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Journal of Chromatography A, 1071 (2005) 41–46
JOURNAL OF CHROMATOGRAPHY A
www.elsevier.com/locate/chroma

Evaluation of a dedicated gas chromatography–mass spectrometry method for the analysis of phenols in water

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Available online 11 September 2004

Abstract

The increasing need to routinely analyse phenolic hydrocarbons in aqueous samples was addressed by the development and implementation of a state-of-the-art, though relatively straightforward, analytical procedure. The proposed method is based on acetic anhydride derivatisation of the native phenols, liquid–liquid extraction of the corresponding phenyl acetate esters and subsequent analysis by GC–MS. The key feature and main strength of the method is located at the injection step which applies 'at-once' large volume injection with a programmable temperature vaporizer (PTV)-type injector. In the proposed method, the sensitivity gain inherent to the higher injection volume was used entirely to proportionally miniaturize, considerably accelerate and effectively simplify the otherwise tedious and time-consuming derivatisation/extraction step. Method performance, as expressed in terms of repeatability, reproducibility, linearity and accuracy, was found to be excellent. R.S.D. values, determined in the framework of an extensive reproducibility study, ranged between 1.47 and 9.02%. Detection limits were in the low ng/L range for all compounds with linear ranges extending up to two orders of magnitude. Method accuracy was determined by analyzing a certified reference material (PH-1JM), spiked water samples and participating in a series of round robin tests and did not reveal any significant bias for the different compounds under investigation.

Keywords: Phenols; Miniaturization; Extraction; Large-volume injection; Gas chromatography–mass spectrometry

1. Introduction

Phenol and its substituted derivatives form a large group of general-purpose chemicals, which are applied extensively in industrial processes, such as production of pesticides, dyes, drugs, plastics and antioxidants, pulp processing, wood, textile and leather preservation, etc. [1]. As a result, phenolic compounds are ubiquitous compounds that can be readily retrieved from water, soil and sediment samples. Because of their persistence and toxicity, a number of them have been classified as priority pollutants and are subject to specific legislation. Within the European Union (EU), for example, the 80/778/EC directive states maximal total and individual phenol permitted concentrations in drinking water of 0.5 and 0.1 μg/L, respectively. However, more stringent regulations are to be expected in the near future. Particularly for this reason it is imperative to hold a fast, yet sensitive, robust and reliable analytical procedure to routinely monitor these compounds at the trace and ultratrace levels they tend to occur.

Over the years, several methods have been published to analyse phenols in aqueous as well as solid samples. Standard procedures, as recommended by the US Environmental Protection Agency (EPA), include Methods 604, 625, 8041 and others [2–4]. Generally, these procedures involve liquid–liquid extraction, evaporative preconcentration of the extract and subsequent analysis by GC–MS or GC with electron-capture detection (ECD) in a dual-column set-up. Although these methods report on the direct analysis of the extracted phenols, derivatisation prior to extraction is generally considered to be a more suited alternative. If omitted, phenolic compounds tend to exhibit severe peak tailing effects, largely compromising chromatographic separation, peak integration and method reliability. Moreover, pronounced activity may result in partial or even complete

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doi:10.1016/j.chroma.2004.07.111

loss of the analytes, while the interference of co-extracted non-specific contaminants may reduce overall method sensitivity and specificity.

By far the most common approach to remove the active hydroxyl hydrogen uses derivatisation with acetic anhydride to transform the phenols to the corresponding acetate derivatives [5]. Prime features of this method involve direct aqueous employment, fast reaction kinetics and high recoveries for most of the target analytes. Although the incorporation of a derivatisation step has uplifted the overall performance of phenol analysis by GC, standard methods still suffer from some serious drawbacks. These drawbacks are mainly situated in the sample preparation step and may be related directly to the strict requirements phenol methods need to fulfil. In order to comply with regulatory demands, i.e. to reach the desired detection limits with sufficient reliability, considerable sample volumes need to be used and adequately processed. As a result, method performance and total analyst bench time per sample are often negatively influenced. Solid-phase extraction (SPE) methods, which have been proposed as valuable alternatives in a number of scientific papers [6], may seem attractive in this respect, though are not able to entirely address the time issue either, since large sample volumes continue to be processed and treated accordingly. At the same time, the apparent risk of cartridge clogging when preparing dirty samples and the batch-to-batch variability of the extraction material remain important matters to bear in mind, as well, when considering such methods as viable for routine analysis.

