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**SEPARATION AND PRECONCENTRATION
OF TRACE SUBSTANCES.
I—PRECONCENTRATION FOR INORGANIC
TRACE ANALYSIS**

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SEPARATION AND PRECONCENTRATION OF TRACE SUBSTANCES.
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INTRODUCTION

The development of the theory and practice of preconcentration helps analytical chemists to solve many important problems which arise in connection with progress in nuclear technology, electronics, metallurgy, environmental research etc. The wide use of modern instrumental techniques of determination does not remove the necessity for preconcentration methods, as is sometimes believed. On the contrary, new advantages come to light, that will ensure the use of these methods in the future.

In this report, some general aspects of preconcentration are considered - terminology, importance, fields of application, and the rational combination of preconcentration with determination methods.

TERMINOLOGY

Primarily, the terminology of methods of separation and isolation (extraction, precipitation, ion exchange etc.) is used for the description of preconcentration. However specific terms may be emphasized:

Preconcentration - an operation (process) as a result of which the ratio of the concentration or the amount of microcomponents (trace constituents) and macrocomponents (matrix) increases. The term "enrichment" is not recommended.

Absolute preconcentration - an operation (process) as the result of which microcomponents are transferred from the sample of larger mass into the sample of smaller mass, so that the concentration of the microcomponents is increased. Examples include the decrease in solvent volume during distillation or evaporation, and the transfer of microcomponents from an aqueous solution into a smaller volume of organic solvent by extraction.

Relative preconcentration - an operation (process) as a result of which the ratio of concentration or amounts of microcomponents and main macrocomponents increases. This is a particular example of a separation, but at greatly differing concentrations of components. The ratio of the masses of the initial and final samples is not of great importance. Frequently, the main purpose of relative preconcentration is the replacement of a matrix unsuitable for analysis by a suitable medium. In some cases, it is difficult to trace a boundary between the absolute and the relative preconcentrations as, for example, in preconcentration by zone melting.

Selective preconcentration - an operation (process) as a result of which microcomponents are selectively isolated from the sample. It is used when the simultaneous presence of several components in the concentrate may distort the results of analysis. Selective preconcentration usually is achieved by isolation of the microcomponent to be determined.

Group preconcentration - an operation (process) as a result of which several microcomponents are isolated in one step. This may be achieved in one of two ways, by transportation either of the matrix or of the microcomponents into a second phase. The first method is widely used in the analysis of relatively simple substances and materials.

Recovery (R) - the yield of a preconcentration step expressed as quantities of the concentrated microcomponent: $R = Q_T / Q_T^0$, where Q_T and Q_T^0 are the quantities of the microcomponent in the concentrate and in the sample, respectively (mass units or concentration units). Usually, recovery is calculated as a percent: $R(\%) = 100 Q_T / Q_T^0$

Preconcentration coefficient (K) of a desired microcomponent. This is defined as $K = (Q_T / Q_M) / (Q_T^0 / Q_M^0)$, where Q_M^0 and Q_M are the quantities of the matrix before and after preconcentration, respectively. If the recovery is 100 %, $K = Q_M^0 / Q_M$. The terms "enrichment coefficient" and "enrichment factor" are not recommended.

IMPORTANCE AND FIELDS OF APPLICATION

Frequently, preconcentration enables a relative detection limit to be improved by two to three orders of magnitude or more. Spectroscopic methods with chemical pretreatment of the sample are an example. The use of preconcentration enables emission spectrographic analysis at the same concentration levels as methods like neutron activation analysis. In some cases, preconcentration also ensures the lowering of the absolute detection limit. Thus, in atomic absorption spectrophotometric analysis, the introduction of a combustible extract of microcomponents into the flame usually favourably effects the conditions of sputtering and atomization, so that the sensitivity of determination increases and the detection limit improves.

The use of preconcentration may increase the accuracy and precision of determinations as a result of the elimination of interferences, but it also may decrease them owing to losses of components being determined or to contamination. The total effect is determined by a rational choice of operations. Multielement analysis of various samples is often carried out, where "background" interferences of different nature and origin may badly influence the accuracy and precision of the determination (and hence the detection limits). Preconcentration decreases the "memory" of the analytical instrument, thus increasing the reliability of the determination.

Preconcentration allows the use of unified standard reference materials when samples of various types have to be analysed, especially by physical methods. The dissolution of samples and the transfer of microcomponents to be determined into a single universal matrix, for example into an acidic solution of some microcomponents as a trace concentrate or into carbon powder for spectrochemical analysis, substantially facilitates calibration. Thus solutions of different elements are used as standard samples (irrespective of the nature of the samples under analysis, after the separation of the matrix) in spectral emission, atomic absorption, X-ray fluorescence, neutron activation, solution spectrophotometry and luminescence methods.

