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REPETITIVE STRIPPING AND TRAPPING APPLIED TO THE DETERMINATION OF TRACE HYDROCARBONS IN AQUEOUS SAMPLES*

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SUMMARY

The method of repetitive stripping and trapping of analytes was investigated to determine the reliability of the quantitative results. Different experimental variants of the method, *viz.*, a closed circuit and an open arrangement, were tested by analysing water–air model systems with low μg/l levels of benzene, toluene, *n*-decane, *n*-undecane, *n*-dodecane as analytes in the aqueous phase. Whereas in a closed circuit the stripping/trapping process can be conducted either in a conservation or in an equilibration regime, in an open arrangement the conservation or pseudoequilibration (non-stationary conditions) mode of trapping is possible. All these variants yielded good quantitative results. The results of the determination of *n*-decane, *n*-undecane and *n*-dodecane consistently suffered from systematic negative errors of 20–40%, which was attributed to non-constant (concentration-dependent) matrix effects associated with the adsorption of the analytes at the water–air interface. The ability of the repetitive stripping/trapping method to eliminate such effects was tested by comparing the results for the determination of low μg/l levels of benzene, toluene and *n*-decane in a water–air and in a water–Carbowax 400 (9:1)–air system. Whereas the method of external calibration by means of a reference water–air system gave erroneous results for the system with Carbowax 400, the results obtained by the repetitive stripping/trapping method were correct.

INTRODUCTION

Dynamic headspace analysis in combination with sample-enrichment procedures affords a high sensitivity for the determination of volatile compounds in condensed matrices. The process which takes place during the quasi-equilibrium passage of a gas through a condensed material was described more than 20 years ago[1,2]. This process is utilized analytically in combination with a sample-enrichment unit (charcoal filters, short sorbent-packed columns) to which the analyte is gradually transferred from the condensed phase by the stripping process, either in an open arrange-

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ment[3,4] or in a closed circuit[5]. In order to obtain quantitative data, it is necessary to know the relationships between the initial amounts of the analytes in the material (system) analyzed and the amounts recovered from the trap. The amount of the analytes obtained from the trap is often called the recovery of the entire procedure. This recovery depends, among other factors, on the partition coefficients of the analytes in the given system and, in principle, cannot be assumed to reach 100% in any finite time, as has been documented by several authors[6,7]. Quantification based on a comparison with the analysis of a reference sample containing known amounts of the analytes is sometimes complicated by the so-called matrix effects, *i.e.*, by possible differences in the compositions of the matrices of the actual and reference samples and, consequently, the different partition coefficients of a given analyte[8]. One of the methods by which these matrix effects can be eliminated is repetitive gas-phase sampling. The potential of this method has been demonstrated by the determination of hydrocarbons in water[9] or solvent residues in polymers[10] in a static arrangement, and by the determination of alcoholic compounds in water[11] in a dynamic arrangement.

In this paper, the method is discussed for cases in which the stripping process is combined with sample enrichment in either a closed circuit or in an open arrangement. The potential of this method is demonstrated by the determination of traces of hydrocarbons in water at concentrations of a few μg/l.

PRINCIPLES OF QUANTIFICATION

If a stream of inert gas passes at a volumetric flow-rate, F, through the liquid and gaseous phases of volumes V_L and V_G in a gas–liquid system with initial mass m_{io} of analyte i, the change in the mass of the analyte with time, t, is[2,12]

$$\frac{dm_i}{dt} = -\frac{m_i F t}{V_G + K_i V_L} \tag{1}$$

where m_i is the instantaneous mass of the analyte in the system and K_i is the partition coefficient of the analyte, defined as the ratio of the equilibrium concentrations of analyte in the liquid and gaseous phases, *i.e.*, $K_i = c_{iL}/c_{iG}$. Integration and eqn. 1 for the initial conditions $t = 0$, $m_i = m_{io}$ gives:

$$\frac{m_i}{m_{io}} = \exp\left(-\frac{Ft}{V_G + k_i V_L}\right) \tag{2}$$

Eqns. 1 and 2 are valid provided the liquid matrix is virtually non-volatile (V_L = constant), and the analyte concentrations in the liquid phase and stripping gas bubbles leaving the liquid phase are in equilibrium. The stripping gas containing the equilibrium concentration of the analyte passes through a trap and, provided the analyte is captured quantitatively in the trap, the mass of the analyte entrapped in time t, m_{iS}, is:

$$m_{iS} = m_{io} - m_i \tag{3}$$

Combination of eqns. 3 and 2 gives[12]:

$$\frac{m_{iS}}{m_{io}} = 1 - \exp\left(-\frac{Ft}{V_G + K_i V_L}\right) \tag{4}$$

