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Rapid Neutron Activation Analysis of Arsenic in A Wide Range of Samples by Solvent Extraction of the Iodide

A. R. Byrne and A. Vakselj

Department of Nuclear Chemistry, »J. Stefan« Institute, University of Ljubljana, 61000 Ljubljana, Slovenia, Yugoslavia

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A method is described for neutron activation analysis of arsenic in biological samples, inorganic matrices, water samples, metals and glass, based on the quantitative and selective solvent extraction of arsenic iodide into toluene. Depending on the sample, extraction is carried out from sulphuric, perchloric or hydrochloric acids containing alkali iodide. After washing the organic phase, arsenic may be either measured directly or stripped back into an aqueous medium. The 0.56 MeV γ-ray of ^{76}As is counted in a well-type 3 × 3 inch NaI(Tl) crystal and multichannel analyser. For moderate neutron doses (20 h at 2×10^{12} $cm^{-2} s^{-1}$), levels down to 0.1 ng/g may be measured. Results are presented for various samples including some international standard materials.

Conventional radiochemical separations for arsenic (as ^{76}As) after neutron irradiation rely almost exclusively on distillation of arsenic as the chloride or bromide, and/or precipitation as elemental arsenic or the trisulphide[1,2]. These procedures are time consuming where a number of determinations need to be made. The present paper describes the application of a more rapid technique for the determination of arsenic in biological samples, waters and various inorganic matrices, based on the quantitative and selective solvent extraction of arsenic tri-iodide into toluene from acid solutions containing alkali metal iodide. Depending on the demands of the sample, this solution may be either sulphuric, hydrochloric or perchloric acid; this choice of medium provides a very flexible approach. One or two washes of the organic phase then give a high degree of decontamination from any radiochemical impurities and the ^{76}As may be measured already in the organic phase or stripped into a dilute acid solution. For highest sensitivity the gamma spectrum is measured in a well-type NaI(Tl) crystal coupled to a multichannel analyser. A wide range of samples have been analysed using this approach with good results, at arsenic levels from ppm to below 0.1 ng/g. Apart from greater simplicity, the method also safely eliminates chemical yield determinations.

METHOD

Solvent Extraction of AsI_3

In general solvent extraction techniques have been little used for determining arsenic in radiochemistry. Extraction of As(III) from concentrated hydrochloric[3] or hydrobromic acid[4] with various solvents is possible, as is

also extraction of the diethylammonium diethyldithiocarbamate complex from mineral acid solutions[5]. However, these processes are either rather unselective or not studied for radiochemical interferences. Prestwood[6] used the chloroform extraction of arsenic tri-iodidide as a stage in a radiochemical purification of arsenic. However, Tanaka[7] first systematically studied the extraction of arsenic tri-iodide from hydrochloric, hydrobromic and sulphuric acid-iodide mixtures. A spectrophotometric method was developed for determining arsenic in steels[8]. For radiochemical work more information was needed on certain interferences, especially the behaviour of antimony which has a γ-ray peak of almost identical energy at 0.56 MeV. A rapid and selective approach for the radiochemical separation of arsenic was provided by recent work[9] on the extraction of many elements as iodides into toluene from sulphuric acid-potassium iodide media. It was previously used[10] for the simultaneous determination of arsenic and antimony in biological materials. This medium is suitable for most biological materials following wet ashing in sulphuric-nitric acid or hydrogen peroxide—sulphuric acid mixtures. Table I shows the extraction behaviour of As and Sb for two of the most useful iodide concentrations as a function of acid concentration. Since the sulphuric acid system has been most extensively studied, and for over 20 elements (in addition to the elements described[9] also Co, Cs, Os and Pd do not extract), this will form the system of choice, or be used for the 'clean-up', washes after the first extraction.

