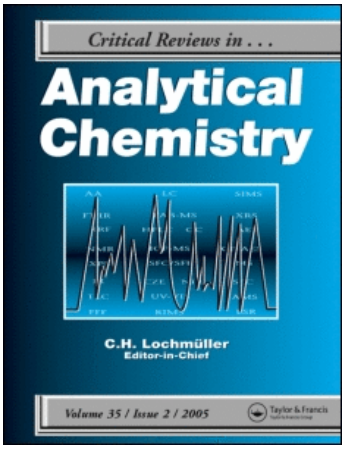

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Accelerated Solvent Extraction (ASE) in the Analysis of Environmental Solid Samples — Some Aspects of Theory and Practice

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ABSTRACT: Theoretical basis, equipment, and some practical considerations regarding the use of a new extraction technique, Accelerated Solvent Extraction (ASE), are presented. ASE is quite a new technique, but its use has so far been limited due to its high cost. This review attempts to summarize some aspects of the theory, results, and conclusions of research related to ASE found in the literature. Applications of this technique to the extraction of various environmental pollutants from solid samples are presented. Finally, the comparisons of ASE to other extraction techniques and its advantages and disadvantages are discussed.

Classic techniques of analyte isolation from solid samples (such as Soxhlet extraction, flask extraction) are tedious and time-consuming, which lowers the sample throughput. Accelerated Solvent Extraction is a fully automated technique, so it could be especially useful for routine analyses of environmental pollutants and food.

Key Words: accelerated solvent extraction (ASE), extraction methods, environmental analysis, sample preparation.

I. INTRODUCTION

Despite recent progress in analytical chemistry, preparation of environmental samples before final analysis still remains a daunting task. Experiments performed by many laboratories are focused on finding the optimal conditions during the extraction step of trace analytes from various matrices. Analytes from solid samples (e.g., soil, sediments, tissues) should be extracted before the final determination. Modern analytical techniques, such as HPLC and GC, require matrix exchange (from a solid to a liquid). During this step, the removal of many undesirable compounds can also be achieved, although usually a clean-up step is also required.

The analysis of contaminants in soil or sediments (pesticides, polychlorinated biphenyls — PCBs, polycyclic aromatic hydrocarbons — PAHs) involves an extraction step, which can be performed in different ways.[1] Most common and simple techniques, such as shake flask extraction,[2] Soxhlet extraction,[3] or sonication,[4] use large volumes of solvent, and they are time-consuming and tedious. There are several new techniques, including microwave assisted extraction (MAE),[5] supercritical fluid extraction (SFE),[6] and accelerated solvent extraction (ASE), which are faster, use less extraction fluids than the "classic" extraction techniques, and can be readily be automated. The use of these techniques requires the development of appropriate operation parameters. The better this optimization step is performed, the less complicated sample clean-up method can be used. This article reviews some aspects of the theory, practice, and the applications of ASE (also known as Pressurized Fluid Extraction — PFE, or Pressurized Liquid Extraction — PLE) — the method what was first described in 1995.[7, 8] Characteristics of the ASE technique have been evaluated on the basis of literature data as well as the authors' experience.

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II. THEORETICAL BASIS

In the ASE system, the extraction process is carried out at temperatures exceeding the boiling point of a solvent what implies that the pressure inside the extraction cell must be kept high in order to maintain the solvent in a liquid state. Both temperature and pressure influence the process efficiency.[9–12]

A. Extraction Process

The desorption of analytes from solid samples can be described by a model presented in Figure 1a. Three steps can be distinguished during the extraction:[13]

- Desorption from a solid particle,
- Diffusion through the solvent located inside a particle pore, and
- Transfer to the bulk of the flowing fluid.

Each step depends on many factors, which can be varied by temperature and pressure modification.

B. Effect of Temperature and Pressure

High temperature used during the process affects properties of a solvent and is one of the most important parameter for this type of extraction.[14] Increased temperature causes:

- An increase in solubility of analytes (e.g., in the temperature range from 50 to 150°C, the solubility of anthracene in an ideal solution is estimated to increase by a factor of 13).[10] Solubility of water in nonpolar solvents increases at higher temperatures and allows the analytes situated in pores filled with water to be reached by the solvent;
- An increase in diffusion rates (e.g., in the temperature range from 25 to 150°C, diffusion rates are estimated to increase 2 to 10 times);[10]
- Weakening and disruption of strong interactions between analytes and matrix components, that is, van der Waals forces (dipole-dipole, dipole-induced dipole, and dispersive), and hydrogen bonds.[12] These interactions depend on the chemical structure (e.g., the presence of functional groups) of matrix and analytes. Thermal energy can lower the activation energy for the desorption process;
- A decrease in viscosity (Figure 2[15]) and surface tension (Figure 3[15]) of the solvent. This allows a better penetration into the pores and between the matrix particles, which improves mass transfer (e.g., the viscosity of 2-propanol decreases 9 times when the temperature increases from 25 to 200°C).[12]

The main reason why high pressure is used during the extraction process is to keep the solvent in a liquid state at elevated temperatures far above the boiling point. High pressures improve the extraction efficiency also by "pushing" the solvent into pores and in this way making the analytes available. Nevertheless, no relationship between pressure and recovery was observed either during the extraction of PAHs from soil[12] or from polymer samples.[16]

