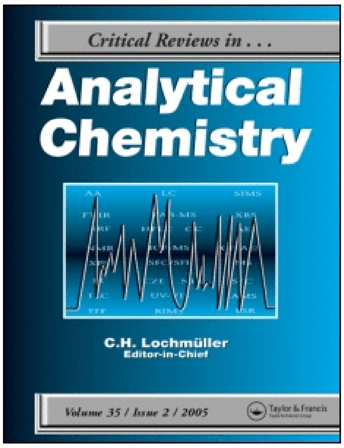

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Trace Analysis — Challenges and Problems

Jacek Namieśnik

Department of Analytical Chemistry, Chemical Faculty, Gdańsk Univ. of Technology, 11/12 G. Narutowicz Str., 80-952 Gdańsk, Poland. tel.: (058) 374-10-10, fax: (058) 347-26-94. e-mail: chemanal@pg.gda.pl

ABSTRACT: Analytical procedures and techniques employed to determine trace components are objects of growing interest to analysts. The determination of those components in samples of different matrix composition is becoming ever more common. Hence, attention should be paid to problems involved in this type of activity.

The article presents specific problems connected with trace analysis. The problems and solutions proposed are documented with data from the literature.

KEY WORDS: trace analysis, sources of error, prevention, wall memory effect, cross-contamination.

I. INTRODUCTION

Even a cursory perusal of any analytical journal must lead one to the conclusion that trace and ultra-trace analysis is a domain of chemical analysis that is gaining in importance recently. This conclusion is corroborated not only by the feelings and opinions of analysts. By the current definition of the term 'trace component' proposed by the IUPAC, the limit from which we can talk about trace analysis is the concentration of 100 ppm (100 μg/g). Naturally, this limit is purely conventional and is not a constant. As recently as 30 years ago 'trace analysis' was understood to denote activities aiming to determine components at a concentration level one order of magnitude higher (i.e., below 1000 ppm, or 0.1%).

Even today the determination of components at a concentration level of 100 ppm, even in samples with complex matrices, does not pose major problems and is done routinely in many laboratories. This is mainly due to the rapid development of instrumentation, or the science of the construction and use of monitoring and measuring devices. Hence, one may expect the definition of the term 'trace component' to change again soon. Table 1 presents a classification of analytical methods and techniques by analyte concentration in the sample examined.

II. AREA OF APPLICATION OF TRACE ANALYSIS

It is possible to distinguish three areas of science and technology that spur the development of analytical methods and techniques employed in the determination of low and very low analyte contents in samples of various kinds. They are

- technologies of the production of high-purity materials; to date, the purity of the cleanest man-made material is denoted by 11 N, which means that the sum total of all impurities it contains does not exceed 10^{-9}%, or 10 ppt;

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Table 1. Classification of analytical methods and techniques by analyte concentration in a sample.

General name of analyte	Analyte concentration	Common term for analytical procedure	Examples
Sub-microtrace component	< 1 ppt (< 10^{-8}%)	TRACE ANALYSIS	Determination of dioxins in samples of various matrices
Ultra-microtrace component	< 1 ppb (< 10^{-6}%)		Determination of trihalomethanes in drinking water and human urine. Determination of volatile organic compounds in indoor air
Microtrace component	< 1 ppm (< 10^{-4}%)		Determination of carbon monoxide in ambient air
Trace component	< 100 ppm (< 0.01%)		Determination of methane in ambient air
Secondary component (admixture)	< 1%	SEMI-MICROANALYSIS	Determination of carbon dioxide in ambient air
Primary component	1-100%	MACROANALYSIS	Determination of oxygen in waste gases. Determination of oxygen in flue gases.

- genetic engineering and biotechnology;
- environmental protection.

The determination of ever lower concentrations of analytes has brought into common use special ways of expressing such concentrations. Table 2 lists the units employed to denote concentrations in trace analysis.

Efforts to determine analytes at ever lower concentration levels in samples with complex matrices that additionally exhibit a high degree of nonhomogeneity are the most important tendency in the development of modern chemical analysis.[1,2] Advances in this field are connected primarily with three types of activity:

- the use in analytical practice of new kinds of detectors and sensors (including biosensors) characterised by:
  —a low threshold of detectability and determinability
  —high selectivity, and often even specificity of response;
- the designing of techniques of sample preparation for the stage of final determination. There is no doubt that this avenue of research opens up especially many new possibilities of determination of trace and ultratrace components. Table 3 lists the main tasks of the stage of sample preparation for analysis;
- the use of hyphenated techniques in analysis. Their development is connected with the growing significance of speciation analysis. Complex analytical systems have been devised which are often composed of three elements connected on-line:[3-6]
  —a system for preliminary sample preparation, usually involving the technique of extraction to the solid phase (Figure 1 shows a diagram of the basic stages of sample preparation using the SPE technique);
  —a separation system. Here a special role is played by chromatographic methods

Table 2. Units employed to denote concentrations of trace and ultra-trace components.

