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On-line preconcentration of rhodium on an anion-exchange resin loaded with 1,5-bis(2-pyridyl)-3-sulphophenyl methylene thiocarbonohydrazide and its determination in environmental samples

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Abstract

A method for the determination of rhodium in different samples at trace levels is presented. The investigated metal is preconcentrated on a chelating resin microcolumn (1,5-bis(2-pyridyl)-3-sulphophenyl methylene thiocarbonohydrazide (PSTH) immobilized on an anion-exchange resin (Dowex 1 × 8–200)) placed in the autosampler arm. The modification of the autosampler in the tubing line and circuit allowed either the flow of the sample through the column or the operation of the autosampler in the normal mode, where microlitres of 4 M HNO_3, which acts as the elution agent, pass through the microcolumn eluting Rh(III), which is directly deposited in the graphite tube as drop of a precisely defined volume. The detection limit is 0.3 ng ml^{-1}. Linearity is maintained in the concentration range 0–50 ng ml^{-1} for rhodium, with correlation factor of 0.999 and relative standard deviation of 1.8% for 10 ng ml^{-1} of Rh. The effects of various parameters such as pH, concentration and volume of eluent, sample loading time, sample flow rate and interference of a large number of metal ions and anions on the determination of this metal was studied in detail to optimize the conditions for their determination in various samples. The method is found to be highly selective, fairly sensitive, simple, rapid and economical and may be safely applied to their determination in different complex materials such as environmental samples and catalysts.

Keywords: Rhodium; Electrothermal atomic absorption spectrometry; Microcolumn; Environmental samples

1. Introduction

Recent interest in the medical and industrial significance of platinum and to a lesser extent palladium and rhodium has been accompanied by an increasing interest in their determination at low levels.

Rhodium has been introduced into catalytic converters. Since then, approximately 73% of the world production of rhodium is consumed in the production of autocatalysts [1]. Efforts were made to assess their impact on the environment with respect to the emission of noble metals.

During the first years of automobile catalyst impact research, the focus lay on Pt as the main component of Pt/Rh-catalysts. Consequently, much effort was invested in the development of analytical methods possessing sufficient detection capacity. Pt levels of a variety of environmental matrices are already known [2]. However, corresponding Rh data are mostly missing. Analytical intercomparison projects have been performed in order to supply information on the applicability of different methods of Pt analysis [3]. This is not yet the case for Rh.

Spectrophotometric methods have been reported in the literature for the determination of rhodium [4,5] in various materials, but these methods are not sufficiently sensitive and selective. Both electrochemical techniques and atomic spectrometry have been considered for trace platinum group metals (PGM) determination. Cathodic stripping voltammetry (CSV) can be used for the determination of Pt and Rh at ultratrace levels, although this method suffers from organic matter interference [6]. Up to now, there is only one routine method available that is applied for geochemical rhodium analysis: soil samples are enriched with the aid of a nickel sulfide fire-assay (docimasy) followed by GF-AAS, ICP-MS or INAA detection [7] However, this method is not suitable

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to determine low concentrations as they are expected in biological matrices, such as plants or human body fluids. In addition, although fire-assay is used extensively, it is highly labor intensive, and one of its most serious limitations is a dependence of the quality of results on the experience of the analyst. A more robust method would clearly be advantageous. Moreover, ICP-MS detection of Rh as a monoisotopic element can suffer from possible interferences with polyatomic ions, as reviewed by León et al. [8].

The main advantage of preconcentration procedures is the possibility of determining lower analyte concentrations and avoiding matrix effects by effective separation of the analyte from interfering matrix components. In this respect, flow systems combined with liquid–liquid extraction, ion exchange, sorbent extraction [9–13] and electrochemical deposition [14,15] are of growing interest.

The determination of very low contents of the PGM in environmental samples usually suffers from inadequacies of the analytical methods, e.g. of direct GF-AAS determination [16,17]. The use of direct instrumental methods is also restricted owing to interferences caused by matrix elements and the lack of standard reference materials for the extreme trace concentration range [14]. These difficulties can only be overcome by combination of suitable decomposition, separation, preconcentration and determination steps [15–18].

Until now, ion-exchange, liquid, plane and gel-filtration chromatography, liquid–liquid extraction, coprecipitation [$Fe(OH)_3$, $La(OH)_3$, $Zr(OH)_4$, etc.], electrolytic deposition, electrophoretic separation, evaporation, flotation, freezing (cold-trap), sorption and adsorption (activated carbon, alumina, glass beads, silica gel, tungsten wire, etc.) and ultrafiltration have been reported as preconcentration (collection) and separation methods of trace metals in various samples. These preconcentration methods combined with instrumental analysis have frequently been used for the determination of trace analytes in complex matrix samples. Most of them are complicated and time-consuming. Of all preconcentration and separation procedures, sorption possesses several advantages: large preconcentration factors that can be obtained in a short time, simplicity of phase separation and suitability for automation. Column based sorption techniques are frequently applied for the separation and preconcentration in flow injection atomic spectroscopy, first of all with AAS and ICP-AES, but also with AFS and ICP-MS [19–21].