C. Effect of the Type of Solvent

A wide range of solvents can be used in the ASE system, except for those with an autoignition temperature of 40 to 200°C (e.g., carbon disulfide, diethyl ether, and 1,4-dioxane). In general, strong bases and acids should be avoided as solvents, because they are corrosive.[17] Transferring the extraction conditions used in conventional methods to ASE usually does not require the change of the solvent. The ASE technique provides an opportunity for the use of a variety of solvents, even those that are not effective in conventional methods. This is done by the temperature and pressure adjustment during the process, which generally increase the solvent solubilizing power. This advantage of ASE over other extraction methods is evident, for example, in the extraction of polymeric samples.[16] However, the extraction of solid environmental samples (soils, sediments), which consist of complex mixtures of different species at different concentration levels, is a more complicated task. At elevated tempera-

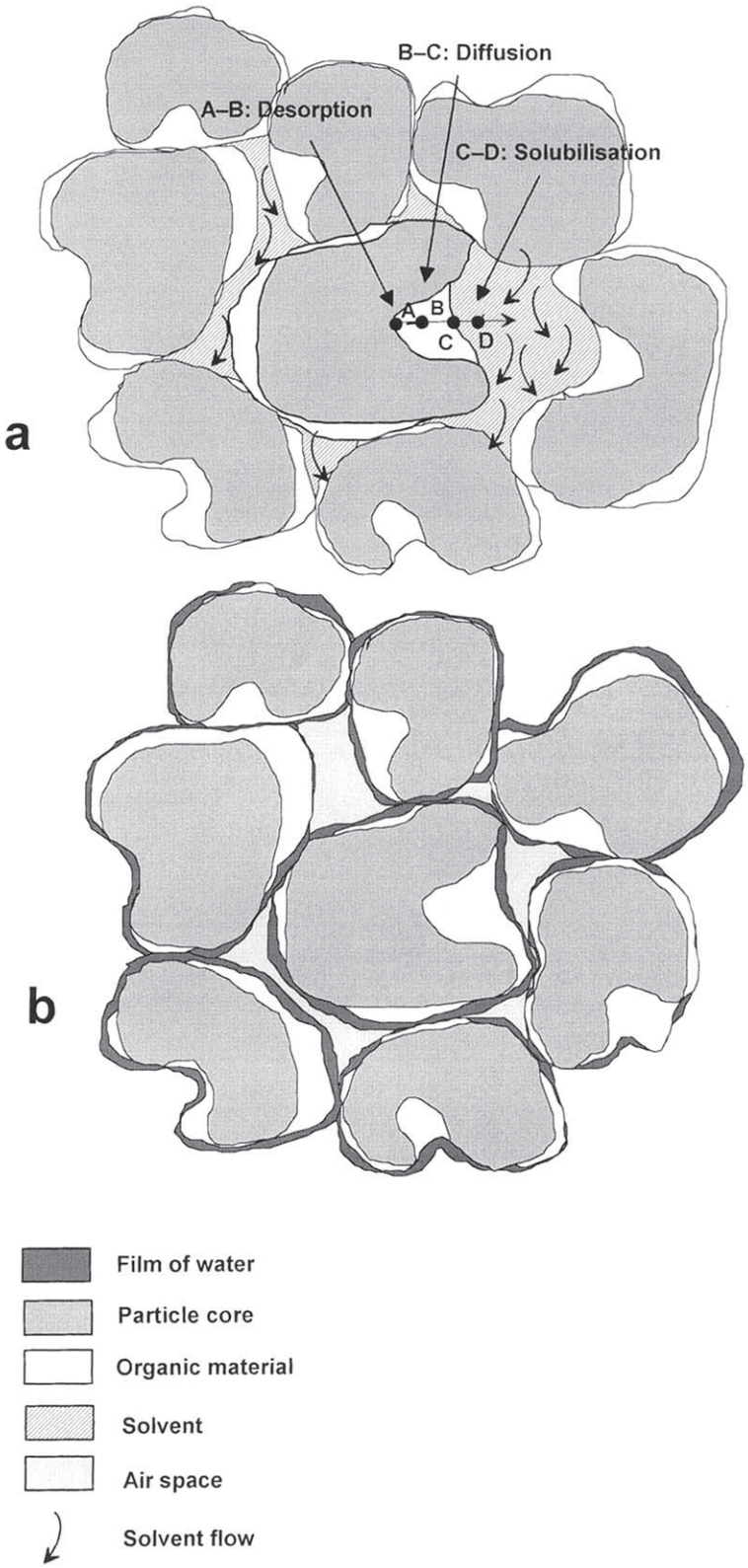

FIGURE 1. The model of lateral profile of soil frgment. (a) The schematic representation of the individual steps in the extraction process, (b). The profile of wet solid sample.[20]