Name of concentration unit	*Part per thousand*	*Part per million*	*Part per billion*	*Part per trillion*	*Part per quadrillion*	*Part per quintillion*	*Part per sextillion*
Volume/volume concentration	vpth (ppth v/v)	vpm (ppm v/v)	vpb (ppb v/v)	vpt (ppt v/v)	vpq (ppq v/v)	$\mathrm{vpq_{ui}}$ ($\mathrm{ppq_{ui}}$ v/v)	vps (pps v/v)
Mass-mass concentration	ppth	ppm	ppb	ppt	ppq	$\mathrm{ppq_{ui}}$	pps
Percentage (%)	10^{-1}	10^{-4}	10^{-7}	10^{-10}	10^{-13}	10^{-16}	10^{-19}
Amount of analyte in 1 g sample	1 milligram (1 mg)	1 microgram (1 µg)	1 nanogram (1 ng)	1 picogram (1 pg)	1 femtogram (1 fg)	1 attogram (1 ag)	1 zeptogram (1 zg)

(mainly GC and HPLC). They are usually employed to separate mixtures into individual components;

—a suitable detection system. For example, Figure 2 presents combinations of separation techniques and spectroscopic detection systems that are most widely used in analytical practice.[6]

III. MAIN SOURCES OF ERROR IN TRACE ANALYSIS

Obviously enough, the determination of components that occur in a sample at very low concentrations presents a completely different challenge to analysts. The various operations performed on the sample during its preparation for the stage of final determinations can be a source of many errors crucially affecting the final result of the analysis.

The chief sources of error are changes in the composition of the sample under study brought about by a variety of factors. In Table 4 three principal kinds of error distorting the result of analysis are presented.

The bias of the final determination result caused by the errors listed (Table 4) may be due to various processes occurring in the sample and due to the influence of the external environment on the sample. By way of example, Figure 3 presents diagrammatically the sources of error that can distort the result of determination of a trace component in a liquid sample. The intensity of the processes and phenomena shown in the figure can vary depending on:

- the temperature of the sample and its changes,
- the presence and intensity of solar radiation, and
- the presence of living organisms in the sample.

The contact of analytes present in both gas and liquid mixtures with the walls of vessels, tubing and appliances crucially affects the concentration levels of trace and ultratrace components. It is the result of adsorption and desorption of analytes on the surface of a solid. These phenomena distorting the sample composition are described collectively as 'the wall memory effect' and 'cross-contamination'. Table 5 shows diagrammatically the magnitude of polar organic analyte-wall surface interaction.

What gives rise to many misunderstandings is the use in analysis of water of suitable purity. Table 6 lists data concerning the efficiency of some water-purifying methods, while Table 7 gives specific electrical conductance values for water with various degrees of purity.

Obtaining ultrapure water, which is the basic reagent in laboratories performing trace analysis, is usually a multistage process. In the installation for the production of water of this level of purity the following processes are employed:

- **preliminary filtering** to remove most suspended matter;
- **activated-carbon filtering** to remove free chlorine (to a sub-1-ppm level), small particles of suspended matter (1 to 5 μm) and most organic matter (to a level below 1 ppm of carbon);
- **preliminary chemical processing**, consisting in the neutralization of water with acid or alkalies, as required, and in the transformation of dissolved CO_2 into carbonates and bicarbonates;
- **ionic exchange**, during which ions present in water, including those introduced in the processing, are exchanged for hydrogen and hydroxyl ions on the cation and anion exchangers;
- **membrane filtration** — microfiltration, ultrafiltration, reverse osmosis;

Table 3. Stages of sample preparation for final determinations.

Task	*Way of implementation*
Ensuring sample stability during transport and storage	Chemical conservation Thermal conservation Lyophilization
Sample homogenization	Grounding Mixing Screening
Removal of interfering components	Dust removal (gaseous samples) Suspension removal (liquid samples) Sample drying Sample deoxidation Removal of reactive components
Chemical conversion of analytes (derivatization) into forms easy to: - isolate - separate mixture components - detect and quantitate	In situ derivatization (while sampling analytes) Derivatization in column Derivatization in eluate
Exchange of sample matrix for one 'friendly' to measuring device used	Extraction of analytes from sample using: - stream of washing gas - suitable solvent (including supercritical fluids) - membrane devices - thermal desorber
Increasing analyte concentration in examined sample to level enabling quantitative analysis	Use of wide variety of analyte enrichment techniques (in many cases this is connected with matrix exchange)
Reducing amount of reagents used (including solvents)	Use of so-called solvent-free techniques of sample preparation Reduction of determination scale Introduction of Total Chemical Analysis System (***TAS***) into analytical practice

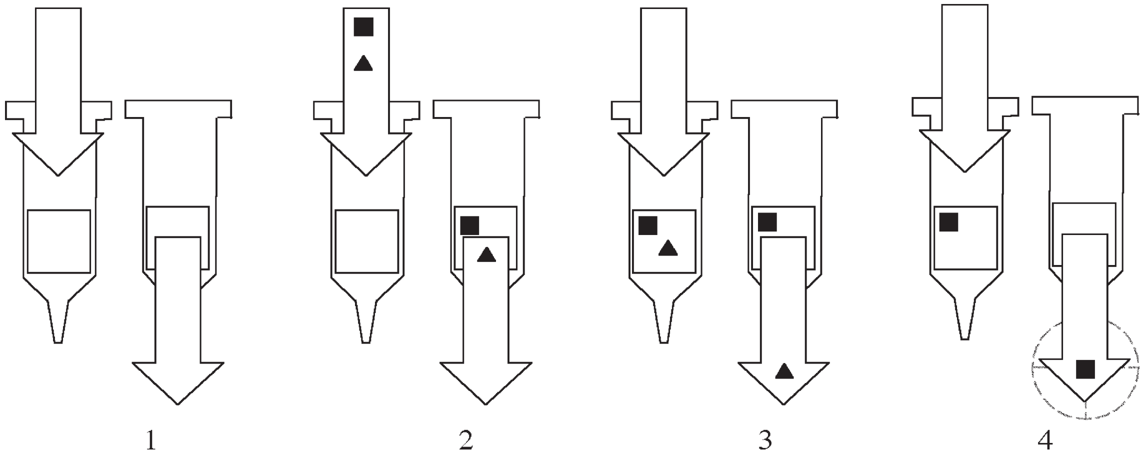


FIGURE 1. Diagram of the main stages of extraction to the solid phase.

