those obtained by similar methods, and, despite the complicated matrix, the recoveries of methyl-, chloro- and nitrophenols are comparable to the recoveries obtained for drinking water. Thus, Heberer and Stan [19] analyzed 15 phenols (1 L drinking water, 0.25 g sorbent, $1 \mu\text{g/L}$, 8 mL/min) with recoveries of

Table 4
Recoveries and detection limits for the final SPE method. Concentrations of phenols ($\mu\text{g/L}$) in four landfill samples

	Recovery ^a $\pm\text{SD}$ (%)	Detection limit ($\mu\text{g/L}$)	Forlev	Soerup	Vejen 1	Vejen 2
Phenol	26 (± 1.3)	0.6	2.2	ND	ND	ND
<i>m</i> -Cresol	81 (± 3.8)	0.2	17	1.0	NQ	ND
<i>o/p</i> -Cresol	86 (± 5.1)	0.02	29	6.8	0.95	0.15
2/3-Chlorophenol	88 (± 2.0)	0.004	1.6	ND	0.22	0.01
4-Chlorophenol	91 (± 2.8)	0.03	1.3	NQ	ND	ND
2-Methoxyphenol	62 (± 4.2)	0.02	ND	ND	ND	ND
3,5-Dimethylphenol	82 (± 1.5)	0.006	27	3.1	4.9	ND
2,5-Dimethylphenol	93 (± 4.0)	0.006	4.5	1.6	0.03	ND
2,4-Dimethylphenol	86 (± 3.3)	0.02	13	3.7	1.7	ND
3,4-Dimethylphenol	82 (± 2.5)	0.006	10	1.6	0.26	ND
2,6/2,3-Dimethylphenol	82 (± 2.5)	0.05	1.9	1.5	1.1	ND
4-Chloro- <i>o/m</i> -cresol	92 (3.8)	0.01	4.6	1.2	5.3	0.26
3,5-Dichlorophenol	100 (± 1.2)	0.01	0.37	0.26	0.09	ND
2,6-Dichlorophenol	95 (± 2.5)	0.003	ND	ND	ND	ND
2,4-Dichlorophenol	94 (± 1.9)	0.002	ND	ND	ND	ND
2-Nitrophenol	82 (± 2.4)	0.006	ND	ND	ND	ND
3-Nitrophenol	85 (± 2.4)	0.02	ND	ND	ND	ND
4-Nitrophenol	86 (± 2.5)	0.04	ND	ND	ND	ND
2,4,6-Trichlorophenol	103 (± 1.3)	0.01	ND	ND	ND	ND
2,3,5-Trichlorophenol	103 (± 1.3)	0.009	ND	ND	ND	ND
2,4,5-Trichlorophenol	103 (± 1.3)	0.01	ND	ND	ND	ND
2,3,6-Trichlorophenol	104 (± 1.1)	0.01	ND	ND	ND	ND
2,3,4-Trichlorophenol	97 (± 5.9)	0.009	ND	ND	ND	ND
2,3,5,6-Tetrachlorophenol	92 (± 4.9)	0.004	ND	ND	ND	ND
2,3,4,5-Tetrachlorophenol	99 (± 12)	0.004	ND	ND	ND	ND
Pentachlorophenol	79 (± 10)	0.07	ND	ND	ND	ND
Nonylphenol	NM	0.1	ND	ND	ND	ND

NM, not measured; NQ, not quantified ($<10/3 \cdot \text{LOD}$); ND, not detected ($<\text{LOD}$).

^a Recovered concentrations were linear functions of the expected concentrations ($R^2 = 0.98335\text{--}0.99992$). Zero was included in the 95% confidence intervals of the intercepts, which is why recoveries could be taken as constant within the investigated concentration range and equal to the slope of the calibration line [23].