Preconcentration facilitates sampling. The error of sampling of solids increases with decrease in the amount of the analytical sample. For some methods, the need to use a small sample is an important factor, as, for example, for spark source mass spectrometry, operating with samples of 10-20 mg. Preconcentration allows the components to be determined to be isolated from a large, weighed amount of the sample, thus diminishing the sampling error and eliminating the influence of sample non-homogeneity on the analytical measurement.

Preconcentration of microcomponents widens the range of materials which may be analysed by a chosen method. Thus, the spectrographic determination of elements in a matrix giving a line-rich spectrum (lanthanoid, actinoid, iron, molybdenum) is complicated by the superposition of the macrocomponent's spectral lines upon the analytical lines of the microcomponent elements. In spectrophotometric determinations, the aqueous solution may contain other coloured substances, in particular, the reagent in excess. Spark source mass spectrometry allows electroconducting substances, dielectrics and insulators to be analysed only after mixing with a suitable metal powder and pressing them to form an electroconductive electrode. The neutron activation analysis of materials containing a strongly activated matrix becomes difficult owing to the high induced radioactivity. The preconcentration of microelements,

and the separation of the matrix diminishes such difficulties.

In some cases, preconcentration widens the number of components which may be determined by the given method (though sometimes the number of components is diminished).

Preconcentration has its drawbacks. In most cases, it lengthens the analysis and frequently complicates it; reagents of high purity are needed, especially when common elements, for example, aluminium, magnesium, calcium, iron are determined, sometimes in great amounts; in some cases, special equipment and mastering of specific working procedures are desirable. Preconcentration may be affected by losses of the elements being determined or by introduction of external contaminants. However the advantages of preconcentration frequently exceed its drawbacks.

METHODS

The following methods of preconcentration are most widely used: 1) extraction (including extraction chromatography), 2) sorption methods (absorption, ion-exchange), 3) coprecipitation and precipitation, 4) distillation (including fractional evaporation, sublimation), 5) electrochemical methods (electrodeposition, electrodialysis, cementation, ionophoresis), 6) zone melting (zone recrystallization), 7) ashing. Other methods are also known: flotation, ultracentrifugation, ultrafiltration, gel filtration, dialysis, diffusion, thermodiffusion.

A method should be relatively simple, ensure pure separation of macro- and microcomponents and be in accord with the method of determination. One of the important factors determining the choice of the techniques of preconcentration is the history of the sample to be analysed. The preconcentration method must differ from the method of substance purification.

When the method of preconcentration is chosen, information or assumptions about the forms of the microcomponents in the sample is also important (the state of oxidation, the homogeneity of distribution by volume, its uniformity of nature - mineral or organic, the ionic or colloid state in solutions etc. should be considered). This is important irrespective of the fact that preconcentration (and other chemical operations of sample treatment) converts the microcomponents to a homogeneous state independent of the initial form. The mutual influence of the matrix and the microcomponents during sample processing must also be taken into account. For example, the possibility of azeotrope formation during distillation, and of coprecipitation during precipitation must be taken into consideration.

It is reasonable to discuss some particular aspects of certain methods of preconcentration. Liquid-liquid extraction is the most important and widely used preconcentration method. It is based on the distribution of the dissolved substance among immiscible liquid phases. The method is characterized by versatility, it is suitable for removing the matrix and for the isolation of microcomponents, with high efficiency. The extraction may be used for selective or group preconcentration. The method is simple, and in most cases, rapid and may be automated fairly easily. A limitation of the technique is the relatively low preconcentration coefficient, e.g. though one of the extraction variants, extraction chromatography, can ensure a very high coefficient of absolute preconcentration.

Sorption techniques include a large group of methods and are based on the distribution of components being separated between two phases, one of which is stationary and the other is mobile. The advantage of some chromatographic methods is the possibility of combining the preconcentration and determination steps, as well as improving the speed of determination, and the possibility of separation of components with similar properties, and of achieving high values of the preconcentration coefficient. The methods enable analyses of microamounts of substances. Ion-exchange chromatography is not widely used owing to the great volumes of solutions being treated and, consequently, to a great degree to the variation in the blank, and to some diffusional limitations. Static ion-exchange, which is much simpler and more readily carried out, is practised quite widely.

Coprecipitation based on the isolation of microcomponents in the precipitate of organic or inorganic collector is not as widely used as extraction owing to the great expenditure of labour and time. However, it is one of the best methods because of the high coefficient of absolute preconcentration and the simplicity of the equipment necessary. Coprecipitation of

microcomponents is accomplished either by the introduction of additional compounds or by the precipitation of a small fraction of the matrix. The method is often used in combination with methods of analysis of solids. Precipitation as a method of preconcentration requires large amounts of reagents and yields insufficient accuracy, caused to a considerable degree by the blank error.

Distillation methods based on the difference in temperature of evaporation (sublimation) of the matrix and the microcomponents are widely used owing to their simplicity, high coefficient of absolute concentration, rapidity and small blank error. One of the variants of the method, fractional distillation, is achieved in emission spectrographic analysis and favours the attainment of high accuracy and precision.