1 - conditioning of sorbent bed, 2 - adsorption (passing stream of sample through sorbent bed), 3 - rinsing and cleaning of sorbent bed, 4 - elution of analytes (obtaining of solvent concentrate)

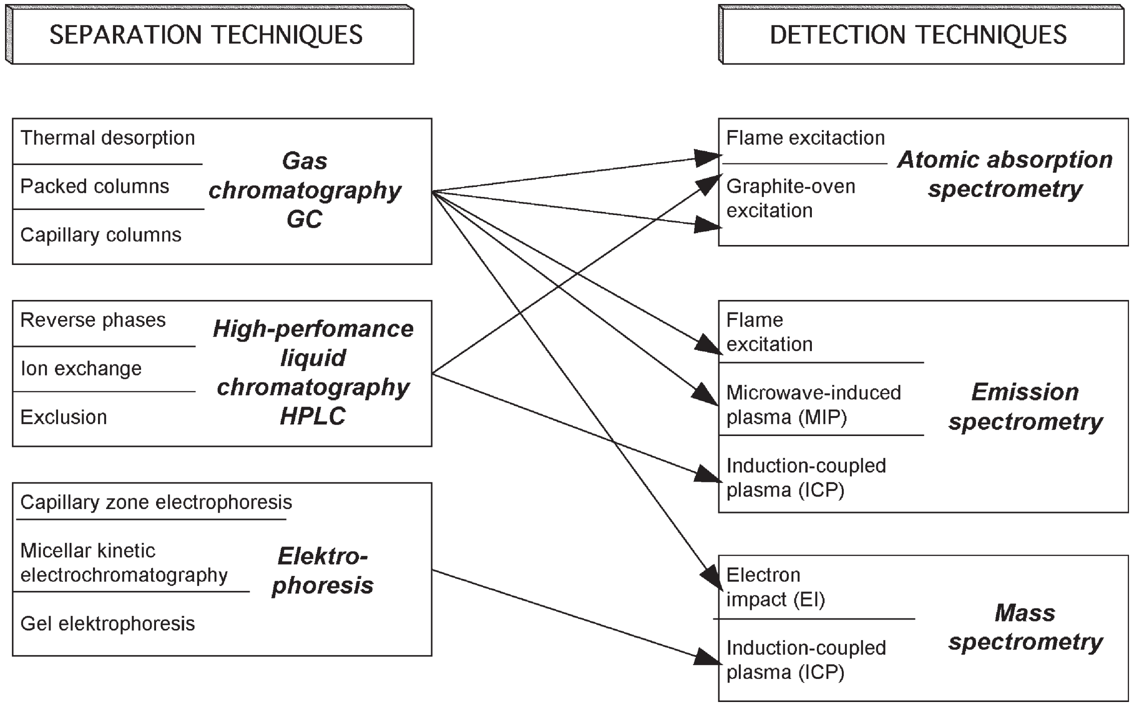


FIGURE 2. Combination of separation techniques and spectroscopic detection techniques most widely used in analysis.

Table 4. Sources of error affecting the ultimate result of analysis.

Source of error	*Name of error*
"Delivery" of additional quantities of analyte to sample	Positive error
Loss of analyte	Negative error
"Appearance" of additional components in sample	Interferences

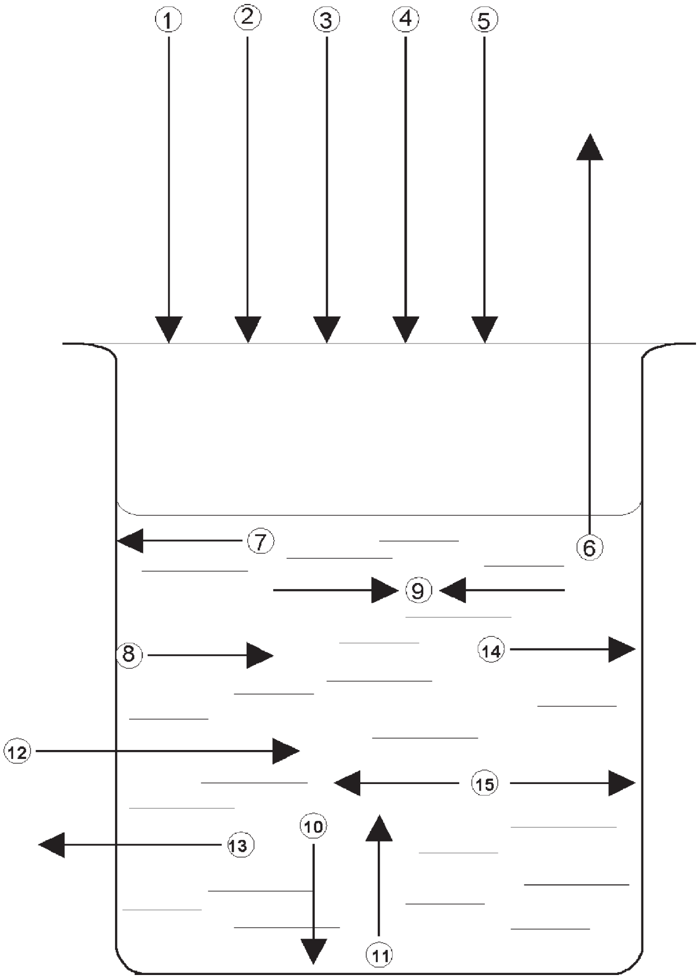


FIGURE 3. Diagrammatic presentation of factors that can affect concentration levels of trace components in a liquid sample.