A number of methods for separation and preconcentration of PGM have been developed: sorption on metal hydroxides [22–24], chemically modified silica [25–31] and also on many types of polymeric sorbents [32–41]. Some chelating agents has been used for the PGM studies as CMDTC, formazan and dehydrodithizone.

This paper describes an automatic on-line FI-GF-AAS method for the determination of trace amounts of rhodium. A chelating ion-exchange resin was used for the separation and preconcentration of rhodium from different matrices.

2. Experimental

2.1. Instrumentation

A Perkin-Elmer Zeeman/4100 ZL atomic absorption spectrometer equipped with an AS-70 furnace autosampler was used throughout. Pyrolitic graphite coated tubes with pyrolitic graphite platforms were used in all experiments. The primary radiation source was a rhodium hollow cathode lamp operated at 15 mA; the selected wavelength was 343.5 nm with a spectral slit width of 0.2 nm.

The graphite furnace temperature program for the determination of rhodium is shown in Table 1.

The microcolumn containing the PSTH-Dowex was a glass tube (3 cm × 3 mm i.d.) packed to a height of 0.2 cm; at both ends of the microcolumn, polyethylene frits were fixed to prevent material losses. On the end of this column was placed a piece of sample capillary of the sampler arm, in imitation of the sample tip of the sampler arm (Fig. 1). Thus the sample tip of the sampler arm was replaced with this microcolumn, permitting normal working of the sampler.

A peristaltic pump, P (Gilson Minipuls 3), fitted with a vinyl pump tube (1.65 mm i.d.), was used for loading of the sample. A Rheodyne Type 50 six-port rotary valve was used as a switching valve. Transport lines were made using 0.8 mm i.d. PTFE tubing. The peristaltic pump and the selection valve were readily controlled electronically via two switches on the autosampler that were actuated when the autosampler arm was down. The process was thus fully automated without altering the software of the AA spectrometer. A schematic diagram of the circuit and peripherals is shown in the ESI [42]. For sample digestion, a microwave oven, Prolabo TX-32, was used. All glassware used was washed with 10% nitric acid for 24 h and rinsed with de-ionized water immediately before use.

2.2. Reagents

All reagents were of highest available purity and at least of analytical-reagent grade. De-ionized water (18 MΩ cm^{-1}) obtained from a Milli-Q water system (Millipore, Bedford, MA, USA) was used throughout. PSTH-Dowex was synthesized as described elsewhere [43]. A standard 1000 μg ml^{-1} Rh(III) solution (CertiPUR, Merck) was used. Standards of working strength were made by appropriate

Table 1
Graphite furnace temperature program (V_i = 40 μl)

Step	Temperature (°C)	Ramp time (s)	Hold time (s)	Argon flow rate (ml min^{-1})
1	110	1	30	250
2	140	5	30	250
3	1700	10	20	250
4	2400	0	5	0
5	2400	1	2	250

