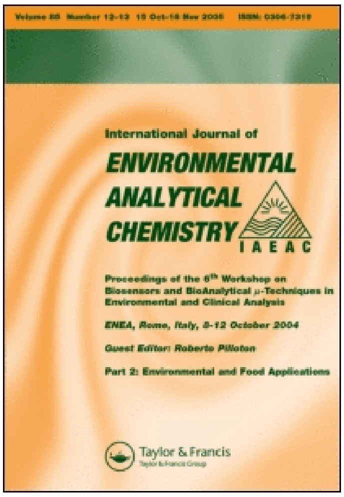

International Journal of Environmental Analytical Chemistry
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713640455

Identification and Behaviour of Trace Inorganic Elements in an Urban Sewage Treatment Plant by Monostandard Activation Analysis

J. I. Kim[a]; I. Fiedler[a]; H. J. Born[a]; D. Lux[a]
[a] Institut für Radiochemie, TU München, Garching, F. R. Germany

To cite this Article Kim, J. I. , Fiedler, I. , Born, H. J. and Lux, D.(1981) 'Identification and Behaviour of Trace Inorganic Elements in an Urban Sewage Treatment Plant by Monostandard Activation Analysis', International Journal of Environmental Analytical Chemistry, 10: 2, 135 — 148

To link to this Article: DOI: 10.1080/03067318108071538

URL: http://dx.doi.org/10.1080/03067318108071538

Intern. J. Environ. Anal. Chem., 1981, Vol. 10, pp. 135 148
0306-7319/81/1002-0135 $06.50/0

Printed in Great Britain

Identification and Behaviour of Trace Inorganic Elements in an Urban Sewage Treatment Plant by Monostandard Activation Analysis

J. I. KIM, I. FIEDLER, H.-J. BORN and D. LUX
Institut für Radiochemie, TU München, 8046 Garching, F. R. Germany

(*Received September* 5, 1980)

An analytical method is described for the multielement determination of water samples using the monostandard activation analysis. The method is applied to investigate the behaviour of trace inorganics in an urban sewage treatment plant. The concentration of trace elements in sewage is assessed by an appropriate sampling of effluent: the sewage collected either from the inflow or the outflow of the plant is centrifuged and filtered with a 0.45-μm micropore filter in order to distinguish sewage sludges, suspended solids and effluents. Based on the analytical results of suspended solids and effluents, the behaviour of trace elements in the cleaning process is interpreted with respect to their individual chemical properties.

INTRODUCTION

The neutron activation analysis (NAA) and inductively coupled plasma atomic emission spectrometry (ICP-AES) are receiving extensive attention for multielement analysis of water samples. The latter method is fast and requires a relatively small amount of sample but its sensitivity (1–3) is still inferior to the NAA for many elements. On the other hand, the NAA does not provide satisfactory sensitivity for Pb, Cd and some others. A preconcentration technique helps the ICP-AES to improve the sensitivity (4–5) but the procedure is negated by the possibility of contamination and it is not always quantitative for many elements (7–11). The NAA involves difficulties in irradiation and counting of voluminous water samples, and also in handling of standards in diverse analytical conditions (8–16).

This paper deals first of all the problems associated with the analysis of water samples by NAA. The method proposed in this work provides the possibility of activating a large volume of water sample for any desired length of time and of direct gamma spectrometric assay in a stable geometry. Such a possibility without chemical elaboration, therefore, increases sensitivity and accuracy. Furthermore, the use of standards is eliminated by introducing the monostandard method (MS-NAA), which replaces the standards by nuclear data. The method facilitates attaining the high precision of analysis without sacrificing the accuracy.

The method is applied for the investigation of trace inorganic constituents in an urban sewage treatment plant by identifying and studying the behaviour of elements in the course of cleaning processes. Such a knowledge may eventually be beneficial for the prevention of the elements of ecological importance, leaking out in an elevated level to the surface water. For obvious reasons, this kind of investigation demands an analytical method which offers the capability of multielement determination of water samples with appreciable precision and accuracy. Discussion is extended to the analytical results which yield implicit insight into the behaviour of elements in effluents as well as in suspended material in the sewage treatment plant.

Monostandard neutron activation analysis(MS-NAA)

The monostandard method (19–21) differs from the relative method for its treatment of standards: it simplifies preparation, activation and measurement of individual standards through replacing them by the nuclear data. For this reason the method is advantageous over the relative method for multielement analyses of voluminous water samples, because it precludes the problems involved in preparation of multielement standards in a condition similar to water samples.