1 - contact of sample with laboratory air; 2 - residues of components of dishwashing mixtures; 3 - distilled water; 4 - reagents and solvents employed; 5 - contact with analyst; 6 - vaporization of the most volatile components; 7-8 - processes of adsorption-desorption (wall memory effect); 9 - adsorption of analytes on suspension; 10 - precipitation of sediment; 11 - leaching of components from vessel; 12-13 - permeation of solution components through vessel walls; 14 - reaction of analyte with vessel material; 15 - chemical reactions among solution components.

Table 5. Comparison of the polar organic analyte-wall surface interaction for various construction materials and ways of preparing vessel walls.

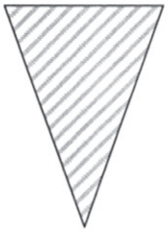
Construction material	*Magnitude of analyte-wall interaction*
Chromium-molybdenum steel Teflon-coated steel Aluminium Glass (after washing with acid) Aluminium (after surface cleansing) Electropolished stainless steel Electropolished stainless steel (after surface cleansing) Glass (after silanization)	

Table 6. Comparison of the efficiency of some water-purifying methods.

Water-purifying techniques	*Pollutant*			
	colloids	*organic substances*	*ions*	*bacteria*
Sorption on activated carbon	0	1	0	0
Reverse osmosis	3	2	1	3
Ionic exchange	1	1	3	0
Filtering through filter with pores 0.2 μm in diameter	3	0	0	3

0	unsatisfactory purification
1	satisfactory purification
2	good purification
3	very good purification

Table 7. Comparison of specific electrical conductance values of raw, distilled and deionized water.

Kind of water	*Specific conductance, μS/cm*
Raw water	300 - 1000 and over
Distilled water:	
- distilled once	4 - 6.5
- distilled twice from quartz	0.5
- distilled twice from glass	1.0
Deionized water	0.2 - 0.1 or under (to 0.055)

- **water degassing** to remove oxygen and carbon dioxide, and to reduce the content of volatile organic compounds to a level below 1 ppb;
- **UV sterilization**, which ensures a reduction of bacteria in water to a level below 1 bacterium/100 cm^3 of water, and the oxygenation of the remaining part of organic matter to a level below 1 ppb;
- **water ozonation** to further reduce the content of organic matter.

For years the amount of organic matter in water (usually expressed by the total content of organic carbon) has been considered the chief measure of quality of high-purity water.

Analytic work in the field of trace analysis should be carried out not only with the use of highest-purity reagents, but also in rooms conforming to the ‘clean room’ standards. Such a room is a space separated from ambient air and only accessible through a sluice. A stream of air cleaned by a system of filters is pumped into the room under a slight positive gauge pressure. The excess is removed through a lattice floor. The HEPA filter that is part of the system stops dust particles of more than 0.3 μm with an efficiency of up to 99.99%. With the air cleaned in this way, controlled humidity and temperature, and suitable working rules of the personnel, the risk of sample contamination is reduced to a minimum in such a laboratory. There has appeared a first mention of the so-called ultraclean chemical laboratory (UCCL) in the literature. In its case the air quality must meet even more stringent criteria. Unfortunately, a significant limitation to ‘clean rooms’ is the very high cost of such an investment. That is why it is much more common for laboratories to utilize much cheaper ‘clean boxes’ with a laminar flow of an inert gas. They can be used successfully in sample processing. The introduction of the robotization and automation of sample processing into analytical practice may be a solution to many problems connected with the cleanness of the laboratory environment.

The quality of the air is defined through the number of dust particles in a unit volume. The American standards set in the *U.S. Federal Standard 209* distinguish six classes of laboratory air on the basis of the number of particles from 0.5 to 5.0 μm in diameter contained in 28.32 dm^3 of air. The classification is presented in Table 8.

In Table 9 some measures are presented which can eliminate, or at least reduce the

Table 8. Cleanness classes of laboratory air. The number of particles in 28.32 dm^3 of air.

Particle diameter / *Class of air cleanness*	*0.1*	*0.2*	*0.3*	*0.5*	*1.0*	*5.0*
1	35	7	3	1	-	-
10	350	75	30	10	-	-
100	-	750	300	100	-	-
1,000	-	-	-	1,000	-	7
10,000	-	-	-	10,000	-	70
100,000	-	-	-	100,000	-	700

Table 9. Ways of eliminating or reducing the influence of factors affecting concentration levels of trace components in a liquid sample (see Fig. 2).

Factor affecting concentration of trace component in liquid sample	*Countermeasures*
1. Contact of sample with laboratory air	- making all activities and operations air-tight - use of clean boxes and clean rooms to carry out operations involved in sample preparation for analysis
2. Residues of components of dishwashing mixtures	- use of proper dishwashing agents and suitable (tested) procedures of cleaning, washing and drying of vessels
3. Water used in sample preparation operations	- proper techniques of water preparation (deionization, distillation, etc.)
4. Reagents and solvents employed	- use of high purity reagents (HPRs) - use of reagents from same manufactured unit - addition of reagents in justified excess - reducing scale of determinations - use of so-called solvent-free techniques of sample preparation
5. Contact with analyst	- use of protective clothes (headgear, gloves, etc.)
6. Vaporization of volatile components	- making sample preparation operations air-tight - storage of solutions and samples in vessels filled up to stopper - use of vessels of appropriate capacity
7-8. Processes of adsorption-desorption of trace components on walls (wall memory effect)	- use of vessels made of suitable materials - special preparation of vessel surface (deactivation) through: - electropolishing - electropassivation - silanization - lowering of storage temperature of samples and solutions - rinsing of vessels with bit of sample or solution
9. Adsorption of components (primarily organic analytes) on surface of teflon-coated mixer	- another way of mixing sample - use of glass-coated mixer
10. Adsorption of analytes on suspension	- preliminary suspension removal through: - decanting - filtration - centrifugation
11. Precipitation of sediment	- acidification of sample
12. Leaching of components from vessel material	- use of vessels made of suitable materials