In this contribution, the performance of a dedicated method to analyse phenolic compounds in water is discussed. The method uses derivatisation with acetic anhydride, truly miniaturized liquid/liquid extraction and GC–MS analysis using at-once large-volume injection (LVI) with the PTV injector. Prime method characteristics are described in detail and properly discussed.

2. Experimental

2.1. Chemicals

Due to uncertainties with respect to toxicity and carcinogenicity, it is recommended that all phenols be considered hazardous and appropriate safety precautions be taken. Carefully consult the respective MSDS charts prior to handling any of these compounds.

Phenol standards were prepared from the EPA 8040A phenol mix (Supelco, Bornem, Belgium) and neat products from Dr. Ehrenstörfer (VWR, Leuven, Belgium), Janssen Chimica (Beerse, Belgium), and Chem Service (Greyhound, Birkenhead, UK). Labelled phenols were purchased from Wellington Labs (Guelph, Canada). Neat [$^2H_6$]phenol; [$^2H_3$]2,4-dimethylphenol and PCB 15–$^{13}C_{12}$ were purchased from Cambridge Isotope Labs. (Greyhound), neat [$^2H_8$]*o*-cresol was from Aldrich (Bornem, Belgium). The PH-1JM standard reference material was obtained from Chem Service. Water, isooctane and *n*-hexane were of HPLC quality and purchased from Rathburn Chemicals (Walkerburn, UK). Potassium carbonate (>99%) was obtained from Fluka (Bornem, Belgium), acetic anhydride (>98.5%) from VWR.

2.2. Preparation of standards

Calibrator standards were prepared in 10 mL water (pH 10) starting from a general stock solution in methanol. In total seven standards were prepared in this way with concentrations from 0.25 to 100 μg/L. After usage, stock solutions were stored at −18 °C in the refrigerator.

2.3. Derivatization procedure

Derivatization is carried out on 10 mL subsamples. In practice, the pH of the original sample is elevated to 14 and the bottle vigorously shaken for several minutes. Avoiding any matrix constituents to set, a subsample is taken and transferred to a 40 mL amber-coloured glass vials with screw thread cap and PTFE-lined septum. In subsequent steps, the pH of the sample is lowered to 11 and phenols are derivatised according to the standard procedure [5].

2.4. Capillary GC–MS

Phenyl acetate esters were analysed using a Finnigan Trace GC–MS system (Interscience, Louvain-la-Neuve, Belgium). The GC system was equipped with an air-cooled PTV injector and a GC PAL sample injector. The GC PAL was provided with a 250 μL gastight syringe with side-hole and a large volume solvent reservoir set-up. Chromatographic separations were achieved on a DB-XLB capillary column (Agilent Technologies, Palo Alto, CA, USA). The column had a length of 30 m, an i.d. of 250 μm and was coated with a stationary phase film of 0.25 μm. A piece of apolar methyl-deactivated precolumn preceded the analytical column (2.5 mL × 320 μm i.d., Varian, Middelburg, The Netherlands).

Oven was heated from 40 °C (1.64 min) to 100 °C at 15 °C/min (5 min) and subsequently to 140 °C at 2 °C/min, to 240 °C at 15 °C/min and finally to 320 °C at 60 °C/min. Large volume injections were carried out in the ‘at-once’ injection mode, using the instrumental parameters summarised in Table 1. In order to accommodate the solvent during the injection step, a dedicated glass liner (1 mm i.d., Interscience) with glass-sintered interior was used.