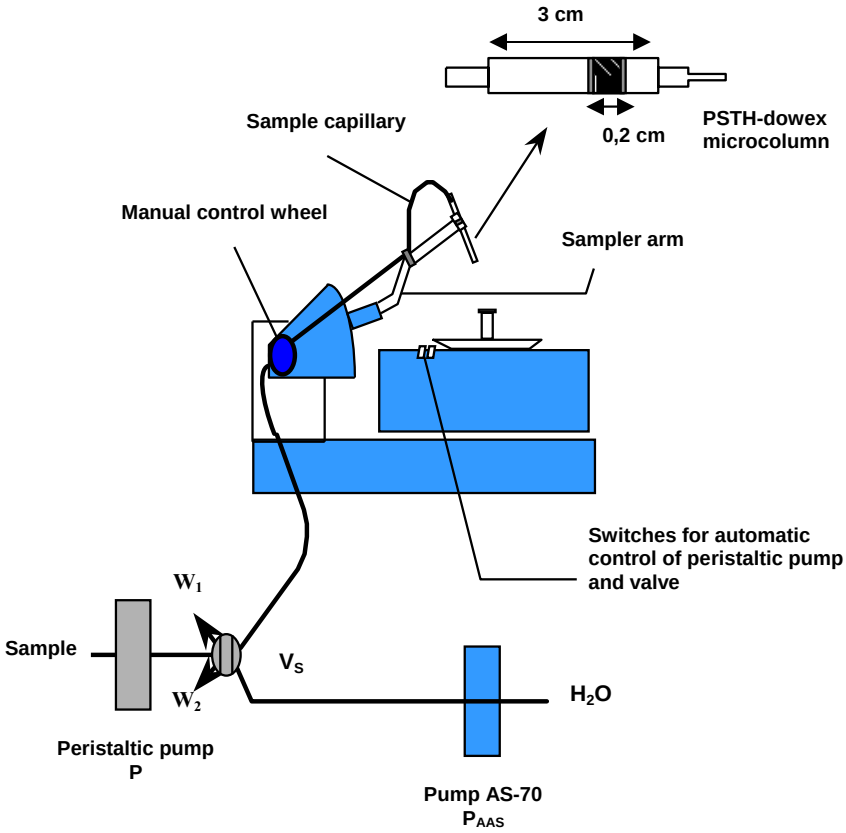

Fig. 1. Schematic diagram of FI-AAS system for the preconcentration, separation and determination of rhodium: W_1 and W_2, waste; V_S, selection valve. For further details see text.

dilution as required, immediately prior to use. A pH 3.6 buffer was prepared by mixing 25 ml of 0.2 M glicine with 2.5 ml of 0.2 M hydrochloric acid and diluting to 100 ml with de-ionized water. HNO_3 (Merck) (4 M) was used as eluent.

2.3. Sample preparation

The certified reference material (CRM) analyzed to determine the accuracy of the proposed procedure was National Institute of Standard and Technology (NIST), Standard Reference Material (SRM) 2557 catalyst. The sample was first prepared in accordance with the instruction of the analysis certificate, after which an accurately weighed amount of 0.1 g was subjected to microwave digestion. The working condition of the microwave oven is listed in Table 2. After digestion the sample was diluted to 100 ml with de-ionized water in a calibrated flask.

In view of the application of the method to the determination of rhodium in other samples, the ability to recover rhodium from samples of vegetation and soil spiked with rhodium were investigated. For this purpose, standard solutions containing different amounts of rhodium were added to 0.1–0.5 g of bignonia leaves, pinus leaves and soil, and the resulting materials were mineralized by microwave digestion as are listed in Table 2.

Table 2
Working conditions for microwave oven

Step	Reagent			Volume (ml)			Power (%)			Time (min)		
	A	B	C	A	B	C	A	B	C	A	B	C
1	HCl HNO_3	HNO_3	HCl HNO_3	15 5	10	7.5 2.5	50	15	15	5	10	10
2	–	HNO_3	–	–	10	–	30	30	–	10	22	–
3	–	HNO_3	–	–	10	–	–	30	–	–	10	–
4	–	H_2O_2	–	–	5	–	–	30	–	–	5	–

A: catalyst, SRM 2557; B: bignonia or pinus leaves; C: soil.

River and sea waters were collected in polypropylene bottles previously cleaned by soaking in 0.1 M hydrochloric acid. Samples were filtered by using a membrane of 0.45 μm pore size, acidified to 0.1% (v/v) with concentrated HNO_3 and stored frozen until analysis. The composition of the synthetic sea water was (in $g\,l^{-1}$): 27.9 of NaCl, 1.4 of KCl, 2.8 of $MgCl_2$, 0.5 of NaBr and 2.0 of $MgSO_4$, according to the specifications of R.C. Weast [44].

2.4. General procedure

The FI manifold is shown in Fig. 1. It operated as follows: during the 1 min sample loading period, a 2.4 ml min^{-1} flow of sample (standard or blank) at pH 3.6, buffered with glicine–HCl, is pumped (via P) through the microcolumn (located in the sampler arm); the metal ion is adsorbed on the sorbent microcolumn and the sample matrix is sent to waste; then, the switching valve (V_s) is actuated and the pumps of the AS-70 furnace autosampler, P_{AAS}, are connected, permitting the operation of the autosampler in the normal mode; a wash step takes place with de-ionized water and, immediately after, the sampler arm lowers the sample capillary into an autosampler cup (filled with eluent) aspirating 40 μl HNO_3 4 M; then, the sampler arm swings over to the graphite furnace and the tip of the sampler capillary is inserted into the dosing hole of the graphite tube where the eluted Rh(III) is deposited as a drop; The sampler arm then returns to its initial position and the cycle of the furnace operation commences (Table 1); while the temperature program is running, the switching valve is again turned to start a new loading of the sample (standard or blank); thus, when the spectrometer gives the measurement, the microcolumn is ready for a new injection of eluent.