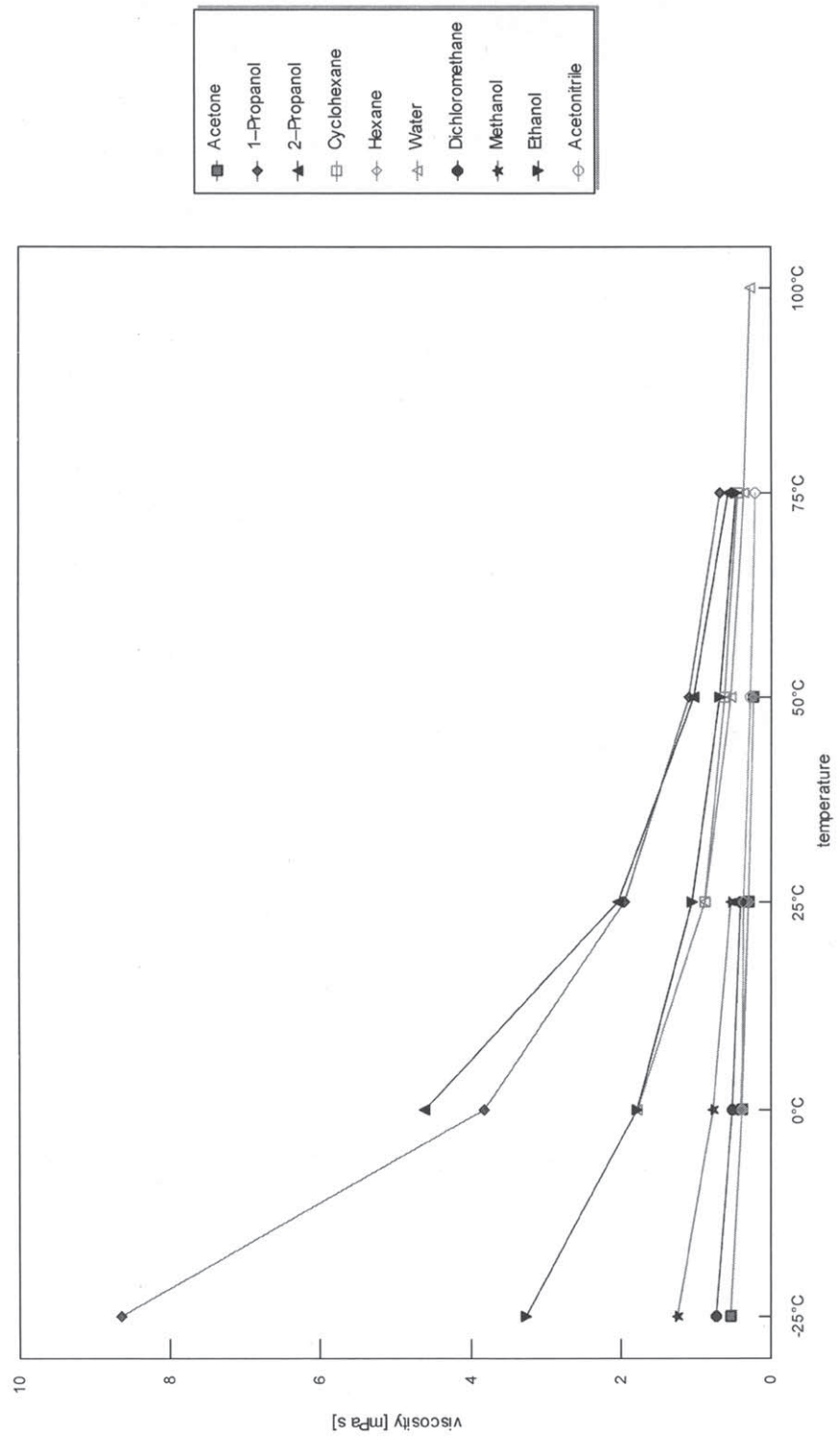

FIGURE 2. Viscosity of liquids [mPa s].[15]