A specific flow program was applied to the helium carrier gas. Namely, 0.7 mL/min for 0.2 min (solvent removal) and 3.0 mL/min just prior to closure of the splitless valve for a pulsed splitless injection (splitless time 1.00 min). Afterwards, flow was reduced to 1.5 mL/min.

MS transferline temperature was set at 325 °C, source temperature was held at 250 °C. The MS system was operated in time-scheduled selected-ion monitoring (SIM) mode to

Table 1
Schematic overview of the large-volume injection parameters

Parameter	Instrumental setting
Injection volume (μL)	100
Injection speed (μL/s)	15
Initial temperature (°C)	30
Initial time (min)	0.44
Final temperature (°C)	350
Final time (min)	37.27
Heating rate (°C/s)	14.5
Vent time (min)	0.27
Splitless time (min)	1.20
Vent flow (mL/min)	190
Split flow (mL/min)	100

achieve highest sensitivity for each analyte. An overview of selected ions is given in Table 2. Dwell times were optimised in order to acquire a minimum of 12 data point per chromatographic peak. Data were acquired and reprocessed using the Xcalibur software platform (Interscience).

3. Results and discussion

3.1. Large-volume injection

LVI method development was carried out with an alkane mixture (C_{10}–C_{40}) in *n*-hexane [7–9]. Based on the response variations observed during method development, the most suitable instrumental parameters were established (viz. Table 1). Initially, experiments were carried out without adding any keeper to the diluted alkane mix. Under this condition, acceptable recoveries of the volatile analytes could not be reached without being faced with seriously disturbed 'stool-like' peak shapes. Only after diluting the alkane standard in a mixture of *n*-hexane–isooctane (9:1), recoveries could be uplifted to adequate levels whilst preserving symmetrical peak shapes. Here, loss of the most volatile alkanes was limited to approximately 75% for C_{10}. Moreover, labelled standards automatically compensate for these losses, which are equal to those occurring during evaporative preconcentration.