Method

With a known concentration m^* of the monostandard element, the determination of a photopeak counting ratio A_i/A^* for a nuclide of interest i in the sample and a monostandard nuclide (indicated with *) irradiated simultaneously leads to the evaluation of the element concentration m_i by the relation:

$$m_i = m^* \frac{A_i}{A^*} \left(\frac{R_s R_d}{C_1 C_2 C_3 C_4} \right) \tag{1}$$

where

$$C_1 = \frac{f_i M^*}{f^* M}, \quad C_2 = \frac{b_i}{b^*}, \quad C_3 = \frac{\varepsilon_i}{\varepsilon^*}, \quad C_4 = \frac{\hat{\sigma}_i}{\hat{\sigma}^*}$$

$$R_s = (1 - e^{-\lambda^* t_1})/(1 - e^{-\lambda_i t_1}), \quad R_d = e^{-\lambda^* t_2}/\varepsilon^{-\lambda_i t_2}.$$

$\hat{\sigma}$—effective activation cross section derived from the reaction rate, $R = nv_0\hat{\sigma}$ (n:total neutron density; $v_0 = 2200\,\mathrm{m/sec}$);

ε – detection efficiency of a detector for a particular γ-ray energy;

b—absolute γ-intensity from the total desintegration corrected for the electron conversion rate;

f—fractional isotopic abundance of the nuclide;

M—atomic weight of the nuclide per mole;

λ—decay constant:

t_1—irradiation time;

t_2—decay time.

Any element can be used as a monostandard if its nuclear data relevant to activation and gamma spectrometry are known with good accuracy. The commercially available Au-Al alloy wire (or Co-Al alloy wire) is a convenient candidate. Use of two nuclides with different nuclear properties facilitates the evaluation of effective cross sections of all nuclides involved in activation for a given irradiation condition (21). The description of MS-NAA is given elsewhere in detail (19–21), including a critical evaluation of input constants required for applying Eq. 1.

Accuracy of the method

The errors of input nuclear data are directly transferred to the analytical results for applying MS-NAA and hence the accuracy of the method is primarily dependent on the uncertainties associated with these data. A critical evaluation of the input data is prerequisite to the use of MS-NAA (19). With a continuous improvement of instrumentations, the nuclear data relevant to MS-NAA have been determined precisely. As a result, the monostandard method can provide the accuracy of analysis comparable with the relative method. Since the MS-NAA relies on the nuclear constants, the precision of analytical results from the method is often superior to that from the relative method.

The accuracy of the monostandard method is demonstrated by analysing the certified reference material SOIL-5 from the IAEA. This material is reported to be analysed by 60 laboratories using over 8 analytical methods. The results obtained by MS-NAA from this laboratory are compared with the certified values in Table I. The values from this work are determined on the basis of Eq. 1. with the nuclear data compiled in the previous work (19). Au and Co are used as standards. A whole analysis is made nondestructively by irradiating samples packed in quartz ampoules with Al-Au and Al-Co alloy wires in a rotating-disk facility in the research reactor Munich(FRM) (20) and by measuring in gamma spectrometer with a Ge(Li)-detector.

TABLE I
Comparison of analytical data from the monostandard method with the recommended values in the IAEA certified reference material SOIL-5