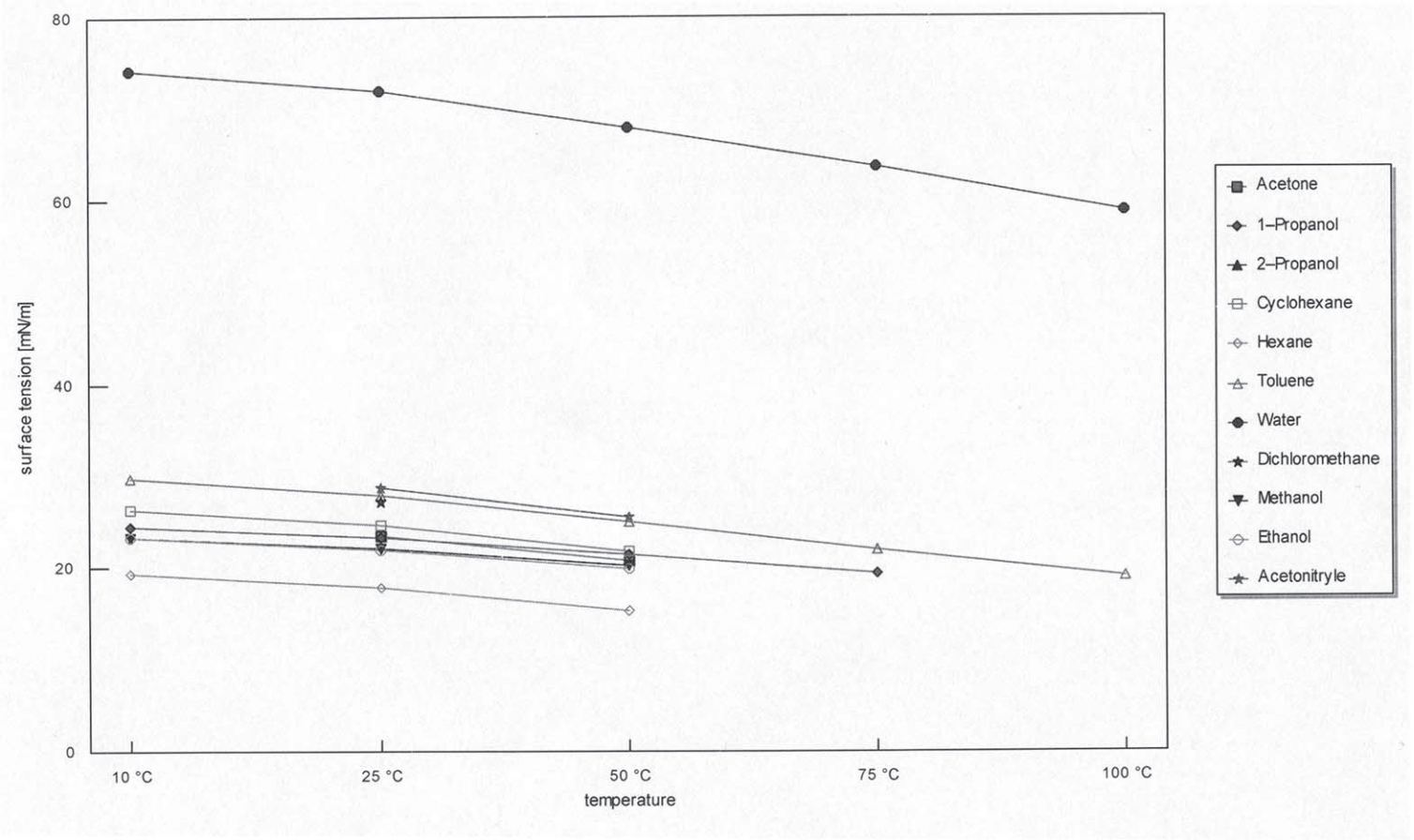

FIGURE 3. Surface tension of common liquids γ [mN/m].[15]

ture and pressure, the extraction process proceeds faster, but the selectivity decreases, because not only the analytes are solubilized.

Although ASE makes possible the use of solvents that are not used in conventional techniques, this may result in solubilization of matrix components, which would otherwise remain insoluble under conditions of conventional extraction. This is why selectivity is often difficult to achieve in ASE when environmental samples are analyzed.

D. Effect of Matrix Composition

The effect of sample matrix depends on sample composition. Solid environmental samples, such as sediments or soils, can differ significantly in their physical-chemical properties, type of compounds present, or granulation (particle diameter). These parameters affect the sorption and retention of analytes. The complexity of analytical procedure increases with the number of organic compounds present in the sample. In order to solubilize the analytes during the extraction, proper conditions (e.g., solvent, temperature, pressure) should be used to overcome the interactions between the organic fraction (e.g., waxes, humic substances[9]) and analytes. This often results in some components of the matrix being co-extracted with the analytes. These co-extracted substances usually should be removed before the final analysis.

One of the parameters, which characterizes the substance and its interaction with the matrix, is the octanol-water partition coefficient. *"An octanol/water partition coefficient, log K_{ow} is a measure of compound hydrophobicity, which in many cases correlates well with various other compound properties, such as aqueous solubility (...).*[18]"

The octanol/water partition coefficient (log K_{ow}) values for a number of organic compounds are listed in Table 1.[18,19]

III. EXTRACTION PROCESS

A typical ASE process consists of several stages. The outline of ASE system is shown in Figure 4.

A sediment or soil sample is usually dried, homogenized, and sieved prior to the extraction. If a sample is not sufficiently dry and a nonpolar solvent is to be used for extraction, the sample should be mixed with a drying agent (e.g., anhydrous sodium sulfate or diatomaceous earth). Figure 1b shows the importance of this step. Particles of soil surrounded by a thin film of water do not allow the analytes bound to the matrix to get in contact with the solvent.[20] Another reason for the addition of a drying agent is the reduction of dead volume of the cell.

In the case of the extraction from wet soil or sediments samples in order to eliminate the negative effect, which was described previously, it is advisable to use a mixture of polar and unpolar solvents, for example, acetone/hexane, acetone/heptane, or acetone/dichloromethane.[21]

A typical ASE process consists of several stages. An outline of ASE system is presented in Figure 5. A weighed sample is placed in the extraction cell. Sometimes copper is also added to the cell to remove sulfur.[22] Several types of extraction cells with different volumes (11, 22, or 33 mL) can be used in the ASE 200 system. The cell can be operated in a preheat or prefill mode.[10] In the former mode, the oven is first heated up to an appropriate temperature and then the cell is loaded into the oven. After the prescribed time, the solvent is introduced into the cell and the extraction process begins. In the latter mode, the cell is filled with the solvent and only after that it is loaded into the oven. The prefill mode allows the removal of interstitial air (Figure 1a) and prevents degradation of compounds that are easily oxidized. For the same reason, degassing the solvent before the extraction is recommended. The prefill mode is recommended for the extraction of thermally labile compounds.[10,24]