Table 2
Peak identification and MS details

Peak no.	Component	Internal standard	t_R (min)	Quantity	Quality
1	Phenol	[$^2H_6$]Phenol	6.71	94	66
2	*o*-Cresol	[$^2H_8$]*o*-Cresol	8.21	108	107
3	*m*-Cresol	[$^2H_8$]*o*-Cresol	9.03	108	107
4	*p*-Cresol	[$^2H_8$]*o*-Cresol	9.28	108	107
5	2-Chlorophenol	[$^{13}C_6$]4-Chlorophenol	10.24	128	130
6	2,6-Dimethylphenol*	[$^2H_3$]2,4-Dimethylphenol	10.32	122	107
7	*o*-Ethylphenol*	[$^2H_3$]2,4-Dimethylphenol	10.48	107	108
8	3-Chlorophenol*	[$^{13}C_6$]4-Chlorophenol	11.37	128	130
9	2,5-Dimethylphenol*	[$^2H_3$]2,4-Dimethylphenol	11.61	122	107
10	4-Chlorophenol*	[$^{13}C_6$]4-Chlorophenol	11.85	128	130
11	2,4-Dimethylphenol*	[$^2H_3$]2,4-Dimethylphenol	11.94	122	107
12	*m*-Ethylphenol*	[$^2H_3$]2,4-Dimethylphenol	12.18	107	108
13	2-Isopropylphenol*	[$^2H_3$]2,4-Dimethylphenol	12.18	121	136
14	2,3-Dimethylphenol*	[$^2H_3$]2,4-Dimethylphenol	12.98	122	107
15	3,5-Dimethylphenol*				
16	*p*-Ethylphenol*				
17	3,4-Dimethylphenol*	[$^2H_3$]2,4-Dimethylphenol	14.53	122	107
18	2,6-Dichlorophenol	[$^{13}C_6$]2,4-Dichlorophenol	15.82	162	164
19	4-Chloro-3-methylphenol	[$^{13}C_6$]4-Chlorophenol	16.65	142	107
20	2,5-Dichlorophenol*	[$^{13}C_6$]2,4-Dichlorophenol	17.08	162	164
21	2,4-Dichlorophenol	[$^{13}C_6$]2,4-Dichlorophenol	17.19	162	164
22	3,5-Dichlorophenol*	[$^{13}C^6$]2,4-Dichlorophenol	17.87	162	164
23	2,3,5-Trimethylphenol*	[$^2H_3$]2,4-Dimethylphenol	17.94	121	136
24	2,3-Dichlorophenol*	[$^{13}C_6$]2,4-Dichlorophenol	18.59	162	164
25	3,4-Dichlorophenol*	[$^{13}C_6$]2,4-Dichlorophenol	20.32	162	164
26	2,4,6-Trichlorophenol	[$^{13}C_6$]2,4,6-Trichlorophenol	22.26	196	198
27	2,3,6-Trichlorophenol	[$^{13}C_6$]2,4,6-Trichlorophenol	24.68	196	198
28	2,3,5-Trichlorophenol	[$^{13}C_6$]2,4,5-Trichlorophenol	25.18	196	198
29	2,4,5-Trichlorophenol	[$^{13}C_6$]2,4,5-Trichlorophenol	25.56	196	198
30	2,3,4-Trichlorophenol	[$^{13}C_6$]2,4,5-Trichlorophenol	28.03	196	198
31	3,4,5-Trichlorophenol	[$^{13}C_6$]2,4,5-Trichlorophenol	28.90	196	198
32	2,3,5,6-Tetrachlorophenol	[$^{13}C_6$]2,3,4,5-Tetrachlorophenol	31.77	232	230
33	2,3,4,6-Tetrachlorophenol	[$^{13}C_6$]2,3,4,5-Tetrachlorophenol	31.91	232	230
34	2,3,4,5-Tetrachlorophenol	[$^{13}C_6$]2,3,4,5-Tetrachlorophenol	33.25	232	230
35	Pentachlorophenol	[$^{13}C_6$]Pentachlorophenol	34.97	266	268
RS	PCB 15–$^{13}C_{12}$		35.14	234	236

* Purchased as neat components.

Prior to development of the miniaturized extraction procedure, LVI method performance was evaluated. Therefore, a standard was prepared with the target compounds at 5 μg/L and analysed nine times successively. Results of this repeatability study (injection and analysis) are presented in Table 3. Since absolute responses of the native phenyl acetates are corrected by internal standardisation (see Table 2 for IS assignation), conclusions with respect to the repeatability of the LVI procedure (RoI) are only relevant when retrieved from the variability in response of the PCB 15–$^{13}C_{12}$ recovery standard. With an R.S.D. <5%, the LVI method was sufficiently repeatable to be applied in routine analysis. Moreover, repeatability of analysis (RoA), i.e. variation in retention times, did not reveal any irregularities, too, even not for the early eluters, which are more vulnerable to retention time fluctuations caused by variations in amount of solvent injected. A typical chromatogram is shown in Fig. 1. For peak identification is referred to Table 2.

In the following sections details with respect to the performance of the LVI procedure when combined with small-scale sample preparation will be discussed.