TABLE I

Extraction of As *and* Sb *from mineral acid-iodide solutions with an equal volume of toluene at* 25 °C

Aqueous phase	% As extracted	% Sb extracted
$[H_2SO_4]$ + [KI]		
1.5 M + 0.05 M	0.0	61.3
3.0 M + 0.05 M	0.2	99.4
4.5 M + 0.05 M	85.7	99.6
6.0 M + 0.05 M	99.8	99.7
1.5 M + 1.0 M	2.1	6.7
3.0 M + 1.0 M	99.0	5.1
4.5 M + 1.0 M	99.9	5.4
$[HClO_4]$ + [NaI]		
2.5 M + 0.05 M	0.2	98.9
5.0 M + 0.05 M	96.1	99.6
7.0 M + 0.05 M	99.8	99.7
2.5 M + 1.0 M	67.5	7.8
5.0 M + 1.0 M	99.9	7.1
7.0 M + 1.0 M	99.9	11.3
[HCl] + [KI]		
3.1 M + 1.0 M	75.3	3.0
5.2 M + 1.0 M	99.7	—
5.7—6.2 M + 0.5 M	99.8	4.6
9.3 M + 0.1 M	99.7	1.4
9.3 M + 0.2 M	99.9	3.9
9.3 M + 0.0 M	92.3	—

Table I also summarizes the extraction behaviour of arsenic and antimony that we found from the HCl—KI system with toluene. (The use of the HCl—KI system to separate arsenic and tin was previously reported by Tanaka[11], and has been used in the radiochemical purification of short-lived tin isotopes[12]). However, a preliminary oxidation step may be necessary to ensure complete dissolution of arsenic from samples into hydrochloric acid. This extraction of arsenic is also apparently quite selective, though the behaviour of many other elements remains to be investigated.

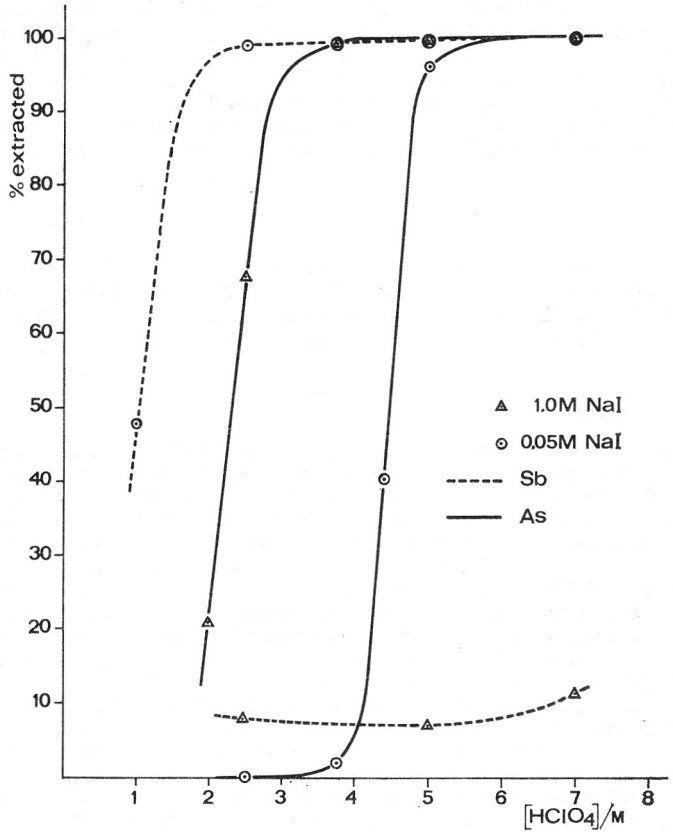

Fig. 1: Extraction of As and Sb from $HClO_4$—NaI solutions with toluene for 2 iodide concentrations.

Fig. 1 and partly, Table I show the extraction behaviour of arsenic and antimony we found with toluene from $HClO_4$ — NaI media. Their behaviour is very similar to that in the H_2SO_4 — KI system, except that antimony is a little more extractable from 1.0 M iodide. Thus $HClO_4$ — HNO_3 is another useful mixture for the dissolution of samples, especially for matrices which would produce insoluble sulphates.