The extraction process can be conducted in a static or dynamic mode.[15] The static process begins with heating the cell with the sample to an appropriate temperature during the equilibration time, which lasts approximately 5 min, and is followed by a so-called static extraction process. During this process, the analytes are isolated from the sample under stable static conditions. The static process can be repeated several times if low recoveries are obtained in a single stage. The

TABLE 1
The Octanol/Water Partition Coefficient, logP[18,19]

Category of substances	Name of substances	oktanol/water partition coefficient [log K_{ow}] at 25°C	Category of substances	Name of subtsances	oktanol/water partition coefficient [log K_{ow}] at 25°C
Organo – nitrogen Pesticides	Atrazine	2.34	Polycyclic Aromatic Hydrocarbons	Acenaphten	3,92
	Simazine	1.96		Acenaphthylene	4.27 c
	Propazine	1.38 c[1]		Anthanthrene	6.89 c
	Anilazine	3.02		Benzo(a)antracene	5,91
	Malation	2.89		Benzo(a)fluorene	5.39 c
	Tertbuthylazine	3.04		Benzo(a)pyrene	6.40 c
Organo – chlorine Pesticides	Aldrin	3.01		Benzo(b)fluoranthene	6.63 c
	γ Lindan	3.61		Benzo(c) phenanthrene	5.91 c
	Endrin	4.79 c		Benzo(e)pyrene	6.40 c
	Heptachlor epoxide	3.47 c		Benzo(g,h,i)perylene	6.89
	Methoxychlor	4.3		Benzo(j)fluoranthene	6.63 c
	o, p'– DDD	5.39		Cholanthrene	6.65 c
	o, p' – DDE	6.51		Chrysene	5.91
	o, p' – DDT	5.92		Dibenz(a,h)anthracene	7.14 c
	p, p' – DDD	5.69		Fluoranthene	5.20 c
	p, p' – DDE	6.51		Fluorene	4.18
Organo – phosphorus Pesticide	Chloro – fenwinfos	4.75 c		Indeno(1,2,3-cd) pyrene	7.12 c
	Chloropiryfos	4.77 c		Naphtacen	5,76
	Dichlorofos	0.65 c		Pentacene	7.14 c
	Ethoprofos	3.41 c		Perylene	6,5
	Fenchlorfos	4.88		Picene	7.14 c
	Gution	2.25 c		Pyrene	4,88
	Protiofos	5.53 c		Triphenylene	5,49

[1] c – calculate

dynamic variation of ASE extraction can be compared with high-performance liquid chromatography conducted at elevated temperatures, where the column packing is replaced by the sample. Although this improves mass transfer, this type of extraction is rarely used, mainly because of higher solvent consumption compared with the static process.

At the end of the extraction the sample is usually rinsed with portions of fresh solvent. Then the entire system is purged with nitrogen. These two steps aim at the removal of all of the sample residues from the ASE system in order to improve the analyte recovery and to prepare the system for the next extraction process.

IV. ASE EQUIPMENT

The commercial equipment for accelerated solvent extraction (ASE 200) is fully automated. The extraction methods are easily loaded into

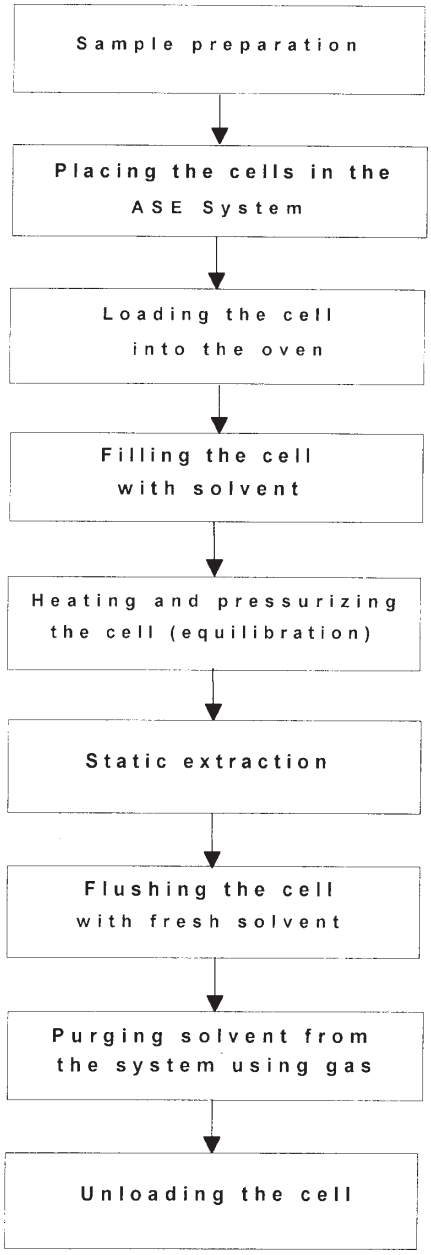

FIGURE 4. The outline of ASE technique.