Group and selective preconcentration may be accomplished by electrochemical methods. In some variants (electrodeposition on mercury or a solid cathode, cementation) high preconcentration coefficients are obtained. The equipment for preconcentration is not complicated, and the blank error is not large because electrochemical methods do not require large amounts of reagents.

Zone melting based on the different solubility of microcomponents in the liquid and solid matrix is a technique which does not require reagents, but it has a limited field of application for the analysis of fusible (and stable) substances. The method is relatively simple, ensures high preconcentration coefficients and is easily automated. The method has limitations, mainly its long duration and the possibility of sample contamination by the container material.

The field of use of ashing (dry or wet mineralization) is the elemental analysis of organic (and organometallic) compounds, and vegetable and animal material. The method is simple. Unfortunately, dry mineralization is frequently affected by losses of elements and wet mineralization by external contamination.

Thus, there is no universal method of preconcentration. Each method has its field of application depending on the purpose, its advantages and limitations.

GENERAL METHODS FOR EVALUATING AND REPORTING PRECONCENTRATION TECHNIQUES

When a new preconcentration technique is proposed, the following items should be included in the report: 1) sample (matrix); 2) sample amount; 3) microcomponent(s) to be separated and determined; 4) concentration level of the microcomponent(s); 5) reagents and apparatus used; 6) procedure; 7) recovery of the microcomponent(s); 8) preconcentration coefficient of the microcomponent(s); 9) contamination; 10) time required; 11) method(s) of determination following the preconcentration.

Recovery. A recovery of less than 100 % is often obtained, because there are several possible sources of loss of microcomponents during the preconcentration and related steps. These include evaporation of microcomponents during decomposition of the sample prior to preconcentration, incomplete decomposition, imperfect separation, careless manipulation, and adsorption of the microcomponent on the containers and other apparatus used.

The required recovery depends on the purpose of the preconcentration. Thus, recoveries larger than 95 % are acceptable in most quantitative trace analyses. Much lower and even variable recoveries are permissible in isotope dilution analysis and radiochemical separations using isotopic carriers.

The recovery and loss of a microcomponent are best investigated by the radioactive tracer technique. In this technique, a radioactively-labelled element or compound chemically identical with the microcomponent is added to the sample before preconcentration, and its behaviour is followed by sensitive, rapid and selective radioactivity measurements. Even continuous measurements can easily be made. The great advantage of this technique is that the recovery is evaluated independently of contamination hazards. Both the isotope and the radiation effects are negligible in most cases.

A limitation of the application of this technique is the difficulty of introducing tracers into solid samples. Radioactive labelling of microcomponents in solid samples is required when the tracer technique is applied to investigate the recovery and loss of microcomponents during decomposition of solids and separations involving volatilization and solvent extraction of microcomponents from solids. Radioactivation and synthesis of artificial samples

containing radioactive isotopes are sometimes useful for this purpose.

In the absence of available radioactive tracers, certified standards, analyzed samples or synthetic samples are used for evaluating recoveries. The method of standard addition is also useful. But in all these cases, contamination should be negligible or reproducible and accurately determinable.

Preconcentration coefficient. The preconcentration coefficient required depends upon the concentration level of the desired microcomponent in the sample and also on the permissible value of Q_M/Q_T for the determination method used. Since most instrumental methods of determination are sensitive and rather selective, preconcentration coefficients of 10^2 to 10^4 are often sufficient.

The preconcentration coefficient is evaluated by simply measuring the quantity of the matrix after preconcentration by one of the conventional determination methods (e.g., atomic absorption spectrophotometry), because the recovery of the microcomponent, measured as described above, and the sample size are known.

Contamination. During the preconcentration and related steps, the sample may become contaminated by the microcomponent to be determined and/or by interfering compounds. The contaminants originate from external sources, which include the laboratory atmosphere, the reagents and the apparatus used, and the analyst performing the analysis. Since most kinds of contamination are not reproducible, it is very difficult to measure them with sufficient accuracy. Preconcentration is often rendered useless by contamination. Therefore, every effort must be made to reduce the contamination level to an acceptable value.

In order to estimate the overall contamination, a "blank run" is usually carried out under the same conditions and at the same time as an actual determination but without the sample. However, this method is unsatisfactory, because the conditions cannot be identical in the absence and presence of the sample, e.g., the corrosion of the surfaces of containers may be different. A combined use of a sample of known composition and a radioactive isotope provides the best information on both loss and contamination. It is recommended that the contamination from each source or in each step, if possible, be estimated separately. The contamination is sometimes more accurately estimated by using several times the normal amount of the reagent in the blank run and dividing the blank by the corresponding factor, in cases where contamination by a particular reagent is dominant.

COMBINATION WITH METHODS OF DETERMINATION

Two groups of techniques may be distinguished which are the result of combining preconcentration with subsequent determination. In the methods of the first group, preconcentration and determination are not closely connected, and the properties of the concentrate do not substantially influence the manner of the final determination. In such cases, it makes almost no difference in which way the concentrate is obtained - by ion exchange, liquid-liquid extraction with subsequent back extraction or by some other way. In all such cases, a concentrate is obtained in the form of an aqueous solution, and may be analyzed by different methods. Such a combination will have no important specific peculiarities. Such methods may be called combined methods.