3. Results and discussion

3.1. Optimization of the preconcentration procedure

Chemical parameters, as well as the analytical flow system, which was coupled on-line to the preconcentration and separation system, were optimized in order to obtain highly sensitive, accurate and reproducible results. Since the solution pH affects the extent of complexation which in turn determines the percentage of metal retained by the resin, the preconcentration of rhodium from solutions buffered at different pH were studied. The pH was adjusted from 2.0 to 3.6 using glycine–HCl buffer, from 4.0 to 6.5 using acetic acid–sodium acetate buffer and from 7.0 to 10.0 using boric acid–borax buffer. The optimum pH range was around 3.2–4.2 as can be seen in Fig. 2. All subsequent studies were carried out at pH 3.6.

Nitric acid was chosen as the eluent owing to its effective elution of the adsorbed analyte complex. The effect of eluent concentration on the absorption signal of 10 ng ml^{-1} Rh, using a constant volume of injection of eluent of 40 μl, was

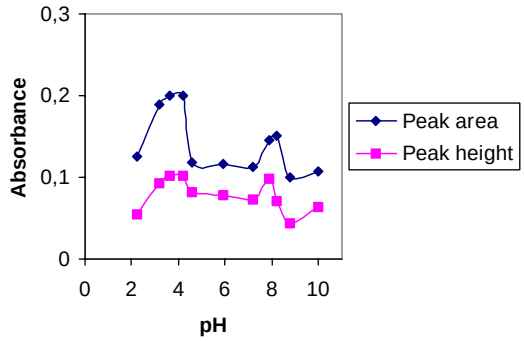

Fig. 2. Influence of pH on the preconcentration of rhodium.

examined. The signal increased as the HNO_3 concentration increased up to 3 M, then remained constant with further increase in the eluent concentration. The results are shown in Fig. 3. A HNO_3 concentration of 4 M was chosen for subsequent studies.

The influence of the volume of eluent used also was studied. The signal increased as the volume increased up to 35 μl, then remained constant with further increase in the volume of eluent. An injection volume of eluent of 40 μl was fixed.

The effect of sample loading time on the absorption signal of 2, 10 and 20 ng ml^{-1} Rh were tested. The signal increased linearly up to 5 min preconcentration time, after which the slope decreased gradually. The sensitivity was increased by increasing the sample loading time; however, a loading time of 1 min was selected in order to fit this time into the cycle of the furnace program and thus achieve a high sampling frequency with a reasonable degree of sensitivity. A longer loading time can be employed for samples with low concentration of rhodium.

Keeping constant the injection volume of 40 μl of 4 M HNO_3, the influence of the sample flow rate was studied. For this purpose, 24 ng of Rh were brought to pH 3.6 and passed through the column at different flow rate (the flow rate was varied by changing the speed of the sample pump). Changes in the flow rates of the sample were studied between 1.6 and 4.7 ml min^{-1}, resulting in an optimum sample flow rate of 2.4 ml min^{-1} with the best sample-to-blank ratio.

On the other hand, prior to the analysis of real samples it was necessary to optimize the instrumental conditions for

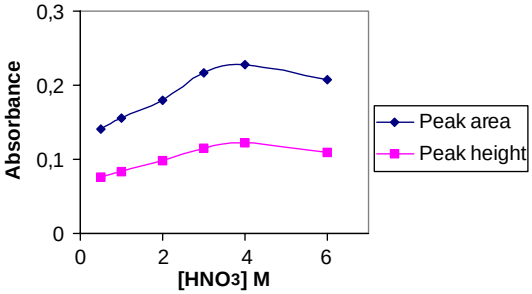

Fig. 3. Influence of eluent concentration.

Table 3
Performance of the FI-ET-AAS system for rhodium determination under the conditions given in the procedure

Analytical parameters	Peak-height	Peak-area
Working concentration range (ng ml^{-1})	0–50	0–50
Calibration function (C_{Rh} in ng ml^{-1})	$A = 0.011C_{Rh} + 0.0074$	$A = 0.0239C_{Rh} + 0.0015$
Correlation coefficient	0.9995	0.9976
Detection limit (ng ml^{-1})	0.3	0.8
Quantification limit (ng ml^{-1})	0.9	2.0
Precision (% R.S.D., $n = 10$, $C_{Rh} = 10$ ng ml^{-1})	3.0	1.8
Precision (% R.S.D., $n = 6$, $C_{Rh} = 5$ ng ml^{-1})	3.1	2.2
Sampling frequency (h^{-1})	30	30
Enrichment factor	18.3	20.0
Concentration efficiency (min^{-1})	9.2	10.0
Consumptive index (ml)	0.13	0.12