Because the extraction coefficients are high, and the extraction selective, iodide extraction seems to provide an attractive simple method for the radiochemical separation of arsenic. Because of the choice of acid media available,

it can be applied to a wide variety of sample matrices. Toluene is a favourable solvent because of its low mutual miscibility with water and ease of phase separation. In all these dissolution procedures, excess of oxidising agent must of course be destroyed before the addition of iodide to prevent oxidation to iodine. The extraction coefficients for arsenic were independent of arsenic concentration in the range studied from 10^{-2} to 10^{-5} M.

EXPERIMENTAL

Standards

A stock solution of 1.0 mg/ml As was prepared by dissolving 330 mg As_2O_3 in 5 ml conc. HNO_3 and diluting to 250 ml. This solution was diluted by volume at intervals to provide a working solution of 10 μg As/ml in 0.2—0.3 M HNO_3. About 100 mg of this solution was weighed into polythene tubing of 3 mm inner diameter, sealed and irradiated alongside the sample. After irradiation a smaller amount (2—3 drops) of the standard was weighed into a 5 or 10 ml vial and diluted to the same volume as the final sample solution and in a solution of the same composition.

Irradiations

Samples were sealed in polythene ampoules (quartz for water samples) and irradiated in the rotating rack facility of the Institute's TRIGA Mark II reactor at a neutron flux of 2×10^{12} $s^{-1}\,cm^{-2}$, generally overnight (15—20 hours), or for shorter periods for samples with ppm levels of arsenic.

PROCEDURE

Biological materials

The irradiated sample (usually 0.1—1 g) is transferred to a long-necked 100 ml Kjeldahl flask and traces of sample washed from the ampoule with about 5 ml of concentrated nitric acid containing 1 mg of As (III) carrier. 4 ml of concentrated sulphuric acid are added to the flask and the mixture boiled. If the solution turns dark one or two more ml of HNO_3 are added, and the solution taken to SO_3 fumes. 30% H_2O_2 is added dropwise to destroy any traces of organic matter and to remove excess HNO_3. Finally the solution is diluted with 5—10 ml water and reboiled to destroy excess H_2O_2 to a final volume of 7—8 ml. This is transferred to a 50 ml separating funnel with two washings of water to a volume of 15—16 ml. 4 ml 5 M KI are added and the volume adjusted to a 20 ml mark. 6 ml of toluene are added by pipette and the funnel shaken for 2 min. The aqueous phase is discarded. The organic phase is washed twice with 10 ml of a solution 3.75 M H_2SO_4—1.0 M KI and the aqueous layers run off as completely as possible. Alternatively, the washings may be made with 10 ml of a solution 5.7 M HCl—0.5 M KI. This gives a slightly better removal of antimony traces and better KI economy: although the behaviour of other elements in the HCl medium is not known the ^{76}As γ-spectra obtained are just as pure as using the H_2SO_4 technique. This HCl—KI washing is now the recommended procedure.

Finally, arsenic is measured directly or stripped from the organic phase with 1·5 M H_2SO_4. Two 5 ml 1.5 M H_2SO_4 strips may be made, and run into a 10 ml vial so that all ^{76}As may be measured in the well crystall. Alternatively, if the organic phase is to be measured, it is convenient to use 6 ml of toluene for the extraction and take a 5 ml aliquot for measurement.

Water Samples

For most water samples (if they contain $\geqslant 0.1$ μg As per litre) direct irradiation of about 5 ml in a carefully cleaned quarz ampoule will give sufficient ^{76}As for measurement. In this case, the procedure is exactly the same as for biological samples. In sampling, if possible, the cleaned ampoules (aqua regia is effective) should be taken to the source, river, well *etc.*, and filled directly without

intermediate transfer vessels. Thus contamination or adsorption errors due to containers and storage can be completely eliminated.