However, the properties of the concentrate frequently require a particular method of determination; specific peculiarities of the combination of a preconcentration with a determination cause, a new quality to appear. The methods of preconcentration and determination do not form a "mechanical mixture", but a "chemical compound". For example, in gas chromatography, the separation device (chromatographic column) and the equipment for non-selective determination of the separated components (detector) have become one apparatus. Such methods may be called hybrid methods. Besides gas chromatography, high pressure liquid chromatography, extraction-spectrophotometric and extraction-atomic absorption methods, etc. can be considered as hybrid methods.

Depending on the task, one or several components may or must be determined. If the preconcentration is orientated to multielement analysis (emission spectroscopy, X-ray fluorescence spectroscopy), the isolation of the largest number of components to be determined becomes its most important task.

Multielement analysis is usually more economical than mono-element analysis as it ensures a rise in the productivity of labour. Unfortunately, selective preconcentration in combination with multielement methods of determination is frequently described.

Most frequently, preconcentration is combined with spectrophotometry, emission spectroscopy and flame atomic absorption spectrophotometry. The combination of preconcentration with the polarography of the extracts is also known; in this case, the decomposition of the extracts and the associated errors are excluded, the duration of the analysis is shortened and the consumption of reagents diminished.

Combinations favouring the indirect determination of elements are of interest. For example, microamounts of phosphorus can not be directly determined by atomic absorption or polarographic methods. However, if phosphorus is concentrated as 12-molybdophosphoric acid, it is possible to determine this element indirectly by determining molybdenum.

The continuous appearance of new determination methods places increasingly strict requirements for claims with regard to the duration of operations, their simplicity, the possibility of isolation of a large number of components etc. Preconcentration accommodates itself to the necessities of new determination methods and frequently widens their possibility. For instance, atomic absorption spectrophotometry with a graphite furnace initially developed as a method without matrix separation. However it was found that the matrix lowers the absolute detection limit (often very strongly) owing to non-selective interferences, and preconcentration proved to be very useful in minimizing this problem. Spark source mass spectrometry, ensuring very low detection limits for at least 70 elements, has a limitation: the size of the weighed sample under analysis is very small, therefore the relative detection limit is usually not lower than 10^{-5} - 10^{-7} %. Preconcentration allows a more complete realization of the possibilities of this method.

Sometimes, it is useful to combine decomposition and preconcentration steps. An example is fire assay. Such a combination proved to be successful in ultra trace analysis, for instance during the determination of picograms of elements which can be easily and quantitatively volatilized in the elemental form or as oxides, halides or hydrides, from non-volatile matrix elements.

MULTIELEMENT PRECONCENTRATION FROM HIGH-PURITY METALS

For the routine analysis of pure metals spectroscopic methods are preferred because of their advantages as to sensitivity, speed and applicability to multielement determination in different sample materials. Whenever possible, chemical pretreatment of the sample is avoided so as to reduce possible sources of contamination, and to minimize the extent of assistance, equipment and time. In the analysis of high-purity metals, however, different interfering effects arise (i.e. spectral interferences, quenching of the analytical signal, "dilution" of the trace content by the matrix) owing to which pretreatment of the sample is indispensable for obtaining useful analytical data. Combined with suitable chemical pretreatment, optical emission spectroscopy, atomic absorption spectrophotometry, radiochemical and electro-analytical methods are widely applied in trace analysis of high-purity metals.

The most important pretreatment step is the separation of the matrix and the preconcentration of the trace elements to be determined. Depending on the chemical properties of the macro- and microcomponents of different pure metals, it may be advantageous to transform either the matrix or the trace impurities of the sample into compounds which dissolve in a separable second phase.

Review of preconcentration methods. The following review outlines the great variety of analytical techniques published mainly in the past 20 years. It is not the intention to cite all the numerous authors and their methods, but to restrict the selection to such procedures which include preconcentration of a comparatively large number of different metallic impurities and which seem to be typical for the pure metal in question. In this review, physical methods of preconcentration are not considered as being an integrated component of a determination step (e.g. carrier distillation in emission spectroscopy, electrolysis in anodic stripping voltammetry). In general, the quotation of a particular method represents several similar publications in this field, without any order of priority. Yet, it cannot be expected that this collection of methods is an unexhaustible source for solving problems

in the field of trace analysis of metals.

When applying trace analytical methods obtained from literature, it must be considered that each procedure is valid only for a limited upper and lower range of sample weight, microelement concentration, etc. Authors often fail to give sufficient information about the limitations of their methods. Furthermore, the successful application of a trace analytical technique may be extremely dependent on the special conditions of the laboratory and the chemist's mode of work. Many examples in this respect can be found in literature. Thus it may be said that perhaps in no other field of analytical chemistry is the careful examination of the utility of a method so important for success as it is in trace analysis, and the smaller the amounts or concentrations to be determined, the more important it is.