3.2. Evaluation of subsampling

The most critical step in miniaturized sample preparation is representative sample size downscaling. Macro-sample homogeneity and reliability of subsampling were evaluated by carrying out two sets of analyses. In the first set, 500 mL of HPLC water, contained in a standard 1 L amber-coloured glass bottle, was spiked with phenols at 5 ng/mL, stabilised with cupper sulphate and stored overnight in the refrigerator at 5 °C. The next day, the bottle was removed from

Table 3
Selected performance data

Component	RoI[a]	RoA[b]	RoP[c]	Linear range[d]	Bias[e]	Reprod.[f]
Phenol	0.42	0.18	0.99	0.01–1.00	−1.81	3.04
o-Cresol	0.59	0.12	0.46	0.01–5.00		4.48
m-Cresol	1.80	0.06	1.04	0.01–1.00		5.20
p-Cresol	1.58	0.11	1.02	0.01–1.00		5.06
2-Chlorophenol	0.66	0.00	1.85	0.01 to >5.00	−10.7	4.63
2,6-Dimethylphenol	0.75	0.10	1.30	0.01–1.00		7.95
o-Ethylphenol	0.51	0.09	0.64	0.01–1.00		4.87
3-Chlorophenol	0.18	0.09	0.35	0.01–5.00		2.93
2,5-Dimethylphenol	1.30	0.09	1.10	0.02–1.00		4.32
4-Chlorophenol	0.10	0.10	0.30	0.02–1.00	2.66	2.80
2,4-Dimethylphenol	0.74	0.09	1.45	0.02–1.00		1.47
m-Ethylphenol	0.34	0.08	0.21	0.01–1.00		3.63
2-Isopropylphenol	0.31	0.08	0.36	0.01–1.00		3.72
2,3-Dimethylphenol }						
3,5-Dimethylphenol	0.35	0.08	0.30	0.01–3.00		4.97
p-Ethylphenol						
3,4-Dimethylphenol	0.55	0.04	0.94	0.01–1.00		3.43
2,6-Dichlorophenol	1.22	0.06	0.49	0.01–1.00		5.84
4-Chloro-3-methylphenol	0.20	0.03	0.35	0.01–1.00		4.85
2,5-Dichlorophenol	1.53	0.02	0.40	0.01 to >5.00		2.56
2,4-Dichlorophenol	0.70	0.05	0.70	0.02 to >5.00	0.24	5.18
3,5-Dichlorophenol	1.19	0.03	0.34	0.02 to >5.00		3.83
2,3,5-Trimethylphenol	0.49	0.02	0.46	0.02 to >5.00		3.09
2,3-Dichlorophenol	0.51	0.02	0.43	0.03 to >5.00		3.78
3,4-Dichlorophenol	0.36	0.00	0.45	0.03 to >5.00		2.43
2,4,6-Trichlorophenol	0.70	0.03	0.84	0.02 to >5.00	0.74	5.26
2,3,6-Trichlorophenol	1.68	0.02	0.95	0.01 to >5.00		7.61
2,3,5-Trichlorophenol	0.86	0.00	0.46	0.01 to >5.00		2.15
2,4,5-Trichlorophenol	0.73	0.00	0.43	0.01 to >5.00		2.39
2,3,4-Trichlorophenol	0.81	0.00	0.66	0.01 to >5.00		4.29
3,4,5-Trichlorophenol	1.19	0.00	0.27	0.01 to >5.00		4.07
2,3,5,6-Tetrachlorophenol	0.85	0.01	2.75	0.01 to >5.00		8.29
2,3,4,6-Tetrachlorophenol	0.62	0.02	0.59	0.01 to >5.00		9.02
2,3,4,5-Tetrachlorophenol	0.51	0.01	0.58	0.02 to >5.00		3.34
Pentachlorophenol	0.25	0.01	1.13	0.03 to >5.00	−7.66	2.57
PCB 15–$^{13}C_{12}$	3.75	0.01	5.40	–	–	–

[a] Repeatability of injection (n = 8), % R.S.D.
[b] Repeatability of analysis (n = 8), %.R.S.D.
[c] Repeatability of the procedure (n = 8), % R.S.D.
[d] ng o.c., $R^2 \geq 0.9995$.
[e] % of true value, n = 3 (Aquacheck 244).
[f] Method reproducibility (n = 15), % R.S.D.