For samples of very low arsenic level, in the absence of possibilities for a higher flux irradiation, a preconcentration stage prior to analysis is required. The following procedure has been tested and succesfully used.

Pre-cleaned quartz beakers of 250 ml or quartz Kjeldahl flasks of 100 ml capacity are used to take a 200 or 100 ml sample. 1—2 ml of conc. HNO_3 are added immediately on the spot and the sample is reduced in the laboratory to a few ml by gentle heating. The sample is transferred quantitatively with a few ml of double distilled water to quartz ampoules and sealed. After irradiation, the procedure is again identical. However, if the sample contains appreciable amounts of calcium, it is better to perform the post-irradiation removal of HNO_3 and extraction of the arsenic from perchloric acid medium. For the extraction the solution should be adjusted to $\geqslant 5$ M $HClO_4$—1.0 M NaI. After this first extraction the rest of the procedure is identical.

Inorganic matrices

The method has been used for samples of the NBS standard material calcite 915, the Czech* standard magnesite (Magnesit 3443/2, Košice) and standard kaolin (Karlovy Vary), some uranium ores and their sulphuric acid extracts, and soils.

For the calcite, originally the irradiated samples were simply dissolved in hydrochloric acid containing As (III) carrier, the solution adjusted to 5.7 M HCl—0.5 M KI and the arsenic extracted into toluene. A single stage washing with 3.75 M H_2SO_4—1.0 M KI then followed. However, because it was thought that dissolution and activity-carrier equilibration might be incomplete in the absence of an oxidant, another method was used in which the sample was dissolved in HNO_3, which was subsequently removed by heating with $HClO_4$. The first extraction was then made from 6 M $HClO_4$—1.0 M NaI. As shown under Results, the arsenic found was in fact the same for both approaches.

For kaolin and uranium ores, the finely powdered material was treated with a mixture of concentrated nitric and sulphuric acids, a mixture which has been recommended for the dissolution of both arsenic and uranium ores by Doležal *et al.*[13]. Extraction was then made from sulphuric acid as above.

The method is ideal for technical extracts of uranium ores in sulphuric acid, which were analysed for the pilot production of uranium from Slovenian ores since high arsenic levels interfere in the subsequent uranium separation.

Soils were treated in the same manner as the ores.

Metals and alloys

As yet, only pure zinc metal has been analysed by the extraction technique, but application of the method to many other samples should be without problems. Tanaka has described a procedure[8] for the spectrophotometric determination of arsenic in iron and steels based on AsI_3 extraction from HCl—KI. The main difference from other samples is that after dissolution in HNO_3 or HNO_3—HCl mixtures with arsenic carrier and boiling down in $HClO_4$ solution, oxidizing ions such as Fe^{3+} must be removed. This was done by reduction[8] with a freshly prepared solution of Cr (II) in HCl. However, hypophosphite ion is also effective, as we showed that in the cold no reduction of As (III) to elemental arsenic occurs in HCl and H_2SO_4—KI media.

For samples containing a high proportion of an element which produces an insoluble iodide, *e. g.* copper, the procedure will not be directly applicable. However, in the presence of mg amounts of arsenic carrier, when co-adsorption should be negligible, the extraction will be workable provided the insoluble precipitate does not disperse in or around the organic phase.

For zinc samples, the metal was dissolved in the cold in a 1 : 1 HNO_3—H_2O mixture. Sulphuric acid was then added and taken to fumes, and the extraction made as usual from 3.75 M H_2SO_4—1.0 M KI. It is important to dissolve the

* Available from the Institute of Mineral Raw Materials, Kutna Hora, Czechoslovakia.

sample in nitric acid, and avoid HCl or H_2SO_4, as otherwise arsenic can be lost through the formation of volatile arsine. Surface contamination was removed by etching after irradiation where possible.