The preconcentration methods listed in the following table have been obtained mostly from Analytical Abstracts or from the abstracts in "Zeitschrift für Analytische Chemie". Data about "Technique of Preconcentration" and "Preconcentrated Elements" are also obtained mainly from these abstracts. No further details of the special procedures for trace concentration, sample weighing, recovery, precision, accuracy, etc. are given, because such data are found only rarely and incompletely in the abstracts of most of the quoted papers. Methods for microelement determination have been omitted which have been used by different authors in connection with the preconcentration technique described. For more details, the list of original publications which is given below, should be consulted.

In the table the following abbreviations are used: C:collector; P:precipitate; R:reagent; S:solvent.

Matrix Element	Technique of Preconcentration	Preconcentrated Trace Elements	Quotation Number
Ag	Matrix precipitation (P: AgCl)	Al, Au, Ca, Co, Cu, Fe, Hg, Ir, Mg, Mn, Ni, Os, Pd, Pt, Rh, Ru	1
Al	Extraction of trace elements (R: NH ₄ -pyrrolidinedithiocarbamate; dithizone S: chloroform)	As, Bi, Cd, Co, Cr, Cu, Fe, Ga, Mn, Mo, Ni, Pb, Sb, Sn, V, Zn	2
	Extraction of trace elements (R: Na-pyrrolidine-1-carbodithioate; S: isobutyl methyl ketone)	Bi, Cd, Cu, Fe, Ga, Ni, Pb, Sb, V, Zn	3
	Extraction of trace elements (R: diantipyrinylmethane; S: chloroform)	Bi, Cd, Cu, Ga, In, Pb, Sb, Sn, Te, Tl, Zn	4
Alkali metals	Extraction of trace elements (R: 8-hydroxyquinoline, diethyldithiocarbamate; S: chloroform/isoamyl alcohol)	Ag, Al, Au, Bi, Cd, Co, Cu, Ga, In, Mn, Ni, Pb, Ti, Tl, Zn	5
	Precipitation of trace elements (C: La(OH) ₃)	36 elements	6
	Precipitation of trace elements (R: Na-diethyldithiocarbamate; C: activated carbon)	Ag, Bi, Cd, Co, Cu, Fe, In, Ni, Pb, Tl, Zn	7
Alkaline earth metals	Precipitation of trace elements (R: H ₂ S, Na-diethyldithiocarbamate; C: carbon powder, CdS)	Ag, Al, As, Au, Bi, Co, Cr, Cu, Fe, Ga, Ge, In, Mn, Mo, Ni, Pb, Sb, Sn, Ti, Tl, V, Zn	8
	Extraction of trace elements (R: Na-diethyldithiocarbamate; S: chloroform)	Bi, Cd, Cu, Fe, Mn, Pb, Sb, Zn	9
As	Extraction of the matrix (AsBr ₃ ; S: benzene)	Ag, Al, Au, Ba, Be, Bi, Co, Cr, Cu, Fe, Ga, In, Mg, Mn, Ni, Pb, Te, Tl, Zn	10
	Distillation of the matrix (As ₂ O ₃)	Al, Ba, Cd, Co, Cr, In, Mg, Mn, Sn, Zn	11
	Distillation of the matrix (AsBr ₃)	Ag, Au, Cd, Co, Cr, Cu, Mn, Ni, Pb, Sn, Zn	12
Au	Extraction of the matrix (AuCl ₃ ; S: isopentyl acetate)	Ag, Al, Bi, Cd, Co, Cu, Fe, Mn, Ni, Pb, Zn	13
	Extraction of the matrix (AuBr ₃ ; S: isopropyl ether)	Bi, Cd, Cu, Fe, Ni, Pb, Zn	14
Be	Extraction of trace elements (R: Na-diethyldithiocarbamate; S: ethyl acetate)	Cd, Co, Cu, Fe, Mn, Ni, Pb	15
Bi	Precipitation of the matrix (P: BiOBr)	Al, Ca, Cd, Co, Cu, Fe, Ga, In, Mg, Mn, Ni, Tl, Zn	16
	Extraction of the matrix (S: tributyl phosphate)	Al, Co, Cr, Cu, Mn, Ni, Pb, Ti, Zn, Zr, alkali metals, alkaline-earth metals, lanthanoids	17
Cd	Precipitation of trace elements (C: MnO ₂ (1); Al(OH) ₃ (2))	As, Bi, Ga, In, Ni, Pb, Sb, Sn, Te, Ti (1) As, Bi, Cr, Ge, In, Mn, Pb, Sn, Te, Ti, V (2)	18
	Extraction of the matrix (R: HI, pyridine; S: chloroform)	Al, Bi, Ca, Co, Fe, Ga, In, Mg, Na, Ni, Sb, Sn, Zn	19

(continued)