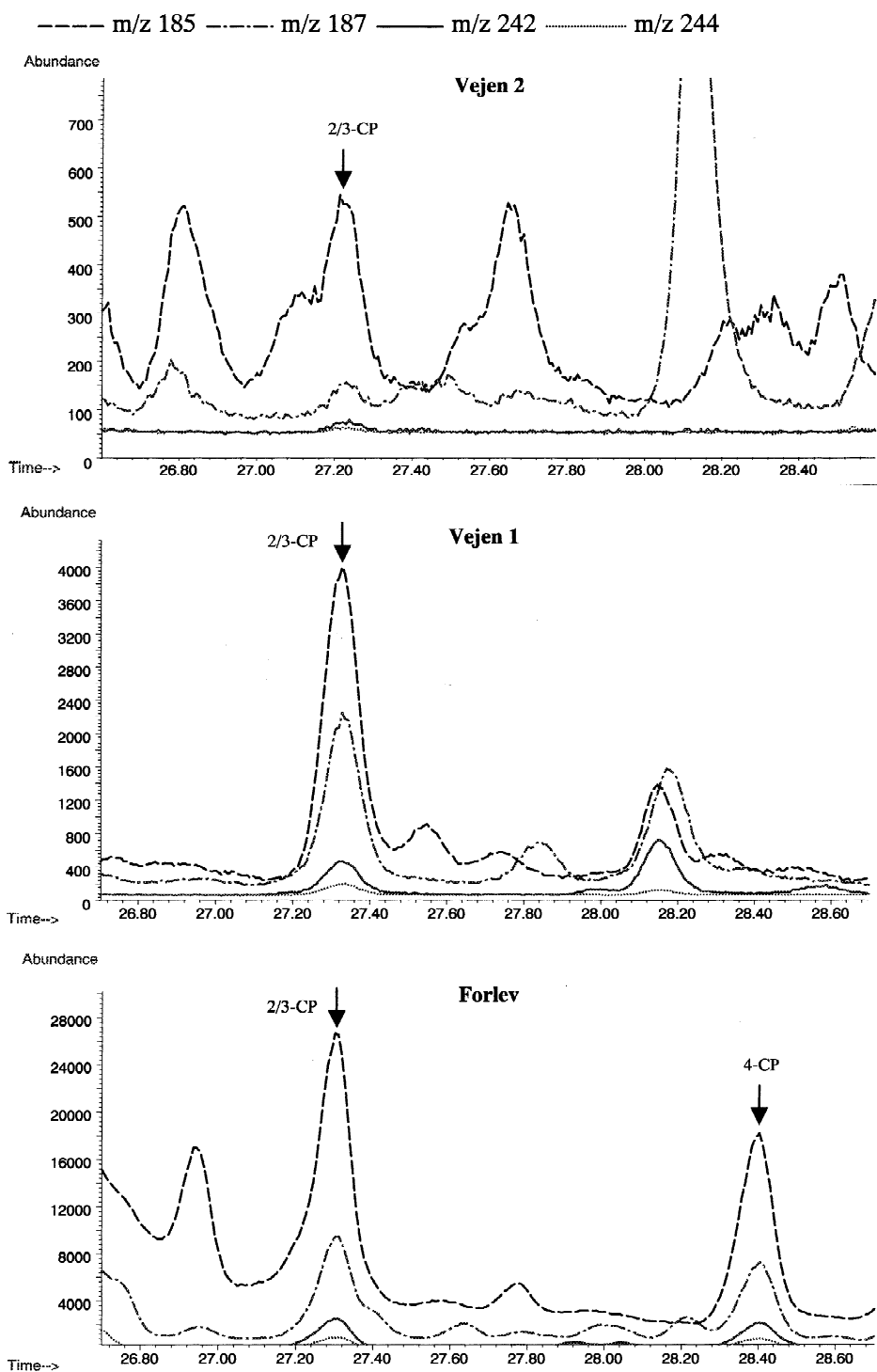


Fig. 1. Extracted ion chromatograms for monochlorophenols in leachate samples. The ion at m/z 242 was used for quantification.

79–108% and detection limits of 0.005–0.025 $\mu\text{g/L}$, while Rodríguez et al. [25] analyzed 16 chlorophenols with recoveries of 84.6–107.3% (1 L drinking water, 0.20 g sorbent, 0.5 $\mu\text{g/L}$, 100 mL/min) and quantification limits ($S/N = 10$) of 0.05–0.13 $\mu\text{g/L}$ (2 L Milli-Q water).

3.2. Application

Finally, the method was applied to four leachate samples. These samples were analyzed with respect to all 27 phenols. Twelve phenols, including methyl- and chlorophenols, were identified (Table 4). Three examples of extracted ion chromatograms for the monochlorophenols are shown in Fig. 1.

Cresols and dimethylphenols were found in all landfills, and several of them in relatively high concentrations (up to 29 $\mu\text{g/L}$). These compounds can be leached from a wide range of different types of waste. Since they are components of, for example, oil and coal tar, they can be seen as being related to the organic content in general, more than to contamination from a specific industry. The chlorophenols, on the other hand, originate from more specific sources. In the landfills investigated in this study, only monochlorinated phenols and 3,5-dichlorophenol were found. The 4-chloro-*o/m*-cresol found in Vejen landfill correlated with large concentrations of the herbicide MCP, which usually contains impurities of 4-chloro-*o*-cresol. The presence of 3,5-dichlorophenol suggests the degradation of higher chlorinated phenols or benzenes, since the compound does not have any commercial use [26,27].

The observed concentrations are at the lower end of the concentration range usually found for phenols in landfill leachates (sub- $\mu\text{g/L}$ to mg/L [28,29]), indicating that phenols are not a major problem at the investigated landfills. At industrial sites such as, for example, coal tar distillation plants [30] or wood preservation sites [31], the concentrations of phenols in contaminated groundwater are typically much higher (mg/L to g/L).

4. Conclusion

Solid-phase extraction of phenols has been shown to be an efficient method for the preconcentration of

landfill leachates. A comparison of four cartridges, EnviCarb, Oasis HLB, Isolute ENV+ and Isolute C₁₈/ENV+, at pH 2 and 7 showed that the best retention was obtained with ENV+/pH 2. Further advantages of ENV+ were low resistance to the flow and different colors for wet and dry sorbents. It was found that the optimal extraction flow-rate (1.5 mL/min) could not be maintained for real samples; an initial flow-rate of 8 mL/min was required. It was possible to co-extract precipitates of metal complexes and humic acids, and sufficient compound identity was obtained by GC–EI–MS analysis.

With a few exceptions the detection limits were $<0.1 \mu\text{g/L}$, and for several of the phenols even $<0.01 \mu\text{g/L}$. Recoveries between 79 and 104% were obtained for all but two phenols.

Twelve methyl- and chlorophenols could be identified in real landfill leachate samples. The observed concentrations were at the lower end of the concentration range usually found for phenols in landfill leachates.

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