Table 9 (continued)

13. Permeation of air components to solution	- use of vessels made of materials of low permeability to gases
14. Permeation of solution components outside	- use of vessels made of materials of low permeability to solution components - use of thick-walled plastic vessels
15. Reaction of analytes with vessel material	- special preparation of vessel surfaces as in points 7-8
16. Chemical reactions among solution components	- lowering of solution temperature - preliminary sample preparation through derivatization of reactive components
17. Photodegradation	- storage of samples in the dark
18. Biodegradation	- addition of biocides

intensity of, factors likely to affect the concentration levels of trace components in a liquid sample. In the case of gaseous or solid samples, there will appear not only their specific sources of error, but also those (or at least some of those) characteristic of liquid samples.

In the literature one can find plenty of more detailed information about sources of error in trace analysis. There have appeared comprehensive overviews devoted to these issues[7-10]. They deal primarily with inorganic trace analysis.

The available literature also supplies much information (of various levels of detail) concerning problems and sources of error involved in the determination of trace components in samples with complex matrices. In Table 10 the relevant information is listed that concerns trace analysis, while Table 11 provides data about specific issues related to trace analysis in three basic types of sample (gaseous, liquid, and solid).

SUMMARY

Trace analysis is an important part of chemical analysis and is becoming an object of interest to ever growing ranks of analysts. In solving analytical tasks in this field, more and more complicated monitoring and measuring equipment is used. Unfortunately, this does not always go hand in hand with the level of knowledge of the personnel that operates such devices. There have been a growing number of cases when an analytical device is treated like a typical black box into which it suffices to put a sample to get the required analytical information. This approach is, of course, false and the determination results obtained in this way are often a source of misinformation. The author hopes that the present article will be useful in pointing out several problems relevant in determining components occurring at low and very low concentrations in samples with complex compositions.

Table 10. Information about sources of error in trace and ultra-trace analysis.

Source of error	*Additional information*	*Source*
Reagents used	Purity of substances used in trace analysis	11
	Contamination sources on sampling high-purity reagents	12
	Stability of standard aqueous solutions of organic substances - interlaboratory test	13
	Purity of acids used in electronic industry	14
	Purity of water, acids and various materials used in trace analysis	15
	Use of ultra-pure water in trace analysis and high-purity materials industry	[16-18]
Condition and equipment of laboratory	Use of clean rooms in trace analysis	[19-21]
Determination of sample size	Phenomena affecting representativeness of groundwater samples	22
	Determination of size of representative sample	23
Phenomena and processes involved in taking, storage and preparation of samples for analysis	Water penetration through walls of vessels made of plastics	24
	Sources of error in trace analysis	25
	Wall memory effect in trace analysis	26
	Effect of various parameters on results of study of degeneration of organic compounds in groundwater	27
	Occurrence of organic film on surfaces of solids and liquids	28
	Accuracy in speciation analysis. Use of isotope dilution mass spectrometry (IDMS)	29
	Sources of error involved in taking, storage and analysis of water samples for metallic content	30
	Effect of humic substances on efficiency of reverse osmosis and nanofiltration	31
	Rules of preparation of solid material samples for examination using atomic spectroscopy	32
	Effect of particular steps of analytical procedure on results of determination of trace amounts of elements	33
	Effect of permeation dryer on operation of analytical device (ICP-AES)	34
Measurement consistency	Organisation of international measuring system	35

Table 11. Sources of error involved in the determination of components in samples of different matrix composition.

Material assayed	*Stage of analytical procedure*	*Description of source of error*	*Source*
Gaseous samples	Sampling	Effect of construction of sampler and measuring set on assay results of analytes from PCDD/F group in combustion gases	36
	Sampling of aerosols in upper part of troposphere	Loss of part of aerosols collected by sampler mounted on board plane	37
	Sampling of aeroslos	Changes in cadmium concentrations in aerosol samples depending on particle diameter	38
		Escape of some analytes from sample taken on filter or in impactor	39
		Effect of rate of gas flow through quartz filter on amount of organic matter trapped	[40-41]
		Adsorption of gaseous organic components on quartz filter	42
		PAH losses from sample trapped on filter depending on volume of air sample passed through filter	43
		Effect of impaction, bounce and reaerosolization on collection efficiency of impingers	44
		Adsorption of organic compounds from gas phase on particles of suspended phase	45
	Sampling of gases	Sources of error involved in sampling of gaseous components	46
		Changes over time in operation efficiency of impingers	47
		Dependence of stability of gaseous samples containing odorous substances on analyte concentrations	48
		Effect of filtration of (dust removal from) gas streams on concentrations of volatile organic compounds	49
		Differences in characteristics of indication tubes and hand pumps used during passing gas stream through tube	50
		Changes in composition of gaseous sample (combustion gases from incinerator of hazardous waste) collected in steel canister	51
		Sorption of volatile organic compounds on particles of suspended matter	52
		Contamination of sample with combustion gases from turbine	53
	Sampling of NO_2 from air to impinger	Changes in efficiency of analyte absorption depending on kind of impinger and rate of air stream flow	54
	Sampling of chlorobenzenes and PCBs from combustion gases	Changes in adsorption of analytes on bed of XAD-2 sorbent as result of presence of HCl in gas stream	55
	Sampling of HNO_3 from air	Permeation of analyte through materials used in construction of sampler (copper, stainless steel, glass, chromium-plated stainless steel, teflon)	56
	Sampling of carbonyl compounds	Formation of artifacts during sampling of carbonyl compounds - passing of air stream through sorption tube (DNPH on C_{18} sorbent)	57
	Sampling of unsaturated aldehydes and ketones	Interferences in quantitative determination when using derivatization technique involving DNPH	58