Element	Radionuclide	γ-energy (keV)	MS-NAA (ppm)	Recommended value(ppm)	RD† (%)
As	^{76}As	559.1	93.5±1.1	93.9±7.5	−0.45±8.53
		657	95.0±1.4		1.17±8.12
Ba	^{131}Ba	373.2	559±30	562±53	−0.53±10.75
		496.3	423±4		−24.73±9.77
Br	^{82}Br	554.3	6.2±0.1	5.4±1.0	14.81±18.81
		776.5	5.6±0.1		3.70±18.60
Ce	^{141}Ce	145.5	62.0±3.4	59.7±3.0	3.82±7.53
Cr	^{51}Cr	320.1	29.2±0.9	28.9±2.8	1.04±10.20
Cs	^{134}Cs	795.8	51.7±3.0	56.7±3.3	−8.82±7.88
		1365	60.5±1.6		6.70±6.48
Co	^{60}Co	1173.2	14.2±0.2	14.8±0.8	−4.05±5.57
		1332.5	14.4±0.3		−2.70±5.77
Eu	^{152}Eu	122	1.23±0.14	1.18±0.08	4.24±13.68
		344.3	1.21±0.05		2.54±7.99
		1407.9	1.21±0.09		2.54±10.20
Fe	^{59}Fe	1099.3	45300±556	44500±1900	1.80±4.45
		1291.9	45400±730		2.02±4.57
Hf	^{181}Hf	133.0	6.5±0.2	6.30±0.30	3.17±5.71
		482.0	6.8±0.3		7.94±6.75
La	^{140}La	487.0	27.1±2.0	28.1±1.5	−3.56±8.90
		815.8	28.2±0.8		0.36±6.12
		1596.2	24.1±0.4		−14.23±5.57
Na	^{24}Na	1368.5	17970±530	19200±1100	−6.41±6.13
Rb	^{86}Rb	1077	134.5±7.6	138.0±7.4	−2.89±8.76
Sc	^{45}Sc	889.4	14.8±0.3	14.8±0.7	0±4.73
		1120.5	15.8±0.2		+6.76±4.93
Sb	^{124}Sb	602.7	14.9±0.1	14.3±2.2	4.20±15.43
		1691	14.3±0.6		0±15.38
	^{122}Sb	563.9	13.5±0.8		−5.60±16.41
Sm	^{153}Sm	103.2	5.43±0.04	5.42±0.39	0.18±0.71
Ta	^{182}Ta	1189.9 1221.3	0.61±0.04	0.764±0.056	−19.74±9.17

†See Eq. 2.

The analytical results shown in Table I are evaluated by choosing, whenever possible, more than one gamma energy of a nuclide with different intensities and counting efficiencies. The relative differences given in per cent in the last column in this table are calculated by the following relation:

$$\mathrm{RD}(\%)=\left(\frac{\text{MS-NAA value}-\text{certified value}}{\text{certified value}}\right)\times 100 \qquad (2)$$

The corresponding relative error expressed by RD(%) is based on the relative standard deviations of the two values under comparison. The RD values for all elements are within the relative error ranges, except one of Ba, Hf, La, Na and Sc values; Cs and Ta values. The significant RD values are observed only for one of Ba and La values and Ta value. As a whole, the analytical results from MS-NAA agree satisfactorily with the certified values and hence the results in Table I verify the accuracy of MS-NAA.

EXPERIMENTAL

Sampling

One of the sewage treatment plants, from which the present samples originate, consists of two reservoirs for the sedimentation of sludges and a biological cleaning between them. Analytical effluent samples are taken from the inflow to the first reservoir and the outflow from the second reservoir which discharges the cleaned effluent to the river. A 5-l solution from each sampling position is collected at the same time by a peristaltic pump for 24 h in a polyethylene flask.

Treatment of samples

The collected effluents are stored in a refrigerator at 4°C before and during their treatment to prepare the samples for analysis. The effluent is centrifuged portionwise at 45,000 g by an ultracentrifuge in order to sediment suspended coarse materials, which are then discarded. Since the object of the present work is to investigate trace inorganics dissolved stably in the effluent, the suspended coarse material is for the moment not taken into consideration. On the other hand, the suspended coarse material in the inflow is difficult to define, because it includes a large part of irregular solid wastes which are assumed to undergo, first, sedimentation to the sewage sludge and second, biological deterioration in the coarse of cleaning processes. The suspended coarse material in the outflow consists of more or less fine particles originated from the floatation of the sewage sludge and undergoes easily sedimentation. It is the reason why our present work is confined to the study of trace inorganics which remain stable in the effluents only. The solution resulted from centrifugation is introduced into a high pressure (9 atm) filter system (17) with a 0.45-μm micropore filter. The filtrate is acidified to pH2 by adding suprapure 14 M HNO_3 and stored in a glass bottle in a refrigerator, whereas fine suspended materials collected on a micropore filter are dried in an oven at 60°C overnight, weighed and stored in a dessicator for subsequent analysis.