Glass

Since losses of arsenic by volatilization are a serious problem in procedures for glass dissolution involving hydrofluoric acid, the dissolution procedure, an adaptation of that of Paul[14], involved shaking the sample in a cold mixture of HF—HCl—ICl inside a screw topped polythene bottle for 10—30 hours. An oxidant like ICl is necessary to ensure that all valency states of arsenic are converted to As (V).

After irradiation, the sample was washed in 1 : 1 nitric acid, and water. It was dropped into a polythene bottle containing 20 ml HCl (11 M), 10 ml 40% HF, 2 ml ICl (10% solution in conc. HCl) and 5 mg arsenic carrier. After dissolution, the resulting solution was transferred to a 250 ml beaker and diluted to 150 ml with a 6 M HCl solution containing 2.5% boric acid (to protect the glass by complexing fluoride ions). Arsenic was then precipitated by reduction with ammonium hypophosphite at 90 °C.

After filtration, the arsenic precipitate containing also other reducible elements such as gold, tellurium *etc.*, is dissolved in nitric and sulphuric acids and extracted as AsI_3, as described above for biological materials. The chemical yield, as determined by titration, is about 95%.

Direct extraction of arsenic after dissolution of the glass is possible, but with lower chemical yield.

Counting

Throughout this work a 3 × 3 inch NaI(Tl) well-type crystal coupled to a 256 or 400 channel analyser was used for measurement of the γ-spectrum of ^{76}As. This provides maximum sensitivity. The area of the 0.56 MeV peak was evaluated for calculation of results. The standard was made up as described above.

RESULTS

In practice, the method has performed extremely well, giving radiochemically clean ^{76}As γ-spectra. The method is simple, and by using an array of Kjeldahl flasks on hot plates in a fume hood, many samples may be treated simultaneously. The extraction stages take only a few minutes.

Losses of arsenic

Since the method relies on a quantitative yield of arsenic, the question arises of losses during the dissolution and extraction steps. As regards the latter, since distribution coefficients for arsenic under the conditions used are over 500 for all the acid media used, the extraction stages are free from any losses. Some biological materials contain sufficient calcium to form a sulphate precipitate in the separating funnel prior to extraction. Tracer experiments have shown that this does not adsorb any ^{76}As in the presence of mg amounts of arsenic carrier. However, this problem can be avoided by the use of perchloric acid for the destruction and extraction stages with such samples. The wet ashing or heating of acid solution containing arsenic has been discussed by Gorsuch[15]. He concludes that provided oxidizing conditions are maintained, no losses occur. This is in accordance with the well known properties of arsenic, *i. e.* that As(V) is not in general volatile or distillable, but that As(III) is, especially in the presence of halide ions.

In the present work all the procedures described above have been tested by tracer experiments for recovery of arsenic.

The results confirmed the conclusions of Gorsuch since quantitative recoveries were always obtained.

Interferences

The only interference ever observed in the γ-spectra were for a few biological samples containing elevated levels of selenium (*ca.* 5—10 ppm) when the organic phase was measured directly. Since Se(IV) is reduced by hydriodic acid to amorphous elemental selenium, which is soluble in toluene, the peaks of ^{75}Se appeared. However, appart from the rarity of such samples, this is completely unimportant since the gamma rays of ^{75}Se all lie well below the 0.56 MeV of ^{75}As, and more practically, on stripping arsenic into a dilute acid solution, selenium remains behind in the toluene phase. (This could form the basis of a technique for selenium analysis.)

In the present work, no difficulties have been encountered with ^{82}Br in the final gamma spectra, even when the organic phase was measured directly.

Sensitivity

Under the irradiation and counting conditions described, the specific count rate obtained for the 0.56 MeV peak of ^{76}As is about 100 min^{-1}/ng of arsenic one day after the end of the irradiation. Using a 3σ background criterion, this represents a detection limit of about 0.2 ng As.

Analytical Results

Biological samples. — The method is routinely used for the analysis of arsenic in our ecological programme and many samples of human and animal organs, plants, fungi, etc. have been analyzed.