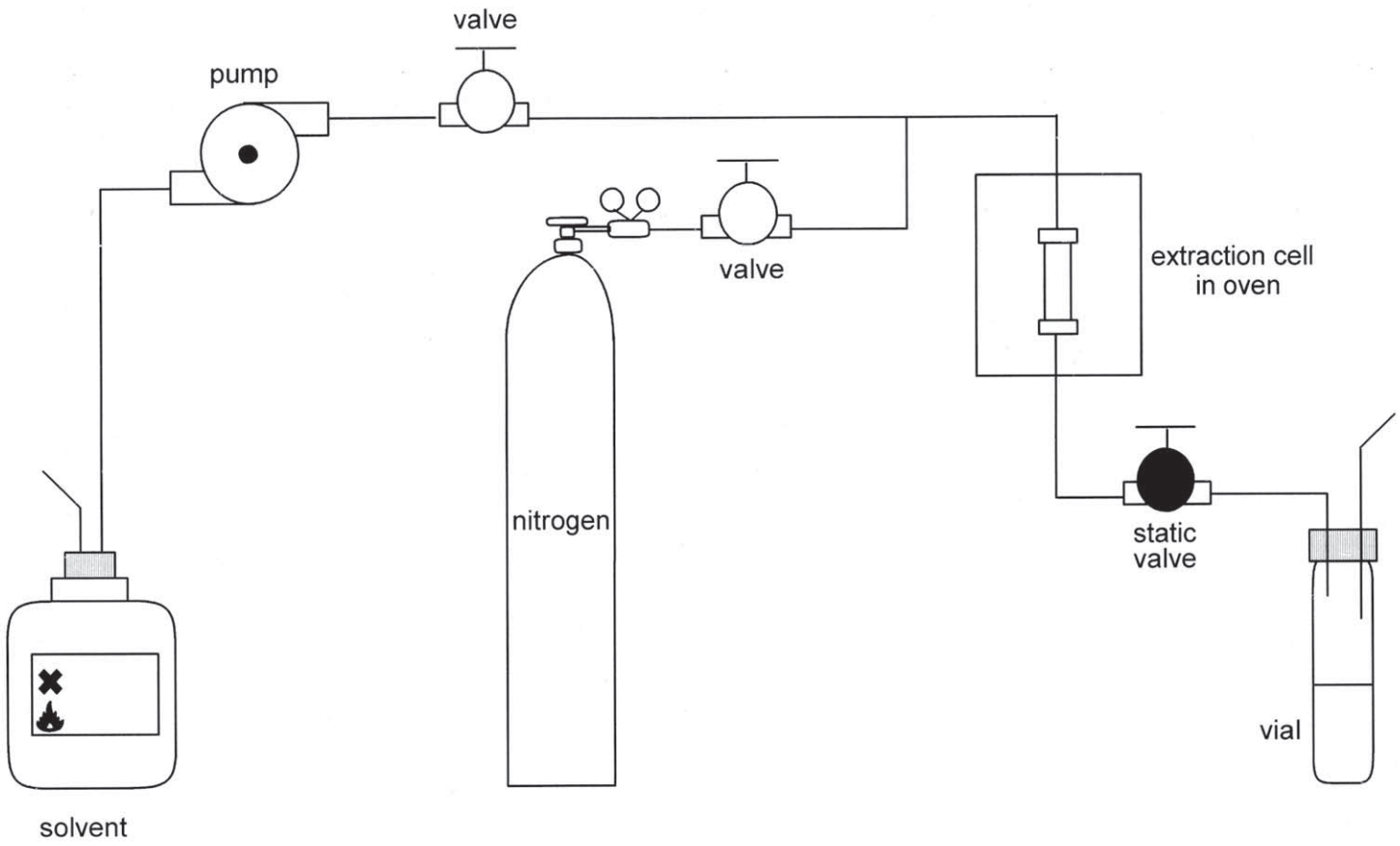

FIGURE 5. Schematic viev of accelerated solvent extraction system.

ASE 200 from the system keypad and can be printed (e.g., operating conditions and schedules) through a direct printer connection or through the computer interface. A schematic view of accelerated solvent extraction system is presented in Figure 5.

For accelerated solvent extraction an ISCO supercricical fluid extractor (Lincoln, NE, USA) or home-made systems with ISCO equipment can be also adapted.[22,25,26]

V. APPLICATIONS OF ASE

Typical applications of ASE systems, including extraction conditions, are listed in Table 2.

VI. ASE AND OTHER EXTRACTION TECHNIQUES

Comparison of accelerated solvent extraction with other extraction techniques for solid samples is presented in Table 3.

ASE allows to reduce the extraction time, but many laboratories will not be able to purchase the equipment because of its high cost. Also, it is difficult to achieve selectivity in the ASE process.

VII. ASE METHOD — SUMMARY

A literature search leads to the conclusion that at present the number of papers and scientific information on applications of ASE is limited. This results from the fact that the ASE technique was introduced into analytical practice only in 1995.

Despite many advantages of the ASE method (in comparison with other extraction techniques — see Table 3), some disadvantages (such as high cost — see Table 2) can be pointed out. Although the extraction time of one sample using the ASE technique is short (compared to Soxhlet extraction or MSPD), the preparation of the extraction cells is time-consuming, tedious, and uses large volumes of solvents (e.g., for rinsing). Due to low selectivity of the process, the obtained extracts should be cleaned up (in case of complicated matrices, such as sediments) and reconcentrated before the final analysis. Accelerated Solvent Extraction as a fully automated technique could be useful especially for routine analyses of environmental pollutants and food. More research needs to be done in order make ASE more popular and useful.