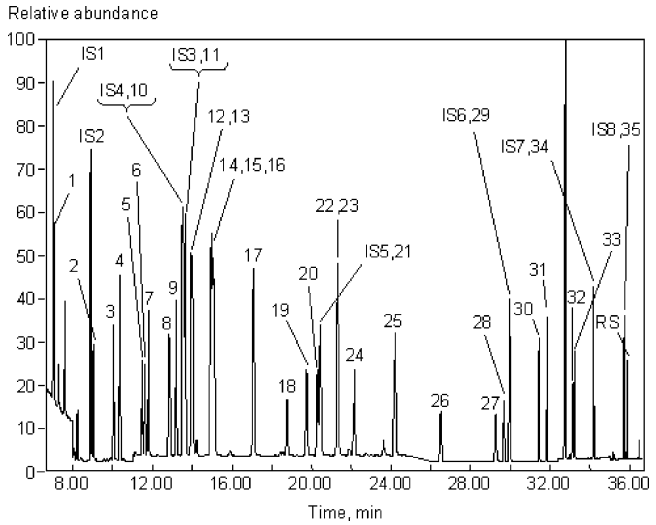

Fig. 1. Chromatogram of a phenol standard at 5 μg/L.

the refrigerator, allowed to reach room temperature and processed according to the procedure described in Section 2. Afterwards, eight subsamples were taken from the bottle, transferred to amber-coloured glass vials, spiked with internal standards, derivatised, extracted and analysed. Mean results were calculated for each phenyl acetate and compared with the results obtained in the second set of analyses. Here, eight samples were prepared with the phenols spiked at 5 ng/mL and processed. When compared with the previous results, no significant deviations were observed, even not for the higher-molecular-mass phenols which are substantially more susceptible for adsorptive losses. Maximal deviation was 8.35% for phenol.

3.3. Method performance

The repeatability of the analytical procedure (RoP) was determined with two matrix types (drinking water and groundwater) spiked at concentration levels of 0.25, 0.1 and 1 μg/L. The study was carried out using 1 L samples of both matrix types, spiked at the required level. Therefore, eight separate subsamples were taken, derivatised and analysed consecutively at each concentration level. The data presented in Table 3 were obtained for groundwater at the low concentration level. It is clear that, the procedure performed remarkably well, even at the low concentration level under investigation. This is a direct consequence of the high response factors of the phenols when derivatised to phenyl acetates combined with their favourable chromatographic behaviour. Within a group of homologue phenols, it is noted that R.S.D.s were highly similar so that internal standard selection is not that critical.

After completing the repeatability study, method linearity was determined. As presented in Table 3, linear ranges (R^2 > 0.9995) were as high as two orders of magnitude and stretched from 0.1 to 10 μg/L for almost all target analytes. This is more than sufficient for routine analytical work, where phenols predominantly occur at ultratrace levels. For this reason, method limits of detection (LODs) are far more important. LODs were calculated as three times the signal-to-noise ratio upon analysis of the lowest standard (0.25 μg/L) and were not higher than 0.01 μg/L for all analytes in both drinking and groundwater. As a consequence, current regulatory prerequisites, which are set at 0.1 μg/L, were met without any problem. Moreover, more stringent regulations, which might be imposed in the future, are easily anticipated by including an evaporative preconcentration step in the procedure.

Also with respect to accuracy, the procedure did not reveal any problems. Accuracy was evaluated based on the results obtained from spiking the EPA 8040A Phenol mix to drinking and groundwater at various levels, analysing the PH-1JM reference material and participating in an Aquacheck round robin test. As expected, the spiking experiments did not reveal any significant biases for the analytes. All recoveries were sufficiently high and were situated between 91% (pentachlorophenol) and 104% (*m*-cresol). Average PH-1JM recoveries (20 analyses in reproducibility) at 10 μg/L were 103% for phenol, 105% for *o*-cresol, 103% for *m*-cresol, 101% for *p*-cresol, 102% for 2-chlorophenol, 98% for 2,4-dichlorophenol, 102% for 2,6-dichlorophenol, 96% for 2,4-dimethylphenol, 101% for 4-chloro-3-methylphenol, 102% for 2,4,6-trichlorophenol, 101% for 2,4,5-trichlorophenol, 103% for 2,3,4,6-trichlorophenol, 103% for 2,3,5,6-tetrachlorophenol and 101% for pentachlorophenol. The results of the Aquacheck round robin test (distribution 244, April 2003) are summarised in Table 3. Also here, no indication of a consistent method bias was observed.