Matrix Element	Technique of Preconcentration	Preconcentrated Trace Elements	Quotation Number
Co	Ion exchange	Bi, Cd, Pb, Sn, Zn	20
	Extraction of trace elements (R: fatty acids; S: chloroform)	Bi, Cu, Fe, Pb, Sn, Zn	21
Cr	Distillation of the matrix (CrO ₂ Cl ₂)	Al, Cu, Fe, Mn, Ni, Si, Ti, Zr	22
	Distillation of the matrix (CrO ₂ Cl ₂)	Ag, Al, Cu, Fe, Mg, Pb	23
	Extraction of trace elements (R: Na-diethyldithiocarbamate; S: chloroform)	Bi, Cd, Co, Cu, Fe, Mn, Ni, Pb, Zn	24
Cu	Electrodeposition of the matrix	Ag, Al, Au, Ba, Bi, Ca, Cd, Co, Cr, Fe, Mg, Mn, Mo, Ni, Pb, Pt, Sb, Sn, Te, Ti, V, Zn	25
	Precipitation of trace elements (C: La(OH) ₃)	As, Bi, Fe, Pb, Sb, Se, Sn, Te	26
Fe	Extraction of the matrix (FeCl ₃ ; S: bis-(2-chloroethyl) ether)	Ag, Al, As, Ba, Bi, Ca, Cd, Co, Cr, Cu, In, Mg, Mn, Ni, Pb, Pt, Te, Ti	27
	Ion exchange	Mo, Nb, Ti, W, Zr	28
Ga	Extraction of the matrix (GaCl ₃ ; S: bis-(2-chloroethyl) ether)	16 elements	29
	Partial dissolution of the matrix(1) or stripping of the trace elements from the melted sample(2)	Ag, Au, Bi, Co, Cu, Fe, Hg, Ni, Pb, Pd, Sn (1) Cd, Co, Fe, Tl, Zn (2)	30
	Extraction of the matrix (R: alkyl phosphate; S: carbon tetrachloride)	As, Cd, Co, Cr, Cu, Mn, Ni, Se, Te	31
Ge	Distillation of the matrix (GeCl ₄)	Ag, Al, Au, Ba, Bi, Ca, Cd, Co, Cr, Cu, Fe, In, Mg, Mn, Mo, Ni, Pb, Sb, Sn, Ti, Tl, V, Zn	32
	Precipitation of the matrix (GeO ₂ ·aq)	Ag, As, Au, Cd, Co, Cu, Fe, Ga, Hg, In, Pb, Re, Zn	33
Hg	Precipitation of the matrix (Hg; R: formic acid)	Al, As, Be, Bi, Cd, Cr, Co, Cu, Fe, Ga, In, Mg, Mn, Ni, Pb, Sb, Sn, Te, Ti, Tl, Zn, alkali and alkaline-earth metals	34
	Distillation of the matrix (Hg)	Ag, Bi, Cd, Cu, Pb, Sn, Zn	35
	Extraction of the matrix (S: isoamyl alcohol)	Ag, Al, Ca, Cd, Cu, Mg, Mn, Ni, Pb, Zn	36
In	Extraction of the matrix (InBr ₃ ; S: isopropyl ether)	Bi, Ca, Cd, Co, Cu, Hg, Mg, Ni, Pb, Zn	37
	Extraction of the matrix (1) (InBr ₃ ; S: diisopropyl ether)	Al, Ba, Be, Bi, Ca, Cd, Co, Cr, Cs, Eu, K, Li, Mg, Mn, Na, Ni, Pb, Pd, Pt, Rb, Rh, Sc, Sr, Ti, V, Y, Yb, Zr (1)	38
	Precipitation of trace elements (R: cupferron (2); C: CuS (3))	Bi, Fe, Hf, Mo, Nb, Sn, Ta, Ti, V, W, Zr (2) Au, Bi, Cd, Hg, Mo, Pd, Sb (3)	

(continued)