Advantages and disadvantages of accelerated solvent extraction are listed in Table 4.

TABLE 2
The Applications of ASE

Analyte	Matrix	Sample size	Extraction conditions	Final determination	References
PAHs[1]	soil	2 g	DCM–acetone (1:1 v/v) heating time 5 min static extraction time 5 min temperature 100 °C pressure 2000 psi purge N_2 60 sec, 150 psi	–	[27]
PAHs	soil	7g	DCM–acetone (1:1 v/v) heating time 5 min static extraction time 5 min temperature 100 °C pressure 2000 psi	GC–FID	[28]
PAHs	soil	7 g	DCM–acetone (1:1 v/v) heating time 5 min static extraction time 5 min temperature 100 °C pressure 2000 psi	GC–MS	[29]
PAHs	sediments	5–10 g	hexane–acetone (1:1 v/v) temperature 100 °C pressure 2000 psi	HPLC	[30]
PAHs	river sediment	—	toluene extraction time 10 min temperature 100 °C pressure 2000 psi 2 cycles	GC–MS	[31]
PAHs, OCPs[2]	soil, clay loam	10 g	DCM–acetone (1:1 v/v) extraction time 10 min temperature 100 °C pressure 1500 psi	—	[32]
PAHs, Pesticides BNAs[3], PCBs[4],	soil, sediments	5–15 g	DCM–acetone (1:1 v/v) heating time 5 min static extraction time 5 min temperature 100 °C pressure 2000 psi purge N_2 30 sec, 100 psi	HPLC	[11]
PAHs, Phenols, PCBs, TPHs[5]	soil	0.1–2 g	hexane static extraction time 5 min temperature 50–150 °C pressure 1500 psi	GC–MS	[24]
PCBs, OCPs	sediments	0.5–2 g	hexane–acetone (1:1 v/v) heating time 5 min static extraction time 5 min temperature 100 °C flush volume 60 % purge N_2 ,60 sec	GC–ECD	[33]

TABLE 2 (continued)

Analyte	Matrix	Sample size	Extraction conditions	Final determination	References
PCBs	sewage sludge, oyster tissue, river sediment, soil	5–10 g	hexane–acetone (1:1 v/v) heating time 5 min static extraction time 5 min temperature 100 °C pressure 1500 psi flush volume 60 % purge N_2 ,60 sec, 150 psi	GC–ECD	[34]
PCBs	fish tissue	3 g	hexane heating time 5 min static extraction time 5 min temperature 100 °C pressure 1500 psi flush volume 60 % purge N_2 ,60 sec 2 static cycles	GC–ECD	[35]
APEOs[6] (nonionic surfactants)	marine sediments	15–25 g	hexane–acetone (1:1 v/v) temperature 100 °C pressure 1500 psi	LC–ESI–MS	[36]
PAHs, Pesticides, Dioxins, Furans	soild waste	—	aceton–hexane or toluene extraction time 5 min temperature 100 °C pressure 2200 psi	—	[32]
OPPs[7]	soil	10–20 g	DCM–acetone (1:1 v/v) heating time 5 min static extraction time 5 min temperature 100 °C pressure 2000 psi flush volume 60 % purge N_2 ,60 sec	GC–NPD GC–ECD	[37]
OCPs	soil	10–20 g	acetone–hexane (1:1 v/v) heating time 5 min static extraction time 5 min temperature 100 °C pressure 1500 psi flush volume 60 % purge N_2 ,60 sec	GC–MS GC–ECD	[38]
Chlorinated herbicides	soil	10–20 g	DCM–acetone (1:2 v/v), with 4% (v/v) H_3PO_4 /H_2O (1:1) heating time 5 min static extraction time 5 min temperature 100 °C pressure 2000 psi flush volume 60 % purge N_2, 60 sec, 150 psi	GC–NPD GC–ECD	[39]
PCDDs[8], PCDFs[9]	chimney brick, urban dust	4–10 g	toluene heating time 5 min static extraction time 5 min temperature 105 °C pressure 2000 psi	GC–MS	[40]

TABLE 2 (continued)

Analyte	Matrix	Sample size	Extraction conditions	Final determination	References
Alkylphenol–ethoxylates, Alkylbenzene–sulfonates, Alkylphenols (anionic surfactants)	sediments	1 g	methanol static extraction time 10 min temperature 100 °C pressure 2200 psi	HPLC	[41]
BNAs	soil, sediments, sewage sludge	2.5 g	1M sodium acetate and 1M acetic acid in MeOH heating time 5 min temperature 100 °C three static cycles of 5 min	GC–MS	[29]
BNAs	soil	10–28 g	distilled water static extraction time 5–25 min temperature 100 °C pressure 1500 psi	NMR	[39]
Phenols	soil	0.5 g	DCM static extraction time 5–25min temperature 30–70 °C pressure 600–3000 psi	HPLC	[41]
Phenols	sediments	—	acetone extraction time 15 min temperature 120 °C pressure 1800 psi 2 cycles	GC–MS	[31]
BNA	soil	10 g	DCM–acetone (1:1 v/v) heating time 5 min static extraction time 5 min temperature 100 °C pressure 2000 psi flush volume 60 % purge N_2, 60 sec, 150 psi	GC–MS	[38]
LASs[10]	sediments	5 g	methanol static extraction time 5 min temperature 100 °C pressure 2000 psi dynamic extraction time 20 min purge N_2, 60 sec, 150 psi	GC–MS	[26]