Irradiation of liquid sample

250 ml of the effluent sample prepared as stated above are irradiated in the FRM (Forschungsreaktor München) in a quartz volumetric flask near the core as illustrated in Fig. 1. The air in the unfilled bottle neck covered by a quartz test tube holds back the pressure of the reacotr water, separating it from the sample solution, while allowing gases produced by radiolysis to evolve out. The gas produced by radiolysis in the present irradiation condition is measured to be 4.5 ml/min per liter distilled water; the rate changes with the concentration of impurities in water. This simple facility (18) enables the irradiation of a large volume of liquid as long as

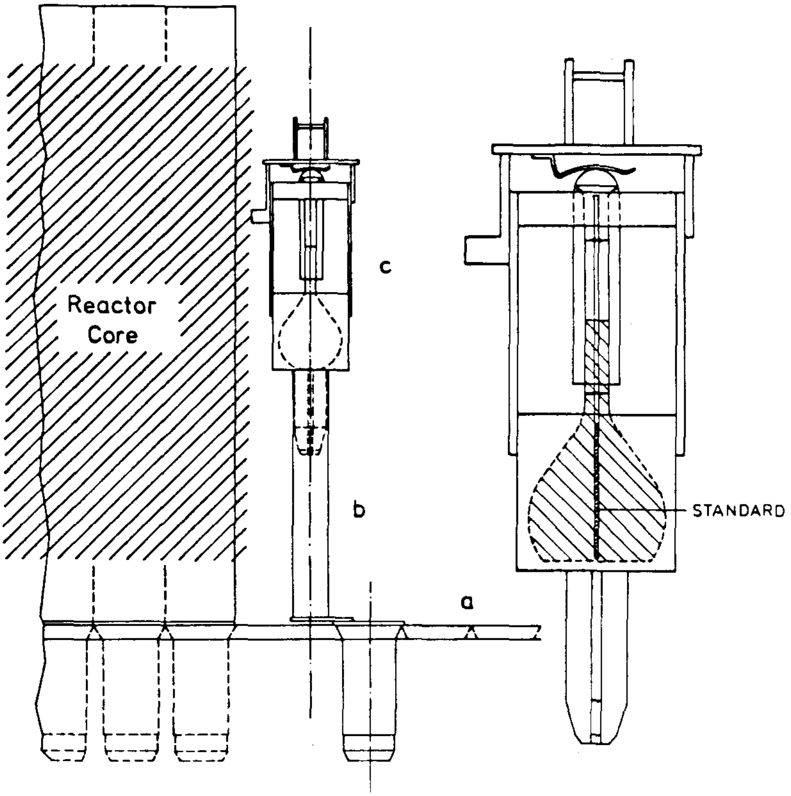

FIGURE 1 An irradiation of water sample in a swimming pool reactor. The irradiation cage(c) with a volumetric flask(250 ml) covered by a test tube, both made of quartz, is placed near the reactor core at a cage holder(b) fixed on the grid-plate(a). The air between the bottle neck and test tube prevents contact of a sample solution with the reactor water but enables to evacuate the gas produced in the bottle by radiolysis. The standard consisted of Al-Au- and Al-Co-alloy wires in a quartz capillar is placed in the middle of the sample solution.

desired for analysis. For the present work, the irradiation time is limited to 20 h at the flux ca. 10^{13} n/sec cm^2. Al-Au and Al-Co alloy wires of 0.5 mm diameter (from Degussa Co.) are irradiated together with samples as standards, which contain Au and Co, 0.1274 ± 0.0004 and 0.0925 ± 0.0008 wt %, respectively. The relative standard deviations of these data prove the homogenity of the wires as well. Chips of wires are attached to the surface of the bottle in hexagonal directions of regular distance by wrapping the bottle with a thin sheet of Al foil. An improvement is made for the irradiation of the standard wires directly in the middle of the solution. The 8-cm Au and Co alloy wires are inserted in a quartz capillar with 2 mm of inside diameter, which is then placed in the irradiation bottle with the help of attached glass corns, such that the capillar remains in the center of the bottle. The illustration is shown in Figure 1.

The suspended material on a micropore filter is packed in a small polyethylene bag with the standard wires and irradiated with usual precaution. A blank filter paper is also irradiated in order to correct its background activities.

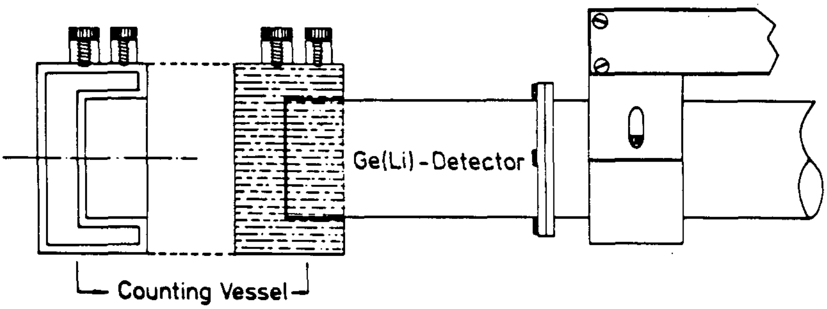

FIGURE 2 A mounting of sample for gamma spectrometry. The counting vessel is made of plexy glass (250 ml). The standard, prepared by dissolving a ctivated Al-Au- and Al-Co-alloy wire in HCl, is mounted similarly.