Talbe 11 (continued)

	Taking and storage of gas samples	Effect of polishing of surfaces of vessels made of PTFE on changes in sample composition	59

Gaseous samples	Sampling of combustion gases	Effect of devices used on conditions of isokinetic sampling	48
	Taking and preparing samples for analysis	Effect of interference (ozone) on determination results of volatile organic compounds	60
	Sampling of dust	Effect of construction material of impactor on results of elemental analysis of dust samples	61
	Enrichment of moderately volatile organic compounds	Disturbances in recovery of analytes from XAD-2 bed (after extraction stage) due to presence of HCl in combustion gas stream	62
	Enrichment of trace amounts of light hydrocarbons from air	Effect of way of preparing sorption tube (filled with several layers of various sorbents) on final determinations	63
	Sampling of analytes to sorption tubes	Analyte losses from sorbent bed due to non-hermetic sealing of tube in storage	64
	Sampling of analytes using high volume air sampler (HVS)	Effect of way of sampler preparation and storage (before use) on results of determination of PCB in air	65
	Sampling of analytes on sorbent bed	Way of purifying sorbent bed before extraction stage	[66-69]
		Changes in analyte enrichment efficiency depending on sorbent characteristics	70
	Release of components from sorbent bed (thermal desorption)	Dependence of recovery rate of volatile organohalogen compounds on conditions of thermal desorption	71
	Storage of PAH samples on quartz filters	Effect of filter storage conditions on analyte losses	72
	Sampling of analytes from stream of combustion gases on sorbent bed	Effect of HCl presence in gas stream on adsorption of chlorophenols on XAD-2 sorbent bed	62
	Sampling of volatile analytes from gas phase from waste tank	Stability of analyte samples on sorbent bed (after extraction stage)	73
	Sampling of analytes from gas stream on sorbent bed	Effect of water vapour on analyte adsorption on carbon bed of molecular sieve	74
	Storage of ozone samples	Instability of absorption solutions (alkaline solution of potassium iodide) after absorption of ozone from air stream	75
	Storage of gas samples	Changes in concentrations of components (H_2S, SO_2, COS, CH_3SH, C_2H_5SH, CS_2) of gas samples stored in containers made of Tedlar	76
		Adsorption of organic components on surface of containers made of Tedlar	77
		Changes in composition of gas samples stored in containers made of PVC, Tedlar, polyester, aluminium foil, polyamide, polyethylene	78
	Storage of standard gas mixture	Changes in composition of standard mixture of reactive hydrocarbons (C_2-C_5) in air stored under pressure	79

	Storage of gas samples and calibration mixtures	Interaction of components of gas mixtures with surface of containers made of aluminium and electropolished stainless steel	80

Gaseous samples	Storage of gas samples in plastic containers	Changes in concentrations of volatile organic compounds in gas sample during its storage in containers made of various plastics	81
	Storage of standard gas mixtures	Changes in composition of standard mixtures during their storage	82
	Storage of gas samples	Effect of technique of preparation of teflon surfaces of containers (polishing) on reduction of sample contamination	83
		Changes in composition of gaseous samples in containers of polished stainless steel	84
	Storage of filters with MDI sample	Changes in stability of sample (diphenylmethane diisocyanate - MDI) trapped on fibreglass filter	85
	Storage of samples in canisters (under pressure)	Changes in humidity of stream of gases released from canister	86
	Storage of sorption tubes	Sunshine- and temperature-induced degradation of α-pinene during storage of tubes with Tenax-TA sorbent (after adsorption stage)	87
	Removal of interfering substances	Changes in composition of gaseous sample depending on ozone concentration; effect of ozone removal technique	76
	Removal of reactive components from gaseous samples	Effect of scrubber filling on ozone concentration in sample (changes in oxygenation levels of analytes from terpene group	88
	Removal of interferences from gaseous samples	Changes in dimethyl sulfoxide concentration in gaseous sample depending on degree of removal of NO_2	89
	Sample drying	Change in concentrations of volatile organic analytes in gas stream while drying it in permeation dryer	90
		Effect of dryer (bed of $Mg(ClO_4)_2$) on assays of non-methane hydrocarbons	91
		Change in volume of gas sample due to CO_2 adsorption in bed of silica gel (drying substance)	92
	Extraction of analytes from sorbent bed	Effect of sorbent properties and its level of purity on course of extraction using supercritical fluid	93
	Final determinations	Changes in retention parameters of chromatographic column	94
	Final determinations (GC-ECD)	Changes in response of electron-capture detector	95
	Production of standard gaseous mixtures	Effect of ozone on changes in concentrations of isoprene and methyl vinyl ketone in 'wet' standard gaseous mixture	96
Liquid samples	Sampling of unsaturated water zone	Dependence of determinations of metallic concentrations on presence of suspension	97
	Sampling of rainwater	PCB adsorption from ambient air (collector surface)	98
	Sampling of groundwater	Adsorption of aromatic hydrocarbons on surface of materials used in construction of wells for groundwater sampling (PVC, PTFE, polyethylene, stainless steel, polyvinylidene fluoride epoxy-impregnated fibreglass	99

Talbe 11 (continued)

		Adsorption of pesticides on PVC surfaces	100
	Taking and storage of water samples	Adsorption of organic compounds dissolved in water on PVC and PTFE surfaces	101
		Effect of construction materials of vessels and reagents (acids) used on determination of metal content	102