In dynamic headspace analysis, when the stripping process is combined with a sample-enrichment step, there are three variants of the method of repetitive gas-phase sampling, namely: (i) conservation trapping either in a closed circuit or in an open arrangement; (ii) equilibration trapping in a closed circuit and (iii) pseudoequilibration trapping, *i.e.*, trapping under conditions at which the frontal zone of the analyte has broken through the sorbent bed in the trap, in an open arrangement. The analytical procedure is the same with all three variants in that a liquid sample is stripped at least twice and the initial mass of the analyte in the sample is calculated from the analyte contents determined in the concentrates recovered from the trap in the two analytical steps. However, the processes of stripping/trapping differ in each variant and, consequently, the derivations of the equations relating the initial analyte mass to the experimental parameters are also different.

Variant (i)

If the trapping of the analyte from the stripping gas proceeds in a conservation regime, *i.e.*, if the analyte is totally captured in the sorbent of the trap, the amount of analyte entrapped in time t is given by eqn. 4. Hence, in the first step, after the stripping/trapping time $t = t_1$, the mass of analyte entrapped in the sorbent, m_{iS1}, is

$$\frac{m_{iS1}}{m_{io}} = 1 - \exp\left(-\frac{Ft_1}{V_G + K_i V_L}\right) \tag{5}$$

and the mass of analyte that remains in the system is $m_o - m_{iS1}$. In the second step, after the stripping/trapping time $t = t_2$ with a new trap, the analyte mass entrapped in the sorbent is m_{iS2}:

$$\frac{m_{iS2}}{m_{io} - m_{iS1}} = 1 - \exp\left(-\frac{Ft_2}{v_G + K_i V_L}\right) \tag{6}$$

Provided $t_1 = t_2$ and V_G, V_L and K_i are constant, the right-hand sides of eqns. 5 and 6 are identical (a system factor), and it is possible to write

$$m_{io} = \frac{m_{iS1}}{1 - \frac{m_{iS2}}{m_{iS1}}} = \frac{m_{iS1}}{1 - \frac{A_2}{A_1}} \tag{7}$$

where A_1 and A_2 are the areas of the analyte peaks in the chromatograms of the concentrates recovered from the trap in the first and the second step, respectively. The same relationship was derived earlier from the solute mass balance[13].

Variant (ii)

If the stripping/trapping process takes place in a closed circuit and the analyte is not retained too strongly in the sorbent of the trap, the frontal zone of the analyte breaks through the trap and after some time the entire gas–liquid–sorbent system reaches equilibrium. The mass of analyte contained in the trap at equilibrium conditions is given by[12]

$$\frac{m_{iS}}{m_{io}} = \left[\left(\frac{K_{iS}V_S}{V_{Gt} + K_{iS}V_S}\right)\left(\frac{V_G}{K_{iS}V_S} + \frac{K_iV_L}{K_{iS}V_S} + 1\right)\right]^{-1} \tag{8}$$

where K_{iS}, V_S and V_{Gt} are the partition coefficient of the analyte in the trapping sorbent–gas system, the volume of the sorbent in the trap and the void volume of the trap, respectively. Eqn. 8 differs from eqns. 5 and 6 in having a different system factor (the right-hand side of the equation). Provided all the quantities occurring in the system factor are kept constant during the entire analytical procedure, the same treatment as that applied with variant (i) leads again to relationship 7. Hence, we can conclude that with the closed-loop arrangement the method of repetitive stripping/trapping can be used for quantitative analysis no matter whether the process of trapping proceeds in a conservation or in an equilibration regime.

Variant (iii)

In the context of quantitative analysis, this variant is apparently the least favourable. For exact quantification the exponential decay of the analyte concentration with time would have to be taken into account. However, it can be supposed that in each successive stripping/trapping step accomplished under these non-stationary conditions the analyte mass retained in the trap is proportional to the mass that would be entrapped under the conditions of equilibration stripping and trapping. Thus, for steps 1 and 2, respectively

$$m^*_{iS1} = k_1 m_{iS1} \tag{9}$$

$$m^*_{iS2} = k_2 m_{iS2} \tag{10}$$

where m^*_{iS} is the mass of analyte entrapped under the non-stationary (pseudoequilibration) conditions and k is a proportionality constant. Provided $k_1 = k_2$, the substitution of eqns. 9 and 10 into eqns. 5 and 6 results in relation 7.