Matrix Element	Technique of Preconcentration	Preconcentrated Trace Elements	Quotation Number
In	Distillation of the matrix (InBr)	Ag, Al, Au, As, Bi, Cd, Co, Cr, Cu, Fe, Ga, Ni, Mn, Pb, Sb, Sn, Te	39
Ir	Ion exchange	Cu, Fe, Ni	40
La	Precipitation of trace elements (R: Na-diethyldithiocarbamate, polyacrylamide, 8-mercaptoquinoline; C: graphite powder)	Co, Cr, Cu, Fe, Mo, Mn, Nb, Ni, Pb, Ta, Ti, V, W	41
Mg	Precipitation of trace elements (C: SnO ₂ ·aq)	Co, Cu, Fe, Zn	42
Mn	Ion exchange	Co, Cu, Fe, Pb, Zn	43
Mo	Extraction of the matrix (R: trialkylbenzylammonium-nitrate)	Al, Ba, Be, Bi, Cd, Cr, Cu, Fe, Ga, Ge, In, Mg, Mn, Ni, Sb, Sn	44
	Precipitation of trace elements (R: 1-(2-pyridylazo)-2-naphthol)	Co, Cu, Fe, Mn, Ni, Zn	45
	Ion exchange	Co, Cu, Fe, Mg, Mn, Ni, Pb, Zn	46
Nb	Extraction of trace elements (R: Na-diethyldithiocarbamate; S: chloroform)	Ag, Au, Bi, Cd, Co, Cu, Fe, In, Mn, Ni, Pb, Se, Tl, Zn	47
	Ion exchange	Ag, Au, Ba, Ca, Cd, Co, Cu, Fe, Ga, Hf, Hg, In, K, Mn, Mo, Na, Nb, P, Pa, Rb, Re, S, Sc, Sn, Sr, Ta, W, Zn, Zr, lanthanoids	48
Ni	Distillation of the matrix (Ni(CO) ₄)	As, Bi, Cd, Co, Cu, Mn, Pb, Sb, Sn, Zn	49
	Ion exchange	As, Bi, Cu, Sb, Zn	50
Os	Distillation of the matrix (OsO ₄)	Au, Fe, Ir, Pd, Pt	51
Pb	Precipitation of the matrix (PbCl ₂)	Ag, Al, As, Au, B, Ba, Be, Bi, Ca, Cd, Co, Cr, Cu, Fe, Ga, In, K, La, Mg, Mn, Mo, Na, Ni, P, Pd, Pt, Sb, Si, Sn, Sr, Tl, U, Zn	52
	Precipitation of the matrix (Pb(NO ₃) ₂)	Ag, Al, As, Bi, Ca, Cd, Co, Cu, Fe, Ga, In, Mg, Mn, Na, Ni, Sb, Tl, Zn	53
	Precipitation of the matrix (PbSO ₄)	Ag, Al, As, Bi, Ca, Cd, Co, Cr, Cu, Fe, In, Mg, Mn, Mo, Na, Ni, Sb, Sn, Te, Ti, V, Zn	54
Pd	Extraction of the matrix (PdBr ₂ ; S: cyclohexanone)	Cd, Cu, Ni, Pb, Zn	55
	Extraction of the trace elements (1) (R: dibutyl phosphorothioate; (0.01N HCl); S: carbon tetrachloride)	Ag, Cd, Cu, Fe, Ga, In, Se, Sn, Te, Zn (1)	56
	Extraction of the matrix (2) (R: dibutyl phosphorothioate; (N HCl); S: carbon tetrachloride)	As, Co, Ir, Mn, Ni, Pt, alkali and alkaline-earth metals (2)	

(continued)

Matrix Element	Technique of Preconcentration	Preconcentrated Trace Elements	Quotation Number
Pt	Ion exchange	Al, Ba, Be, Bi, Ca, Cd, Cu, Fe, Mg, Mn, Ni, Pb, Zn	57
Re	Extraction of the matrix (HReO ₄ ; S: trioctylamineoxide, toluene)	Al, As, Ba, Be, Bi, Ca, Cd, Co, Cr, Fe, In, Mg, Mn, Ni, Pb, Sb, Si, Te, Tl, Zn	58
Rh	Ion exchange	Cu, Fe, Ni, Pb	59
Sb	Extraction of the matrix (HSbCl ₆ ; S: butyl acetate)	Ag, Al, Bi, Ca, Cd, Co, Cr, Cu, In, Mg, Mn, Ni, Pb, Pt, Ti, Zn	60
	Distillation of the matrix (SbCl ₃)	Ag, Al, Bi, Cd, Co, Cr, Cu, Fe, Mg, Mn, Ni, Pb	61
Sc	Extraction of trace elements (three-phase system; S: 1,1-diantipyrinylheptane, chloroform)	Ga, In, Tl	62
Se	Extraction of trace elements (R: 8-hydroxyquinoline, dithizone; S: chloroform)	Ag, Al, Au, Bi, Cd, Co, Cu, Fe, Ga, Hg, Hf, In, La, Mn, Mo, Ni, Pb, Pd, Pt, Sb, Sc, Sn, Th, Ti, Tl, U, V, Y, Zn, Zr	63
	Distillation of the matrix (SeO ₂)	Al, As, Ba, Bi, Ca, Cd, Co, Cr, Cu, Fe, Hg, Mg, Mn, Mo, Ni, Pb, Sb, Sn, Te, Ti, V, Zn, Zr	64
	Precipitation of the matrix (Se; R: hydrazine)	As, Cd, Cu, Fe, Ga, Hg, In, Sb, Te, Tl	65
Si	Distillation of the matrix (SiF ₄)	As, Au, Co, Cr, Cu, Fe, Ga, In, K, La, Mo, Na	66
Sn	Ion exchange	Co, Cu, Fe, Mn, Mo, Ni, Pb	67
	Distillation of the matrix (SnBr ₄)	Ag, Au, Cd, Co, Cr, Cu, Fe, Ga, In, La, Na, Ni, Sc, W, Zn	68
	Precipitation of trace elements (C: La(OH) ₃)	Ag, Bi, Cd, Co, Cu, Fe, Hg, In, Mn, Ni, Pb, Tl, Zn,	69
Ta	Extraction of trace elements (R: Na-diethyldithiocarbamate; S: chloroform)	Ag, Au, Bi, Cd, Co, Cu, Fe, In, Mn, Ni, Pb, Se, Tl, Zn	47
	Precipitation of trace elements (C: CuS)	Bi, Cd, Pb, Sb, Sn	70
Te	Precipitation of the matrix (TeO ₂)	Ag, Al, As, Au, Bi, Ca, Cd, Co, Cr, Cu, Fe, In, Mg, Mn, Na, Ni, Pb, Sb, Si, Sn, Tl, Zn	71
	Precipitation of the matrix (Te)	Al, As, Bi, Cd, Co, Cr, Cu, Fe, Ga, In, Mg, Mn, Mo, Ni, Pb, Sb, Sn, Ti, Tl, V, W, Zn	72
Th	Ion exchange	Sc, Y, rare earths	73
	Extraction of the matrix (R: tributyl phosphate; S: carbon tetrachloride)	rare earth metals	74
Ti	Extraction of trace elements (R: Na-diethyldithiocarbamate; S: chloroform)	Ag, Au, Bi, Cd, Co, Cu, Fe, In, Mn, Ni, Pb, Se, Tl, Zn	47