TABLE 3
Comparison of ASE with Other Extraction Techniques for Solid Samples

Extraction Method	Shake flask	Matrix solid–phase dispersion	Soxhlet extraction	Soxtec extraction	Sonication	Supercritical fluid extraction	Microwave assisted extraction	Accelerated solvent extraction
Description of method	liquid–solid extraction uses separating funnel	the technique uses bonded–phase solid supports as an abrsive to produce disruption of sample architecture and as a bound solvent to aid complete sample disruption during the sample blending process	sample is extracted using solvent at elevated temperature, below its boiling point	version of the automated Soxhlet extraction	extraction with solvent in ultrasonic bath	sample is extracted using supercritical CO_2 with or without modifier (organic solvent) to extract analytes	microwaves used to heat a sample with proper solvent at elevated temperature and pressure (closed system) or at atmospheric pressure and near room temperature (open system)	sample is extracted using solvent at elevated temperature and pressure, fully automated
Average extraction time	5 to 30 min	about 30 min	4 – 48 hr	1 – 5 hr	periods of 3–15 min, (typically three time periods might be used)	usually 30 min – 2 hr	30 min – 1 hr	12 – 18 min
Setup time	entre time of extraction	entre time of extraction	20 minutes	20 minutes	5 minutes	10 minutes	10 minutes (the same time is needed for 1 and for 12 samples)	10 minutes (the same time is needed for 1 and for 24 samples)
Average solvent consumption in extraction step	100–500 mL	10 – 50 mL	150–500 mL	50–100 mL	100–300 mL	8–50 mL	10–50 mL	15–40 mL

Extraction Method	Shake flask	Matrix solid–phase dispersion	Soxhlet extraction	Soxtec extraction	Ultrasonic bath extraction	Supercritical fluid extraction	Microwave assisted extraction	Accelerated solvent extraction
Cost	high cost of solvents, low cost of equipment	very inexpensive, low cost of equipment	high cost of solvents, low cost of equipment	high cost of solvents, low cost of equipment	relatively inexpensiwe	high cost equipment	moderate cost	high cost of equipment
Easy for use	easy to use	easy to use	easy to use	easy to use	easy to use	considered to be difficult to operate	easy to use	easy to use
Sample size	5 – 30 g	1 – 5 g	> 10 g	> 10 g	up to 5 g	> 1 g	0.2 – 5 g	0.5 – 30 g
Main advantages	easy to use	low cost of equipment	high recoveries of extraction step	high recoveries of extraction step	easy to use	possibility of adjustment of selectivity parameters, frequent use of environmentally friendly extrahents (e.g. CO_2)	short extraction time, low solvent consumption	fully automated system, possibility of extraction of 24 samples in one cycle, short extraction time
Main disadvan-tages	large volume of solvent used	labour–consuming	large volume of solvent used, long time of extraction	large volume of solvent used	no possibility of automation and labor–consuming	high capital costs	usually requires solvents of high dielectric constance, cooling the extraction bomb and filtration of sample after extraction	high capital cost

TABLE 4
Advantages and Disadvantages of ASE 200 System

Advantages	Disadvantages
1. Short extraction times (comparing to classical extraction methods, e.g. Soxhlet) usually about 15 minutes.	1.Very high cost of the equipment (both the apparatus and spare parts).
2. The ASE 200 System is easy to use.	2. Generally, the extraction is not selective. (Temperature and pressure have a little influence on selectivity)
3. Sample preparation for the extraction using ASE System is simple and rapid.	3. Extract clean up is usually necessary before the final analysis. Sensitivity and resolution of chromatographic analysis rapidly deteriorate when extracts are not cleaned prior to analysis.
4. High pressure during extraction permits the extraction of thermally labile analytes, even at high temperatures of this process.	4. Sometimes, one cycle of the extraction procedure with e.g. 10 samples yields different extract volumes, even though the same method is used (extract volume is not reproducible). This probably results from perturbation of operation of static valve (in ASE 200 system).
5. Low solvent use in the extraction step (compared with e.g. Soxhlet) about 15 – 25 ml, .	5. The extraction cells consist of 11 pieces (in ASE 200), which makes the wash procedure very complicated and causes a significant increase in solvent consumption.
6. It is possible to use almost any kind of solvent, except for strong acids or strong bases, or solvents with a flash point of 40 to 200 °C (e.g. carbon disulphide, diethylether, 1,4–dioxane).	
7. Either single solvents or solvent mixtures can be used.	
8. Solvents do not generally need to be degassed. They must be degassed only if the analyte of interest is readily oxidized.	
9. A fully automated system controls the process. This results in high reproducibility of the extraction parameters (e.g. temperature, pressure, static time, flush volume).	
10. In ASE 200 System allows extraction of 24 samples in one cycles.	

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