From the practical standpoint, there is a substantial difference between the methods of equilibration in a closed circuit and pseudoequilibration in an open arrangement. Namely, in the first case a constant amount of the analyte is retained in the trap while the analyte leaving the trap is returned back to the system analyzed after a state of equilibrium has been reached. Hence, the amounts of the analyte in both the trap and the system do not change with time at equilibrium, and it is then immaterial how long the stripping/trapping process is continued. However, in the second case the amounts of the analyte both in the trap and in the system decrease continuously with time during the stripping/trapping process, and it is mandatory to keep the stripping/trapping times in the consecutive analytical steps strictly equal in

order for $k_1 = k_2$. Clearly, the same qualification applies also to variant (i) if the system factor (right-hand sides of eqns. 5 and 6) is to be kept constant.

EXPERIMENTAL

Chemicals

Hydrocarbons of analytical grade purity (Fluka, Buchs, Switzerland) were used as model analytes. Standard solutions of the hydrocarbons were prepared using analytical grade acetone (Lachema, Brno, Czechoslovakia) as a solvent. The liquid matrices of the systems studied consisted of distilled water boiled for some time before use. To study the matrix effects, Carbowax 400 GC stationary phase (Carlo Erba, Milan, Italy) was used as an additive to the liquid phases of the systems. The Carbowax was purified by purging it with a 100 ml/min stream of pure nitrogen for 30 min at 80°C. Blank determinations of the hydrocarbon contents were negative.

Instrumentation

The stripping/trapping experiments in a closed circuit were carried out with a laboratory-made set-up[14] as illustrated in Fig. 1. The main component is a pump, consisting of a stainless-steel bellows unit (1) and a dual ball valve (3). The bellows unit is periodically depressed by a cam driven by a motor (2). The stripping gas (headspace air) pumped by the bellows passes via the valves and enters a glass vessel (4) containing the liquid to be analyzed. Near the bottom of the vessel there is a sintered-glass frit, above which a septum (5) is attached to a short thick-walled glass capillary to enable the introduction of samples and/or standards. In the outlet of the vessel a quartz-wool plug is inserted in order to prevent droplets of the liquid from being entrained further into the tubing. The volume of the vessel was about 80 ml. The compounds stripped from the liquid are captured in trap 6, consisting of a 6 cm × 3 mm I.D. glass tube packed with Tenax GC (30–60 mesh). The individual units of the set-up are connected with a 1 mm I.D. stainless-steel capillary, so that the gas

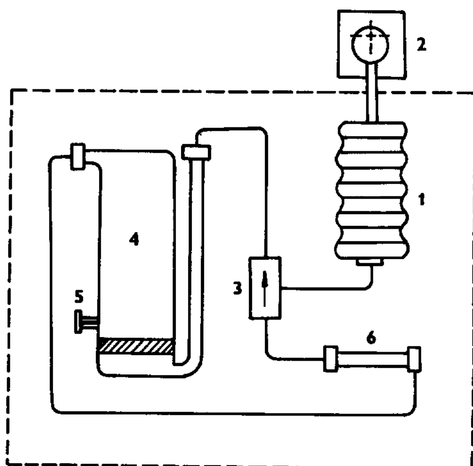

Fig. 1. Flow diagram of the apparatus for work in a closed circuit. 1 = Bellows; 2 = motor; 3 = ball valve; 4 = sample vessel; 5 = septum; 6 = trap.

circulating within the set-up is in contact only with glass and stainless steel. The whole arrangement is fixed to a console whereby it may be immersed in a thermostatted water-bath (shown by a dashed line in Fig. 1).

A schematic representation of the set-up for stripping/trapping in an open arrangement is shown in Fig. 2. The flow-rate of nitrogen (stripping gas) is controlled by needle valve 1 and led by the four-port stopcock 2 either via vessel 3 to trap 4 or to flow meter 5. Septum 6 serves the same function as in the set-up for work in a closed circuit. The thermostatting is again accomplished by use of a water-bath.

The analytes entrapped in the sorbent are thermally desorbed, and the concentrate is entrained by the carrier gas into the analytical column of the gas chromatograph. This is accomplished with the aid of the arrangement shown schematically in Fig. 3. The carrier gas is directed by four-port stopcock 1 into the sample-introduction port of the gas chromatograph, either directly or via trap 2, the latter being heated in oven 3.