Gamma spectrometry

After irradiation, the sample solution is cooled for 2–7 days depending on its activity intensity and transferred into a counting vessel for gamma spectrometry . The standard wires are dissolved in 6 M HCl and diluted to a 250 ml in a counting vessel just the same geometry as the sample mounting.

The counting vessel is made of plexy glass and mounted to the position at a Ge(Li) detector as illustrated in Figure 2. Such a mounting condition ensures a constant geometry for both sample and standard solutions. The spectrometer consists of a ORTEC Ge(Li) detector, with 21 % efficiency relative to a 3 × 3' Na(Tl) detector, connected to a preamplifier, a ORTEC amplifier with pile-up restorer and a ND-4420 analyser coupled to a computer. The sample and standards are measured repeatedly for 3 hours each. The detector calibration is made prior to experiment in the same geometry as the samples are measured.

Evaluation of the results

Evaluation of the results is carried out in accordance with Eq. 1 as the procedure described in the previous papers (19–21). The Au and Co standards enable the monitoring of the flux index at the irradiation condition (21) and provide also the verification of analyses by comparison of the results calculated with respect to either standard. The nuclear data attendant to the method are taken from the previous compilation (19).

RESULTS AND DISCUSSION

Precision of analysis and stability of elements

Samples of the outflow(SOF) are divided in 6 batches, irradiated separately and measured in different elapsed times during 3 months. The evaluation is made for each batch of SOF separately. The results are illustrated in Figure 3 in order to demonstrate the precision of analysis as well as the stability of the analysed elements in the prepared SOF (*cf.* above). The relative deviations of each analytical result from the mean values among batches is found to be significant for Ag, Hg and less for Au, Ba, Cs and Sc, whereas the rest elements, Br, Co, Cr, Fe, Rb, Se and Zn show normal analytical deviations from their mean values. In consideration of pH of the original and prepared SOF, at pH = 7–8 and pH = 2, respectively, it is understood from chemical viewpoint that Ag and Hg remain unstable and are, therefore, determined to be inhomogenous among the batches. The precision of analysis for each batch is $\pm 6.3\,\%$ for Ag and $\pm 14.8\,\%$ for Hg, while the difference of each batchwise average from the mean value exceeds substantially the precision range. Although the inhomogenity is expected to occur also for Au due to its chemical properties, it is difficult to assess the conclusion from a small number of analyses. Considerably large scattering in the values of Ba within a batch is probably attributable to gamma spectrometric measurements of ^{131}Ba, but the reason is yet unknown. The batchwise large deviations of Cs and Sc values are considered not due to the analytical system but their inhomogenity. The analytical results of the Br, Co, Cr, Fe, Rb, Sb, Se and Zn show a homogenous distribution and also the high precision of