Table 4
Tolerance of foreign ions in the determination of 10 ng ml^{-1} rhodium

Ion or specie	Tolerance ratio (m/m)
Mg^{2+}, K^{+}, Na^{+}, Ni^{2+}, Zn^{2+}, Cd^{2+}, Mn^{2+}, Ba^{2+} [a], Cr^{3+} [a], Co^{2+} [a], Br^{-}, PO_4^{3-}, F^{-}, I^{-}, SO_4^{2-}, ClO_4^{-}, EDTA	>4000
Pb^{2+}, Al^{3+}, SCN^{-}	2000
Cu^{2+} [a]	1000
Ca^{2+} [a]	500
Fe^{3+} [a], Fe^{2+} [a]	250
Pt^{4+}	100
Sn^{2+} [a], Zr^{4+}	50
Ce^{4+}, Pd^{2+}	10

[a] With EDTA as masking agent.

the determination of rhodium by ET-AAS. The complete program developed as a result of the normal optimization procedure is as described in Table 1.

3.2. Performance of the method

The characteristic performance data of the FI-ET-AAS system for rhodium determination are presented in Table 3. The detection and quantification limit was defined as the concentration of analyte giving signals equivalent to 3–10 times, respectively, the standard deviation of the blank plus the net blank intensity. The enrichment factor (EF) was determined as the ratio of the slopes of the linear section of the calibration graphs before and after the preconcentration, the concentration efficiency (CE) was defined as the product of the EF and the sampling frequency in number of samples

Table 5
Results for rhodium determination in real samples

Sample	Certified value (mg kg^{-1})	Found value[a] (mg kg^{-1})	Recovery (%)
SRM 2557	135.1 ± 1.9	140.0 ± 2.9	103.6
	Added (mg kg^{-1})	Found[a] (mg kg^{-1})	
Bignonia leaves	125.0	125 ± 3	100
	62.5	63 ± 3	101
Pinus leaves	125.0	122 ± 2	98
	62.5	62 ± 2	99
Soil	50.0	50 ± 2	100
	25.0	25 ± 1	100
	Added (ng ml^{-1})	Found[a] (ng ml^{-1})	
Tap water	5	5.2 ± 0.4	104
	10	10.4 ± 0.3	104
River water	5	4.9 ± 0.1	98
	10	10.1 ± 0.1	101
Sea water	5	5.1 ± 0.2	102
	10	10.1 ± 0.2	101
Synthetic sea water	5	4.8 ± 0.1	96
	10	10.1 ± 0.1	101

[a] Mean ± standard deviation, $n = 4$.

analyzed per hour and the consumptive index was calculated as the volume of sample, in milliliters, consumed to achieve a unit EF.

3.3. Interferences

The results of including significant levels of possible interferents are presented in Table 4. For this study, different amounts of the ionic species tested were added to a 10 ng ml^{-1} solution of rhodium. The starting point was an interferent:rhodium ratio of 4000 (m/m); if any interference occurred, the ratio was gradually lowered until the interference ceased. The tolerance limits found show that rhodium can be determined in the presence of a variety of ions. The interference of diverse ions can be significantly lowered by the addition of EDTA to the medium.

3.4. Sample analysis

In order to test the accuracy and applicability of the proposed method for the analysis of real samples, one reference material was analyzed. The result, as the average of the four separate determinations, is shown in Table 5. As can be seen, the rhodium concentration determined by the proposed method is in close agreement with the certified value.

In view of the application of the method to the determination of rhodium in other samples, the ability to recover rhodium from samples of water, vegetation and soil spiked with rhodium was investigated. For this purpose, standard solutions containing rhodium were added to samples and the resulting material was prepared as described in Section 2. Standard additions method were used in all instances and the results were obtained by extrapolation. The results of these analysis are summarized in Table 5, and indicated excellent recoveries in all instances.

4. Conclusions

Despite its low detection limits, ET-AAS is still inadequate when the sample has a complex matrix. In these cases a preliminary preconcentration and/or separation is required. These operations, at one time often the “bottleneck” of the entire procedure, are now completely compatible with an efficient ET-AAS sequence. FI-on line column preconcentration-ET-AAS has revolutionized trace element analysis in samples with complicated matrices.

The system proposed in this paper has the advantage of being simpler than other FI-ET-AAS because the process is fully automated without complicated hardware and software; in fact modification of the software of the spectrometer was not necessary. The use of expensive and sophisticated instruments is also avoided. High speed, ease of use and automation, selectivity and relative freedom from interference make this method suitable for rhodium determination in different samples.

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