The gas chromatographic (GC) analyses were carried out on a GC4A Shimadzu gas chromatograph (Shimadzu Seisakusho, Kyoto, Japan) with a flame ionization detector and a 1.5 m × 3 mm I.D. stainless-steel column containing 15% (w/w) Carbowax 20M on Chromosorb G (100–120 mesh) (Carlo Erba). The temperature of the GC column was 65°C and the sample inlet port and detector were kept at 130°C. The trap was heated to 180°C. The carrier gas (nitrogen) flow-rate was 35 ml/min. The peak areas were measured by a CI-100 integrator (Laboratory Instruments, Prague, Czechoslovakia).

Analytical procedure

The model samples were prepared directly in the vessel of the set-up for stripping/trapping by injecting 1 μl of a standard solution of the model hydrocarbons in acetone into the liquid phase [distilled water alone and/or distilled water and Carbowax 400 (9:1, w/w)]. The concentrations of the analytes in the standard solutions

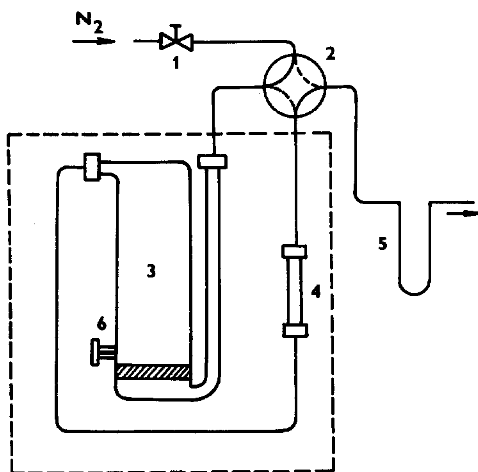

Fig. 2. Flow diagram of the apparatus for work in an open arrangement. 1 = Needle valve; 2 = four-port stopcock; 3 = sample vessel; 4 = trap; 5 = flow meter; 6 = septum.

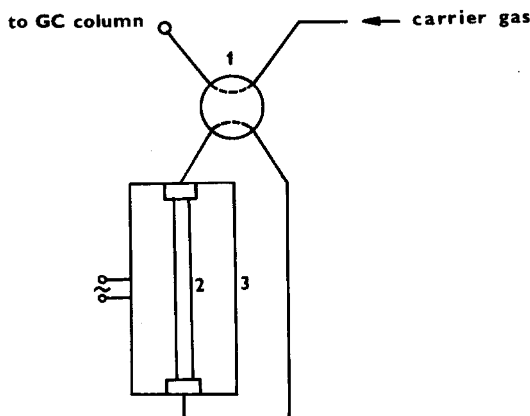

Fig. 3. Flow diagram of the arrangement for the thermal desorption of the concentrate and its introduction into the gas chromatograph. 1 = Four-port stopcock; 2 = trap; 3 = oven.

were chosen so as to obtain samples containing a few μg/l (ppb) of these analytes. The stripping/trapping process was commenced after the vessel had been shaken and heated to 40 ± 0.1°C (410 Ultrathermostat; VEB Prüfgeräte, Medingen, G.D.R.). After each analysis the vessel was washed with distilled water and the entire set-up was purged with a stream of nitrogen. The trap containing the analytes was heated for 3 min at 180°C, whereupon the concentrate was transferred to the gas chromatograph as described above (Fig. 3).

The results obtained by repetitive stripping/trapping were compared with those obtained by injecting samples of the standard solutions directly into the gas chromatograph. By considering the former data as "found" and the latter as "given", the accuracy of the results could be evaluated.

RESULTS AND DISCUSSION

In a previous paper[14], preliminary results obtained by the method of repetitive stripping/trapping in a closed circuit indicated some discrepancies that were attributed to mutual interferences of the analytes. These results have been thoroughly checked in this study, and the revised data are summarized in Table I. The model analytes chosen cover a wide range of boiling points and represent both volatile aromatics and high-boiling paraffins, which both show a tendency to adsorb at the water–air interface[15]. In order to elucidate the mutual interferences of the analytes and the effect of these on the results obtained by the repetitive stripping/trapping method, model samples containing single solutes, several solutes and a relative excess of *n*-dodecane (38 μg/l) were studied separately; the solubility of *n*-dodecane in water was estimated to be about 18 μg/l. For aromatic hydrocarbons the method gives reliable results, although with benzene in the presence of higher *n*-alkanes (especially when in excess) there is a higher probability of a systematic error. In spite of this, however, the relative bias and standard deviation scarcely exceed 10%.