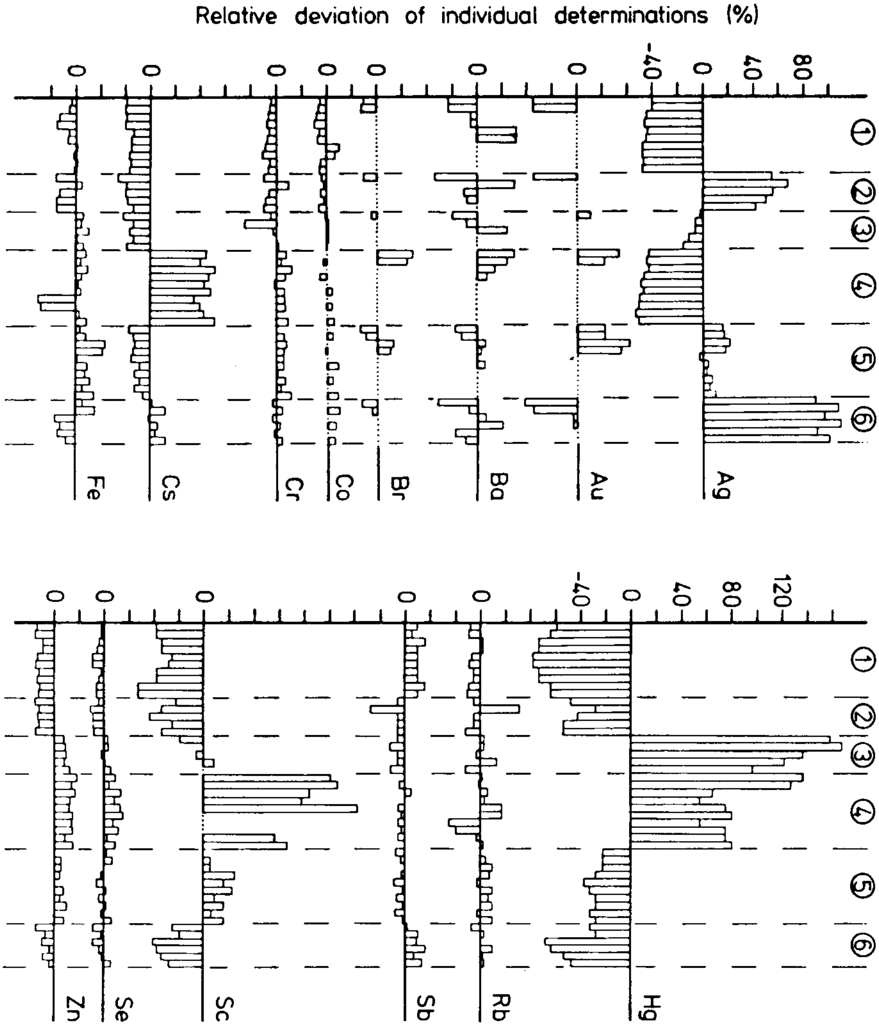

FIGURE 3 Relative deviations of individual analysis of elements from their mean values (see effluent-II of the outflow in Table II). The number above the illustration indicates different irradiations and each column represents the result from a separate gamma spectrometry, different elapsed time after irradiation.

analysis, the results of which imply relatively high stability of these elements in the prepared effluents.

Concentration of trace elements in effluents

The analytical results for the inflow and outflow effluents of a sewage treatment plant in Munich(GLM) are summarized in Table 2. Effluent-I is the solution before filtration and supposed to transport trace inorganic elements in and out of the plant. Effluent-II is the filtrate of effluent-I and considered to retain its inorganic constituents in the solution stably for a considerably long time. The suspended material is a substance resisted to centrifugation but filtered at 0.45 μm. It changes probably its chemical form depending on environmental conditions, discharging to or adsorbing inorganic elements from effluent-II. The amounts of suspended materials found through centrifugation at 45,000 g are found to be 34.14 ± 0.66 mg/l in the inflow effluent and 12.11 ± 1.73 mg/l in the outflow effluent.

Table II presents the weighed mean values of each element in the effluents and suspended materials. The concentration of an element in effluent-I is the sum of its concentrations in effluent-II and suspended material. For Ag, Au, Hg, Cs and Sc, the given standard deviations include analytical errors as well as deviations owing to inhomogenities. Since the elements Ba and Rb are not detected in the suspended materials, both inflow and outflow, their concentrations in effluent-I are assumed to be equal to those found in effluent-II and they are given in brackets. The concentration of Na in the effluents is not determined. The analytical results shown in this table indicate the location of trace elements, either in the solution or in the suspended material. Nearly whole part of Ag as well as Hg is found in the suspended material, whereas other elements are dissolved largely in the solution, except Au which is distributed in both sides. This characteristic is the same for the inflow and outflow, which can be easily apprehended by comparison of the concentration in effluent-I with that in effluent-II.

Furthermore, it is quite interesting to compare the concentration of elements in the outflow with that in the inflow in order to observe the behaviour of inorganic elements along with the cleaning process in the plant. Such comparisons are shown in Table III for the effluent-I, effluent-II and suspended material by producing ratios of concentrations in the outflow to the inflow. It is to be mentioned that the comparison involves only the trace inorganics dissolved stably in the effluent, excluding the amount contained in coarse suspended materials, which undergo sedimentation easily to the sewage sludge and are, hence, separated by centrifugation in the sampling process (*cf.* above). A ratio greater than 1 indicates that the discharging concentration of an element from the plant

TABLE II

Distribution of trace elements in effluents and suspended materials of the inflow and outflow