Some analyses of standard materials have been made both to check the method and as part of cooperative international projects to establish values. These results are shown in Table II. Agreement with established values is good. The reproducibility and radiochemical purity are excellent. With Bovine Liver, for example, samples were wet ashed and the organic phase (5ml from 6) measured directly after two clean-up scrubs with HCl—KI solution within 6 hours of the end of an overnight irradiation, yet no impurities at all could be detected in the γ-spectrum.

Water samples. — The reactor site tap water, which is pumped natural ground water, was analyzed both by the direct and the preconcentration methods, and also some mineral water from the source at Radenci. The results are shown in Table III. For the tap water samples, standard addition experiments confirmed the values. Samples were not stored but taken direct from the tap over a period of about a month.

For the preconcentration technique, the blank values due to added nitric acid have been substracted but they were minimal, as a content of only 0.5 ng/g was found. The ^{76}As γ-spectrum from 200 ml of tap water by the preconcentration technique is shown in Fig. 2.

Inorganic matrices. — Results are shown in Table IV for the standard calcite and magnesite. For the ore samples, in which arsenic levels were from 0.1% to 0.01%, it was possible to make nondestructive comparative analyses by Ge(Li) detector γ-spectrometry. Results showed excellent agreement between the two methods, provided the material was finely ground so that

TABLE II

As *content of various standard biological materials*

Sample:	As found (ppm)	*n**	Certified or Average value (ppm)
NBS Orchard Leaves SRM 1571	9.85 ± 0.54	8	11 ± 2[16]
NBS Bovine Liver SRM 1577	0.054 ± 0.0011	4	(0.055)
NBS Tomato Leaves SRM 1573	0.241 ± 0.016	7	—
IAEA Mashed Potato Run V-4 (1972)	0.0241 ± 0.0007	4	—
IAEA Wheat Flour Run V-2/1 (1972)	0.0185 ± 0.0012	5	—
Bowen's Kale	0.1175 ± 0.002 0.118 ± 0.004**	6 6	0.141[17] (range 0.11 to 0.22)

* *n* is the number of determinations; the variation is one standard deviation.

** Previously reported[10].

TABLE III

As *content of water samples by direct and pre-concentration methods*

Sample:	Pre-concentration [As]/μg dm^{-3}	Direct [As]/μg dm^{-3}
Ground Water (Reactor) + standard addition	0.16, 0.14, 0.14, 0.14. 0.15, 0.14	0.15, 0.17
Radenska Water		
Source No. 7	3.22, 3.78, 3.24	3.24
Source No. 8	0.074	0.088

TABLE IV

As *found in standard calcite and magnesite*

Sample	[As] found/ng g^{-1}	*n*
NBS Calcite $CaCO_3$ — 915	9.37 ± 0.45* 9.7 ± 1.0**	7 3
Czech Magnesite Košice 3443/2	838 ± 33*	3

* samples dissolved in HCl and extracted from HCl—KI.