With benzene and toluene the trapping in the Tenax column took place in an equilibration and a conservation regime under the experimental conditions employed

TABLE I

RESULTS OF THE DETERMINATION OF HYDROCARBONS IN WATER BY THE METHOD OF REPETITIVE STRIPPING AND TRAPPING IN A CLOSED CIRCUIT

Conditions: A, single analyte present in the system; B, the analyte in the presence of all the other ones at the concentrations quoted; C, the analyte in the presence of an excess (about 38 μg/l) of *n*-dodecane; D, B + C. The stripping/trapping time was 10 min at 80 ml/min for each step.

Analyte	*Concn. (μg/l)*	*Rel. bias (%)*	*S.D. (%)*	*Conditions*	*No. of measurements*
Benzene	1.8	−10.3	7.0	A	5
		−11.3	3.4	B	6
		−14.1	4.8	C	5
		−12.1	15.7	D	7
Toluene	1.8	−6.2	5.0	B	5
		−5.1	6.3	D	8
n-Decane	1.5	−22.6	3.7	A	7
		−20.8	6.4	B	5
		−35.8	6.1	C	5
		−34.1	5.7	D	8
n-Undecane	3.0	−27.0	6.0	B	5
		−40.0	3.6	D	7
n-Dodecane	7.5	−32.1	7.9	B	5

(with 16 mg of Tenax the retention volumes of benzene and toluene are about 200 and 1000 ml at 40°C, respectively), but the mode of trapping had no effect on the reliability of the results.

As for the systems with aliphatic hydrocarbons, with *n*-decane alone in water the determination suffers from a negative bias of about 20%, and in the presence of an excess of *n*-dodecane the bias of the *n*-decane determination is even larger. As the precision of determination in these cases is the same as that in the analyses of the aromatic hydrocarbons, systematic errors have to be envisaged in the analyses of systems containing higher alkanes. The adsorption of these alkanes at the water–air interface remains a possible cause of the errors. This explanation is corroborated by the significant effect of the presence of *n*-dodecane on the error. Being present in about a two-fold concentration over its solubility limit, *n*-dodecane probably forms a microphase at the water surface, which might enhance the tendency of other hydrocarbons to adsorb. However, this could be established only by exact measurements of the partition coefficients of the analytes in the systems studied. Though the gaseous phase contacts only glass and stainless steel, the surfaces of these materials are most likely covered with a layer of adsorbed water and thus act as water–air interfaces with respect to the analytes. The systematic negative error indicates that the overall matrix effects are not constant during the stripping/trapping process. Namely, as the capacity of the water surface and/or *n*-dodecane microlayer in the systems studied is small, even very low surface concentrations of the analytes adsorbed may exceed those corresponding to the Henry-law region. If the effective partition coefficient (bulk dissolution plus adsorption) of an analyte in the water–air

TABLE II

RESULTS OF THE DETERMINATION OF HYDROCARBONS IN WATER BY THE METHOD OF STRIPPING AND TRAPPING IN AN OPEN ARRANGEMENT

Conditions: E, trap containing 16 mg of Tenax, single solute; F, trap containing 98 mg of Tenax, single solute; G, trap containing 98 mg of Tenax, solute in the presence of an excess (about 38 μg/l) of *n*-dodecane. The stripping/trapping time was 5 min at 80 ml/min (nitrogen) in each step.

Analyte	*Concn. (μg/l)*	*Rel. bias (%)*	*S.D. (%)*	*Conditions*	*No. of measurements*
Benzene	1.8	−52.2	2.9	E	6
		−0.8	4.5	F	6
		−4.2	3.6	G	6
Toluene	1.8	−8.6	5.6	E	6
		−3.6	7.5	F	8
		−3.5	5.6	G	8
n-Decane	1.5	−12.1	4.1	E	5
		−18.5	4.2	F	7
		−26.8	5.9	G	9
n-Undecane	3.0	−18.7	10.1	E	5
		−18.7	8.9	F	10
		−8.8	31.8	G	8
n-Dodecane	7.5	−21.4	11.6	E	5
		−6.2	18.1	F	6

system decreases during the stripping/trapping process, the ratio A_2/A_1 in eqn. 7 becomes larger than that corresponding to a constant partition coefficient, thus resulting in a negative error.