Element	Inflow			Outflow		
	Effluent-I[a] (ng/ml)	Effluent-II[b] (ng/ml)	Suspended mat[c] (ppm)	Effluent-I[a] (ng/ml)	Effluent-II[b] (ng/ml)	Suspended mat[c] (ppm)
Ag	9.05 ± 0.07	0.41 ± 0.02	253 ± 2.1	4.97 ± 0.18	0.78 ± 0.16	346 ± 7
Au	0.041 ± 0.001	0.021 ± 0.001	0.58 ± 0.02	0.032 ± 0.002	0.016 ± 0.002	1.40 ± 0.06
Ba	(20.5 ± 0.7)†	20.5 ± 0.7	—	(37.8 ± 3.1)	37.8 ± 3.1	—
Br	128 ± 2	127 ± 2	26.5 ± 0.7	188 ± 11	187 ± 11	52.8 ± 2.7
Co	0.83 ± 0.01	0.78 ± 0.01	1.41 ± 0.06	1.16 ± 0.03	1.09 ± 0.03	5.75 ± 0.20
Cr	12.3 ± 0.1	9.7 ± 0.1	76.5 ± 2.5	9.59 ± 0.28	8.42 ± 0.26	96.7 ± 9.6
Cs	0.345 ± 0.002	0.333 ± 0.002	0.345 ± 0.036	0.58 ± 0.06	0.57 ± 0.06	1.02 ± 0.18
Fe	187 ± 5	141 ± 4	1357 ± 63	119 ± 5	100.8 ± 4.9	1496 ± 87
Hg	0.262 ± 0.008	0.045 ± 0.006	6.37 ± 0.18	0.293 ± 0.061	0.19 ± 0.06	8.48 ± 1.08
Na	—	—	2226 ± 82	—	—	5503 ± 826
Rb	(12.6 ± 0.2)	12.6 ± 0.2	—	(13.6 ± 0.55)	13.6 ± 0.55	—
Sb	2.72 ± 0.03	2.62 ± 0.03	2.98 ± 0.13	1.94 ± 0.07	1.87 ± 0.07	5.75 ± 0.53
Sc	0.0072 ± 0.0008	0.0052 ± 0.0008	0.058 ± 0.002	0.0040 ± 0.0006	0.0032 ± 0.0006	0.065 ± 0.003
Se	0.72 ± 0.01	0.643 ± 0.014	2.11 ± 0.10	1.58 ± 0.04	1.54 ± 0.04	3.36 ± 0.27
Zn	96.3 ± 0.7	72.4 ± 0.6	701 ± 13	90.2 ± 3.7	76.2 ± 3.6	1154 ± 65

[a] Effluents after centrifugation at 45000 g, containing suspended materials.

[b] Effluents centrifuged and filtered by a 0.45-μm micropore filter.

[c] Suspended materials filtered from effluent-I by a 0.45 μm micropore filter.

†Values in ( ) are taken from effluent-II.

TABLE III
Ratios of element concentrations: outflow to inflow

	Ratio $= C_{out}/C_{inf}$		
Element	Effluent-I[a]	Effluent-II[b]	Suspended mat.[c]
Ag	0.55 ± 0.02	1.90 ± 0.40	1.37 ± 0.03
Au	0.78 ± 0.05	1.33 ± 0.18	2.40 ± 0.11
Ba	1.84 ± 0.16	1.84 ± 0.16	—
Br	1.47 ± 0.09	1.47 ± 0.09	1.99 ± 0.12
Co	1.40 ± 0.04	1.40 ± 0.04	4.07 ± 0.23
Cr	0.78 ± 0.02	0.87 ± 0.03	1.26 ± 0.13
Cs	1.68 ± 0.17	1.71 ± 0.18	2.96 ± 0.60
Fe	0.64 ± 0.03	0.71 ± 0.04	1.10 ± 0.08
Hg	1.12 ± 0.24	4.22 ± 1.34	1.33 ± 0.05
Rb	1.08 ± 0.05	1.08 ± 0.05	—
Sb	0.71 ± 0.03	0.69 ± 0.03	1.93 ± 0.20
Sc	0.56 ± 0.10	0.62 ± 0.15	1.13 ± 0.07
Se	2.14 ± 0.06	2.40 ± 0.08	1.59 ± 0.15
Zn	0.79 ± 0.03	1.05 ± 0.05	1.65 ± 0.10

[a,b,c] Explanations see Table II.

by the outflow exceeds the concentration introduced by the inflow. The concentration increase is obviously attributable to decomposition of the sewage sludge which is in contact with effluent in the plant. The increase in concentration in the effluent II of the outflow probably is also a large contribution of very fine particles in this effluent, which are leaked out the micropore filtration. It is a known fact that the effect of fine particles occurs (22). The ratio less than 1 indicates sedimentation of an element to the sewage sludge. When the sewage sludge is taken into consideration, the total concentration of individual elements is apparently reduced by the cleaning process. As far as the effluent-I is concerned, as manifested in Table III, the concentrations of some elements are clearly increased in the outflow with respect to the inflow. In addition, the results in Table III reveal the differentiated behaviour of individual elements in the coarse of the cleaning process.