Liquid samples	Sampling of seawater	Effect of cleanness of sampling system on possibility of determining ultra-trace concentrations (<ppt) of lead	103
	Sampling of water	Effect of amount of matter suspended in water on representativeness of water sample taken to assay metal content in dissolved phase	104
	Taking and storage of seawater samples	Changes in concentration levels of volatile organohalogen compounds	105
	Taking and transport of liquid samples	Leaching of components from polymeric construction materials (FEP, PU, PVC, polyamide)	[106-107]
	Sampling of water from underground	Effect of sampling technique (piston pump, submersible centrifugal pump) on concentrations of volatile organic compounds (POC)	108
	Storage of analyte (pesticide) samples on bed of sorbent	Effect of storage temperature and level of drying of sorbent bed on stability of herbicide sample	109
	Storage of natural water samples	PAH adsorption on suspension particles, PAH adsorption on walls of vessels	110
	Storage of drinking water (in bottles)	Changes in concentrations of trace components	111
	Storage of standard solutions	Stability of solution components (arsenic compounds)	112
	Storage of groundwater samples	Adsorption of volatile organic compounds on surface of teflon membrane (in sampling chamber located in container stopper	113
	Storage of seawater samples	Effect of sample storage at -20şC on concentration levels of phosphates and nitrates	114
	Storage of water samples	Formation of unstable organophosphates and inorganic polyphosphates in water samples as result of phosphate hydrolysis	115
	Storage of water samples	Changes in concentration levels of bisphenol A during storage of water samples	116
		Effect of way of water sample storage on determination of copper content	117
		Effect of deep freezing of water sample (-70^{0}C) on determination of DOC	118
	Conservation of water samples	Changes in concentrations of nitrites and nitrates in stored water sample. Use of pasteurization to stabilize sample composition	119
		Effect of conserving agent on stability of water samples for assays of non-ionic surfactants	120
		Effect of conserving agent on results of metal content assays	121
	Conservation of seawater samples	Changes in nutrient concentrations in seawater samples during thermal conservation	122

	Extraction of organic analytes from water samples	Effect of conserving agent ($HgCl_2$) on efficiency of extraction using solvent and using stream of gas (CLSA)	123
	Filtration of seawater sample	Effect of filtration process on concentration levels of metals	124
		Effect of filtering material and its preparation on metal content in sample	125
	Filtration of river water samples	Effect of filtering material and its preparation on metal content in sample	126

Liquid samples	Filtration of natural water samples	Effect of filtering material and its preparation on metal content in sample	127
	Filtration of water samples	Effect of filtering material on results of assays of dissolved carbon content	128
		Effect of sample filtration (*in situ*) on content of various elements in sample	129
		Changes in concentrations of dissolved trace amounts of elements during membrane filtration	130
		Adsorption of organic components on membrane filter	131
	Filtration of liquid sample	Effect of colloidal silica on performance of reverse osmosis device	132
	Filtration of ocean water samples	Effect of filtration stage on results of determination of nitrogen in suspension	133
	Preparation of water samples	Change in level of carbon dissolved in water produced by filters and material of sample containers	134
		Effect of ionic force on low-temperature oxidizing of organic matter to determine dissolved organic carbon (DOC) content	135
		Effect of conditions of complexing phosphorus with molybdenum blue	136
	Preparation of water samples	Effect of presence of Fe(III) ions on concentration levels of arsenates As(III)	137
		Effect of mineralization and filtration stage on results of assays of phosphorus and dissolved orthophosphates	138
	Transport and storage of water samples	Effect of temperature on concentrations of volatile organic compounds	139
	Transport of water samples	Migration of lead from pipes made of unplasticized PVC	140
		Adsorption of volatile organohalogen compounds on surface of teflon, polyethylene, PVC, and silicone rubber	141
		Contamination of stream of water owing to permeation of pollutants from soil	142
	Extraction of analytes from water samples	PAH adsorption on suspension particles and reduction of efficiency of extraction with petroleum ether	143
	Extraction to solid phase	Effect of storage conditions of sorption tubes (after extraction stage) on final determinations	22
	Derivatization of chlorophenols	Effect of derivatization conditions on final determinations	144
	Extraction of phenol derivatives from cider	Effect of temperature of extracting agent (heated methanol) on analyte stability	145
	Extraction of pesticides from water samples	Stability of pesticide samples after lyophilization of water samples	146

Talbe 11 (continued)

	Extraction of organic pollutant of water on GAC bed	Effect of sorbent characteristics on extraction process	147
	Extraction of organic compounds on sorbent bed	Losses of volatile compounds during extraction	148
	Extraction of volatile organic compounds	Effect of dissolved oxygen on adsorptive capacity of bed of granular activated carbon	149
	Extraction of phenol and fulvic acid	Effect of properties of activated carbon on enrichment process	150
	Extraction of organic compounds with solvent	Formation of artefacts as result of oxidation, halogenation, nitration, autoxidation, and aldol condensation	151