The results obtained by repetitive stripping/trapping in an open arrangement suffer from practically the same errors as those obtained in a closed circuit (see Table II). With the aliphatic hydrocarbons the systematic errors are somewhat smaller as compared to the corresponding results obtained in a closed circuit, however, the large random error of the determination of *n*-undecane in the presence of *n*-dodecane suggests that the small bias in this case is most likely incidental.

TABLE III

RESULTS OF THE DETERMINATION OF HYDROCARBONS IN WATER–CARBOWAX 400 (9:1) BY THE METHODS OF EXTERNAL CALIBRATION (REFERENCE SYSTEM: PURE WATER AS A LIQUID MATRIX) AND REPETITIVE STRIPPING

Conditions: open arrangement, trap containing 98 mg of Tenax, stripping/trapping time 5 min at 80 ml/min (nitrogen) in each step. Six measurements in each case.

Analyte	*Concn. (μg/l)*	*External standard*		*Repetitive stripping*	
		Rel. bias (%)	*S.D. (%)*	*Rel. bias (%)*	*S.D. (%)*
Benzene	1.8	−13.0	5.7	−5.9	6.7
n-Decane	1.5	−60.8	3.4	−15.4	5.1
Toluene	3.6	−11.9	8.2	−4.6	4.6

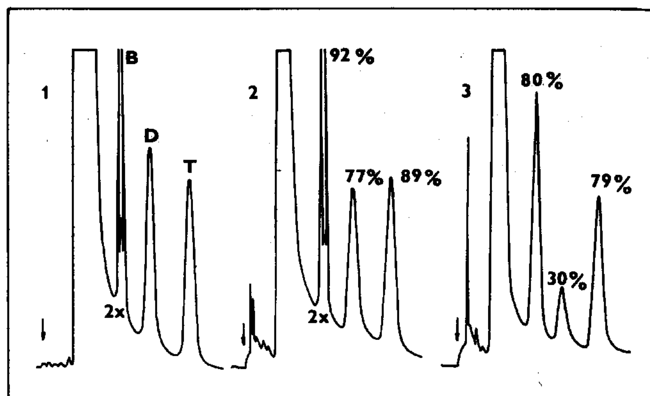

Fig. 4. Efficiency of the stripping of benzene (B), *n*-decane (D) and toluene (T) from liquid matrices of different compositions. (1) Chromatogram of 1 μl of a standard solution of B, D and T (each about 3 μg/l) in acetone; (2) and (3) chromatograms of the concentrates obtained by the 5-min stripping/trapping of B, D and T from systems with pure water and water–Carbowax 400 (9:1), each system having been doped with 1 μl of the standard solution.

In order to demonstrate the ability of the repetitive stripping/trapping method to eliminate the matrix effects, systems in which the liquid phase comprised water–Carbowax 400 (9:1, w/w) were analyzed by this method (open arrangement), and the results were compared with those obtained by external calibration using reference systems with pure water as the liquid matrix. Benzene, toluene and *n*-decane at concentrations of a few μg/l in the liquid phase were used as model analytes. The results of this comparison are summarized in Table III. The effect of the composition of the liquid matrix on the efficiency of stripping is shown in Fig. 4. The first chromatogram was obtained by injecting 1 μl of the standard solution of the model analytes in acetone directly into the gas chromatograph. The second and third chromatograms represent the analyses of the concentrates recovered from the Tenax trap after 5-min stripping/trapping of liquid–air systems with pure water and the water–Carbowax 400 mixture, respectively, each containing 1 μl of the above standard solution. The effect of the presence of Carbowax is manifested with all the analytes, most markedly with *n*-decane. If the peak areas of chromatogram 2 are used as reference data to quantify the analysis of the "actual sample" represented by chromatogram 3, the systematic errors are as quoted in Table III, whereas the repetitive stripping/trapping method yields fairly reliable results, independent of the presence of Carbowax.

CONCLUSIONS

The method of stripping and trapping of analytes both in a closed circuit and in an open arrangement gives good quantitative results. Whereas in a closed circuit the stripping/trapping process can be accomplished either in a conservation or in an equilibration regime, conservation or pseudoequilibration modes of trapping are possible when working in an open arrangement. All the above variants can be used for quantitative analysis, however, when working in an open arrangement better results are obtained by conservation trapping.

Larger negative errors in the determination of higher aliphatic hydrocarbons in water indicate that the matrix effects stemming from the adsorption of the analytes at the water–air interface are not quite constant during the stripping/trapping process. In spite of this, however, results obtained by the method of repetitive stripping and trapping are fairly insensitive to changes in the composition of the liquid matrix of the system studied.

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