Conclusion

Since analysis of urban effluents is very sensitive to its conditions of treatment and it is a system depending on so many factors which can hardly be summarized into simplified forms, there is not a general scheme to which the analytical procedure which enables us to observe the behaviour of trace inorganic constituents in the cleaning process. The high precision of analysis is prerequisite for the purpose, more than the accuracy. The monostandard method fulfils this requirement better than the conventional relative method, particularly for the problem that is subject to analysis of voluminous samples with, if possible, the least chemical elaboration, because the former method eliminates the errors of the latter arising eventually in preparation, irradiation and counting of many standards.

The analytical sample is prepared in such a way that it does not change significantly during storage and analysis and, furthermore, that the results can be compared with one another with a sufficient confidence in order to be able to follow the behaviour of individual elements in sewage treatments. The method discussed in this paper manifests its capability to eliminate many difficulties involved in the determination of trace elements in water samples by neutron activation analysis. The careful evaluation of the nuclear data attended is however the important part of the analytical procedure (19–21).

Ackowledgements

This work is financially supported by the Bundesministerium für Forschung und Technologie (BMFT). Thanks are due to Mr. G. Birebent for his technical assistance.

References

1. R. K. Skogerboe, *Toxicological and Environmental Chem. Rev.* **2,** 209 (1978)
2. R. M. Barens, *Toxicological and Environmental Chem. Rev.* **2,** 187 (1978)
3. R. K. Wingle, V. A. Fassel, R. N. Kniseley, E. De Kalb and W. J. Haas Jr., *Spectrochimica Acta,* **32B** 327 (1977)
4. W. B. Kerfood and R. L. Crawford, *ICP Inform. News Lett* **2,** 189 (1977)
5. D. S. Hackett and S. Siggia, in "environmental Analysis", Ed. G. W. Erwing, Academic Press, New York pp. 257–265 (1977)
6. R. M. Barens, R. Faria and J. S. Geuna, cited in Ref. 2
7. R. E. Van Grieken, C. M. Bresseleers and B. M. Vanderborght, *Anal. Chem.* **49** 132b (1977)
8. P. Burba, B. Gleitsmann and K. Lieser, *Z. Anal. Chem.* **289,** 28 (1978)
9. K. H. Lieser, W. Calmano, E. Heuss and V. Neitzert, *J. Radioanal. Chem.* **37,** 717(1977)
10. P. Burba and K. Lieser, *Z. Anal. Chem.* **286,** 191 (1977)
11. C. Lee, N. B. Kim, I. C. Lee and K. S. Chung, *Talanta,* **24,** 241 (1977)
12. J. I. Kim and H. Stärk, *Radiochim. Acta.* **13,** (1977) 13
13. E. Y. Uken, P. Hahn-Weinheimer and H. Stärk, *J. Radioanal. Chem.* **37,** 741 (1977)
14. G. Küpers and G. Erdmann, 8th Seminar: Aktivierungsanalyse Neuherberg, 19/20 Nov. 1979, GSF, München
15. G. Bart and H. R. von Gunten, *J. Environ. Anal. Chem.* **6,** 25 (1979)
16. H. A. Van Der Sloot, *J. Radioanal. Chem.* **37,** 727 (1977)
17. J. Müller, W. Kratzer and A. Hirner, *gwf-Wasser-Abwasser,* **117,** 220 (1976)
18. J. I. Kim, S. Stärk and I. Fiedler, *Nuclear Methods and Instruments,* **977,** 557 (1980).

19. J. I. Kim, *J. Radioanal. Chem.*, **63**, 121 (1981).
20. J. I. Kim and H. Stärk, *Proc. Activation Analysis in Geochemistry and Cosmochemistry*, NATO Advanced Study Institute, Kjeller, Norway, 7–12, Sep. 1970; Universitesforlaget, Oslo, 1971
21. J. I. Kim and H. -J. Born, *J. Radioanal. Chem.* **15,** 535 (1973)
22. U. A. Th. Brinkman, private communication