Finally, an extensive reproducibility study was carried out. Same as in the repeatability study, 1 L samples of drinking and groundwater were spiked at concentration levels, corresponding with 0.25, 0.1 and 1 μg/L. Subsamples were taken and analysed every day and this for a total period of 3 weeks (15 injections). The results obtained for groundwater at 0.25 μg/L are depicted in Table 3. Maximal deviation was 9.30% for 2,3,4,6-tetrachlorophenol.

3.4. Analysis of a real sample

A typical chromatogram of a groundwater sample is depicted in Fig. 2. The only distinct signals present in the chromatogram arise from the internal standards and the recovery standard. Such chromatograms are very common when using the procedure and directly result from the selective nature of the sample preparation step as well as the selectivity of MS detection in the SIM mode.

Upon transferring the method to routine, a number of control measures were defined, in order to guarantee result accuracy. First of all, the success of each injection, albeit sample or standard, is verified by means of the response of the PCB 15–$^{13}C_{12}$ recovery standard. Whenever deviations >30% are observed, the analytical sequence is halted and appropriate actions are undertaken to define and solve the problem. In

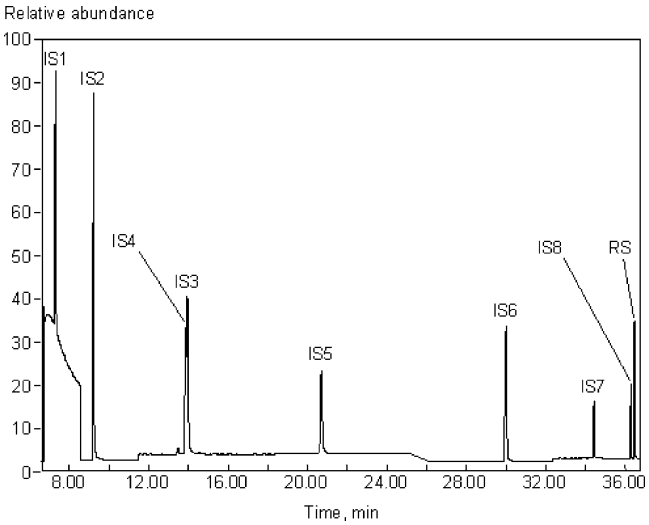

Fig. 2. Typical chromatogram of a real groundwater sample.

this respect, primarily liner and precolumn cleanliness need to be considered as prime causes for the deviant behaviour. In addition, the performance of derivatisation too, is checked carefully. Therefore, the absolute responses of the internal standards (in relation to the recovery standard) are monitored and compared with mean values. Whenever deviations in the analysis of the standards are higher than 30%, the derivatisation procedure is repeated. Finally, blank levels are verified as well. Primarily phenol is susceptible to interferences.

4. Conclusions

A method is presented in which miniaturized sample preparation in combination with at-once large-volume injection is applied in order to analyse phenolic hydrocarbons in water samples (drinking water and groundwater). The proposed method is truly miniaturized and, therefore, elegantly addresses the most common, i.e. durational, problems associated with other procedures. The method is fast, simple, highly specific owing to the use of derivatisation and produces reliable results. Furthermore, the method is highly sensitive, requires no evaporative preconcentration and can be easily automated using commercial autosampler devices. Currently, adapted versions of the proposed method are being evaluated to include the analysis of phenols in wastewater, soils, sediments and sludges.

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