** Samples dissolved in HNO_3 and extracted from $HClO_4$—NaI.

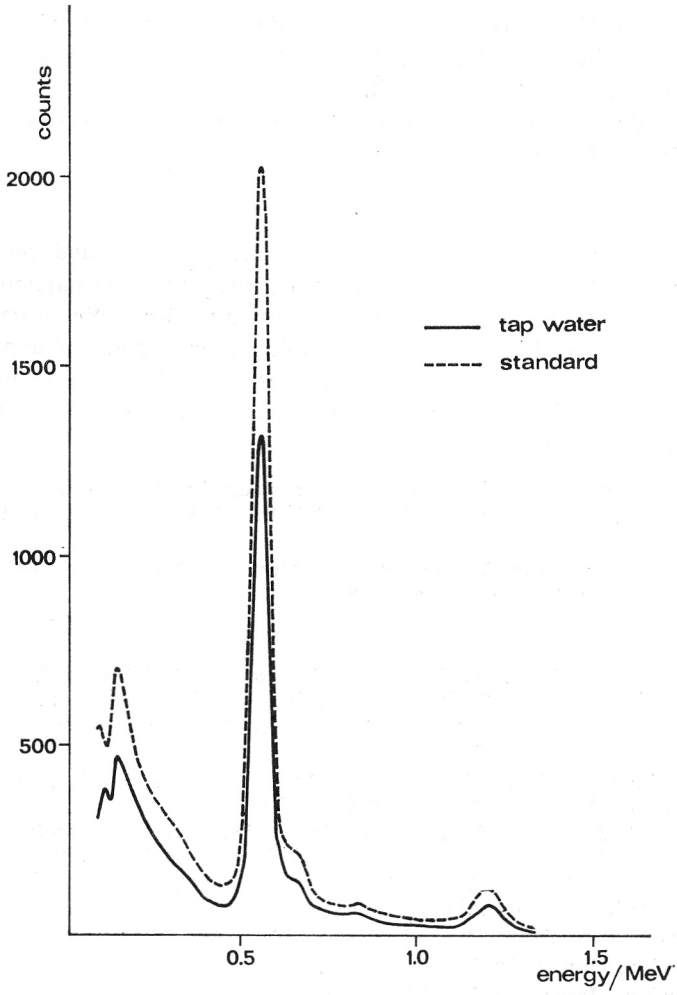

Fig. 2: γ-spectrum of ^{76}As from 200 ml tap water sample (pre-concentration method) and standard. Counting period 10 min for sample, 1 min for standard.

TABLE V

As *found in pure* Zn *samples*

Sample:	[As] found/ng g^{-1}	n
Koch-Light, Zinc Sheet 99.9998%, 8836 L	6.51 ± 0.37	4
Mallinckrodt, Zinc Sticks Analytical Reagent, 8721, Control XNJ-1	4.6 ± 0.6	4
Kemika, Zagreb, Zinc powder p. a., 710649	410 ± 11	5

acid attack effectively removed all arsenic from the sample. The values obtained on sulphuric acid-ore extracts were compared to a spectrophotometric method and agreed very well.

Zinc samples. — The results of the analyses of pure zinc samples are shown in Table V.

Conclusions

From the results presented, it is clear that the method possesses good characteristics with respect to accuracy and precision. It is particularly useful for biological materials in environmental research. Its main advantages over other activation procedures lie in the simplicity and speed of analysis, which can be performed by technical personnel. Because of the range of mineral acids from which arsenic triiodide may be extracted, the method is applicable to virtually every type of sample.

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IZVLEČEK

Hitra določitev arzena z nevtronsko aktivacijsko analizo v različnih vzorcih z uporabo solventne ekstrakcije njegovega jodida

A. R. Byrne in A. Vakselj

Opisana je metoda za določitev arzena z nevtronsko aktivacijsko analizo v bioloških vzorcih, anorganskih materialih, vzorcih vode, kovinah in steklu. Metoda sloni na kvantitativni in selektivni ekstrakciji arzenovega (III) jodida v toluen. V odvisnosti od vzorca ekstrahiramo iz raztopin žveplove, perklorove ali solne kisline z raztopljenim alkalijskim jodidom.

Po izpiranju organske faze lahko arzen merimo direktno ali pa ga prej ekstrahiramo nazaj v vodno fazo. γ-žarke ^{76}As z energijo 0.56 MeV merimo v 75 × 75 mm NaJ(Tl) kristalu z utorom (well type), ki je povezan z večkanalnim analizatorjem. Po 20 urah obsevanja v fluksu termalnih nevtronov 2×10^{12} $cm^{-2} s^{-1}$ lahko še ugotovimo koncentracije do 0,1 ng/g. Podani so rezultati analiz različnih vzorcev, in nekaterih mednarodnih standardnih materialov.

ODSEK ZA NUKLEARNO KEMIJO,
INSTITUT »JOŽEF STEFAN«,
LJUBLJANA

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