Liquid samples	Derivatization of analyte (3 butyn-2-one)	Side reactions during derivatization with use of DNPH	152
	Reduction in volume of extract sample	Losses of analytes during evaporation of solvent	153
	Extraction of analytes from seawater on sorbent bed	Changes in efficiency of extraction depending on acidity level of sample (for conservation purposes)	154
	Sampling of PAHs on sorbent bed	Changes in efficiency of extraction depending on concentration of dissolved organic matter	155
	Sampling of pesticide samples from water using SPE technique	Disturbances of extraction stage by presence of dissolved organic matter in water	156
	Extraction to solid phase	Stability of bed of DTC resin after stage of extraction of organometallic compounds (Pb, Hg)	157
	Isolation of organic analytes from water - drying of stream of gases after extraction stage	Effect of trap with LiCl bed on results of determination of organic compounds	158
	Separation of suspended matter using tube filter	Effect of stage of membrane separation on content of nitrate and amino acids in water sample	159
	Sampling of organic analytes using SPMD technique	Effect of presence of matter suspended in water and back diffusion on process of analyte sampling by dosimeter	160
	Purification of water samples	Contamination of membranes used for nanofiltration by organic matter of natural origin	135
	Purification of solvent extracts	Changes in level of extract purification depending on character and properties of sorbents	74
	Storage of extraction tubes (after extraction stage)	Effect of storage method of sorption tubes (SDB) on changes in pesticide concentrations	161
	Storage of sorption tubes	Instability of reactive volatile hydrocarbons on bed of carbon sorbent	162
	Derivatization of mercury compounds	Effect of conditions of derivatization (using Grignard reagent) on final determinations	163
	Evaporation of excess solvent from extract	Purification level of gas used at stage of excess solvent evaporation	164
	Final determinations using AED detector	Effect of labelled and unlabelled compounds on determination of ^{15}N content	165
	Determination of pH of rainwater samples	Effect of low values of ionic strength of rainwater samples on performance of glass electrode	166
	Final determinations (using GC-MS)	Effect of water on sensitivity, accuracy and breaks in operation of mass spectrometer as result of incomplete drying of gas stream	167

	Separation and final determinations of components	Corrosion and passivation of part of surface of stainless steel devices as result of contact with aggressive components of mobile phase	[168-169]
	Determination of TOC levels in waste water	Effect of process of sample preparation (filtration, acidity) on determination results	49
	Determination of phosphates in aqueous medium	nterferences caused by presence in solution of chromates, germanates, vanadates and tungstates (when using phosphoantimonymolybdenum blue technique)	170
		Interferences caused by presence in solution of arsenates, fluorides and silicates (when using phosphoantimonymolybdenum blue technique)	171
	Chromatographic determination of nitrophenols in water (without derivatization)	Effect of derivatization level of separation system on determination results	172

Liquid samples	Stage of final determinations (GC)	Losses of analytes as result of use of inappropriate transfer line between column and detector	173
	Preparation of vessels for samples	Effect of preparation measures (cleansing and washing) on results of Pb determination	174
	Preparation of standard solutions	Stability of solution composition (selenium compounds)	[175-176]
		Stability of mercury compounds (organic and inorganic) in organic solvents	177
	Storage of standard solutions	Formation of volatile selenium compounds (DMSe, MMSeS, DMDSe) from inorganic and organic mercury compounds in synthetic seawater	178
		Light-induced degradation of dioxins (PBDD/PBCDD) in dodecane	179
		Stability of solutions of arsenic(III) and (IV) compounds in water sample	180
Solid samples	Sampling	Effect of particle segregation and sample size on final determinations	181
	Sampling of river sediments	Variability of concentrations of aliphatic hydrocarbons in top layer of bottom sediments	182
	Preparation of certified reference materials	Changes in moisture content	183
	Storage of reference materials	Instability of reference materials (biological material)	184
		Changes in amounts of organic analytes stored on bed of sorbent (Tenax-GC)	185
		Stability of materials	186
	Storage of sediment samples	Instability of samples (changes in concentrations of PAHs, n-alkanes, carbon, Al, and Pb)	187
		Oxidation of Cr(III) to Cr(IV) during extraction of chromium compounds from solid samples with hot alkali solution	188
		Escape of PAH analytes from sediment samples stored in surrounding conditions	189
		Effect of contact with laboratory air on concentration levels of PAH analytes	190
	Preparation of sediment samples	Formation of methylmercury from Hg^{+2} during sample preparation for analysis	191
	Extraction of sediment samples	Formation of PCDD analytes during extraction in Soxhlet apparatus	192

Talbe 11 (continued)

	Preparation of high-purity materials	Effect of water quality on characteristics of semiconductors manufactured	115
		Effect of level of air purification in 'clean room' on quality of semiconductors	[193-195]
	Drying of sample	Effect of method of sample drying on results of metal concentration assays	196
	Preparation of sediment samples	Effect of method of sample drying on results of heavy metal assays	197
	Digestion of sediment samples	Effect of sample digestion technique on results of heavy metal assays	198
	Preparation of plant material samples	Effect of rubbing technique on results of heavy metal assays	199
	Digestion of organic material used in electronic industry	Effect of digestion technique on results of heavy metal assays	200
Solid samples	Extraction of organotin compounds from sediment samples	Effect of kind of complexing agent and acid modifier on efficiency of extraction using supercritical fluid	201
	Conservation of samples	Changes in methylmercury concentration and total mercury content of sample during storage. Study of conserving agents	139
	Digestion of sediment samples and extraction of metals	Changes in recovery level depending on sample preparation techniques employed	202
	Ashing of samples	Effect of ashing conditions on determination of trace amounts of metals	[203-204]
	Determination of volatile organic compounds in soil using PT-GC-MS technique	Estimation of sources of error at all stages of analytical procedure	205
	Addition of standard to sediment or soil sample	Losses of analytes (PCDD) from sample during: - evaporation of solvent, - differences in adsorption strength of 'actual' and 'added' components of sample	206
	Validation of method of TBT and TPT determination in sediments	Defining effect of way of addition of standard to sample on level of analyte recovery	74
	Determination of heavy metals in solid waste samples	Interference during determination of trace amounts of metals using ICP-AES-HG technique	207

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