(continued)

Matrix Element	Technique of Preconcentration	Preconcentrated Trace Elements	Quotation Number
Ti	Ion exchange	Ag, As, Au, Ba, Ca, Co, Cr, Cu, Fe, Ga, Hf, In, K, Mn, Mo, Na, Ni, Sb, Sn, Ta, Th, U, W, Zn, rare earth metals	75
Tl	Precipitation of the matrix (TlI)	Al, Bi, Cd, Ce, Co, Cu, Fe, Ga, In, La, Mg, Mn, Ni, Pb, Sn, Th, U, Zn	76
	Extraction of the matrix (TlBr; S: 2-chloroethyl ether)	Ag, Al, Ba, Bi, Ca, Cd, Co, Cr, Cu, Fe, Ga, In, Mg, Mn, Ni, Pb, Pt, Te, Zn	77
U	Extraction of the matrix (S: tributyl phosphate)	Ag, Al, Cd, Co, Cr, Cu, Fe, Ga, In, Mn, Mo, Ni, Pb, Sn, Ti, V	78
	Chromatography	rare earth metals	79
	Ion exchange	Al, Cd, Co, Cu, Fe, Mn, Ni, Zn	80
V	Distillation of the matrix (VCl ₄ , VOCl ₃)	Ag, Al, B, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, Hg, Mg, Mn, Pb, Si, Th, Tl, Zr	81
	Extraction of trace elements (R: Na-diethyldithiocarbamate; S: chloroform)	Ag, Au, Bi, Cd, Co, Cu, Fe, In, Mn, Ni, Pb, Tl, Zn	47
W	Extraction of the matrix (S: benzyltrialkylammonium nitrate/trichloroethylene)	Al, As, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, Ga, In, Mg, Mn, Ni, Pb, Si, Te, Tl, Zn	82
	Ion exchange	Ag, Au, Cd, Co, Cr, Cu, Fe, Ga, Hg, In, Mo, Nb, P, Pa, Re, S, Sc, Sn, Ta, Zn, Zr, alkali metals, alkaline earth metals, rare earth metals	83
	Extraction of trace elements (R: Na-diethyldithiocarbamate; S: chloroform)	Bi, Cd, Co, Cu, Fe, Ni, Pb, Zn	84
Y	Extraction of the matrix (R: tributyl phosphate; S: chloroform)	Ag, Al, Bi, Ca, Cd, Co, Cr, Cu, Fe, Mg, Mn, Ni, Pb, Sb, Si, Sn, Ta, Ti, Zn	85
Zn	Precipitation of trace elements (C: Al(OH) ₃)	Bi, Cr, Ge, In, Mn, Pb, Sn, Te, Ti, V	86
	Ion exchange	30 elements	87
	Partial dissolution of the matrix	Ag, Au, Bi, Cd, Co, Cu, In, Ni, Pb, Pd, Sn, Tl	88
Zr	Extraction of trace elements (R: NH ₄ -tetramethylenedithiocarbamate/dithizone; S: chloroform)	Ag, As, Au, Bi, Cd, Co, Cr, Cu, Fe, Ga, Hg, In, Mn, Mo, Nb, Ni, Pb, Pd, Pt, Sb, Se, Sn, Te, Tl, U, V, Zn	89
	Distillation of the matrix (ZrCl ₄)	Ag, As, Au, Bi, Cd, Co, Cr, Cu, Fe, Ga, Hg, In, Mo, Mn, Ni, Pa, Pd, Sb, Sc, Se, Te, Tl, W, Zn, alkali metals, alkaline earth metals, rare earth metals	90

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