Quantitative Determination of Au, Pt, and Pd in Soil and Stream Sediment Samples by GF-AAS and ICP-MS

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ABSTRACT

A combined method has been developed for the determination of gold (Au), platinum (Pt), and palladium (Pd) in soil and stream sediment samples, based on closed vessel *aqua regia* digestion and solvent extraction by MIBK (methyl isobutyl ketone), followed by analysis with GF-AAS (graphite furnace atomic absorption spectrometry) and ICP-MS (inductively coupled plasma mass spectrometry). Matrix reduction prior to *aqua regia* digestion, solvent extraction after complex formation, and determination up to ppb levels are the key features of this method. The roasted sample was digested with (1:1) HCl in the closed vessel polytetrafluoroethylene (PTFE), filtered, and the residue digested with *aqua regia*. The content was evaporated to dryness and taken in dilute HCl. Gold was first extracted from this solution with

pre-equilibrated methyl isobutyl ketone (MIBK), then determined by GF-AAS using a standard procedure. The Pt and Pd were extracted from the raffinate (residual aqueous solution) by another portion of pre-equilibrated MIBK after complexing with the thiocyanate ligand. Both elements were quantitatively determined by ICP-MS after stripping with dilute HNO₃ from the loaded organic phase. Recoveries were found to be around 95% for all of the elements. The limit of quantitation (10 σ) achieved for Au, Pt, and Pd was 1.0, 0.5, and 1.0 ng/g, respectively. This method has been developed using eight certified reference materials (CRMs) of soil and stream sediment samples of different origin. The efficiency of the proposed method was evaluated by analyzing eight real soil and stream sediment samples collected by the Geological Survey of India.

and environmental issues. The Geological Survey of India is particularly concerned about the determination of three noble metals, i.e., Au, Pt, and Pd.

An extensive literature survey has revealed that a plethora of publications is available concerning the purification, separation, and determination of these precious metals in various geological and industrial materials (10-16). Several spectroscopic techniques (UV-VIS, FAAS, GF-AAS, ICP-AES, and ICP-MS) were extensively applied to quantify these noble metals over a large range of concentrations. Acid digestion under high pressure, coupled with a suitable preconcentration technique, finds wide application for the determination of these elements from both exploration and industrial aspects (12, 17). It is well documented in the literature that acid digestion produces the lowest procedural blanks among all the techniques employed for sample digestion (12). Therefore, it ensures the determination of PGEs at very low levels often found in geological materials. Among the preconcentration techniques of Au and the PGEs, solvent extraction has become a very important tool and is used for separation of these elements from a variety of geological matrices. Various reagents have been studied and proposed (18). Two comprehensive recent reviews in this regard are worth mentioning (19-20).

The analysis of Au by *aqua regia* digestion and solvent extraction by MIBK (methyl isobutyl ketone) has been proven to be a very convenient

INTRODUCTION

The members of the platinum group metals (PGM) and gold are called "precious" or "noble" metals. Gold is often found in nature as the metallic element and also in minerals with such elements as bismuth, antimony, arsenic, and tellurium. The platinum group elements (PGEs) are generally found in connection with sulfide ores of copper and nickel. The most common minerals of the PGEs are metals, metal

alloys, sulfides, arsenides, and tellurides. The average concentrations of gold and the PGEs in the continental crust are very low, i.e., less than 3 ng/g (1-9). Owing to their unique physical and chemical properties, such metals find wide application in industry and medicine. Soil and stream sediment samples are very important in geochemical mapping. Therefore, an accurate determination of precious metals like Au, Pt, and Pd in soil and stream sediment samples is important for identifying prospective mineralized zones, which is very useful in the parlance of economic

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method in most analytical programs in geochemical exploration studies (21). Considering this procedure as a template, we have incorporated some necessary modifications to accommodate both Pt and Pd to develop a combined method for the three metals. In this study, a roasted sample was taken into a PTFE pressure vessel and digested with distilled HCl. The filtered and ignited residue was further digested with *aqua regia* in the same pressure vessel. The contents were evaporated to dryness and finally taken in dilute HCl, filtered, and the volume made up to the required level. From this solution, gold was first extracted with pre-equilibrated MIBK and determined by GF-AAS using the standard procedure. Pt and Pd were extracted from the raffinate by another portion of the pre-equilibrated MIBK as their thiocyanate complexes and were quantitatively determined by ICP-MS after stripping with dilute HNO₃ from the loaded organic phase. The limit of quantitation (10 σ) achieved for Au, Pt, and Pd was 1.0, 0.5, and 1.0 ng/g, respectively. Eight certified reference materials (CRMs) of soil and stream sediment samples of different origin were used to develop this combined method. The efficiency of the proposed method was evaluated by analyzing 10 real soil and stream samples collected by the Geological Survey of India across the country. These samples were collected from Topo sheets 47H/12, 47J/1, 47J/2, 47J/5, 47J/6, 47J/7, 55P/5, 64E/16 and 48I/1. Samples of -200 mesh size were used for the digestion procedure.

EXPERIMENTAL

Instrumentation

The determination of gold was performed using a Varian 120 AA280Z graphite furnace atomic absorption spectrophotometer (Varian, Inc., USA). Details of the

analytical parameters, calibration strategy, and measurement of the samples by this GF-AAS method are published elsewhere (21).

The determination of Pt and Pd was performed with a PerkinElmer® ELAN® DRC™-e inductively coupled plasma mass spectrometer, equipped with a cross-flow nebulizer (PerkinElmer, Inc., Shelton, CT, USA). The instrumental operating parameters for the ICP-MS analysis are shown in Table I. Under these operating conditions, the sensitivity of ¹¹⁵In was >25,000 cps / ng/mL⁻¹, and the oxide production rate, calculated as the ratio of CeO/Ce, was <3%. The isotopes used were ¹⁹⁵Pt and ¹⁰⁵Pd.

Reagents

All reagents used were of analytical reagent (AR) grade (Sigma-Aldrich, Germany). High purity Milli-Q® water (18 M Ω ·cm) (Millipore Corporation, USA) was used in all of the sample preparation procedures. All glassware and digestion vessels were acid-washed and rinsed with Milli-Q water. Double distilled acids were used throughout. Nitric acid (4%) was prepared by appropriate dilution of concentrated distilled nitric acid. Fresh *aqua regia* was prepared before each experiment.

Indium Stock Solution (500 ppb)

A 100-ppm indium solution was prepared by digestion of 0.0302 g indium oxide (278 g/mol) with 10 mL (1:1) nitric acid, and made up to 250 mL volume, maintaining 4% HNO₃ concentration. This solution was used as the stock solution. A 500-ppb indium solution was prepared by appropriate dilution of a 100-ppm stock solution.

Au, Pt, and Pd Standard Solutions

The high purity single-element 1000 ppm standard solutions (Au, Pt and Pd) were purchased from the National Institute of Standards and Technology, USA. The calibration standards for the respective metals in ppm and ppb levels were prepared by appropriate step-wise dilution of the 1000 ppm original standards. A 100-ppb mixed standard solution of Pt and Pd was prepared.

Calibration

Calibration standard solutions of appropriate concentrations for ICP-MS were prepared from a 100-ppb mixed standard solution after adding the indium solution as an internal standard. The calibration standards used in these investigations were found to be stable over

TABLE I
ICP-MS Instrumental and Data Acquisition Parameters

Instrumental Parameters			
RF Power	1100 W	Plasma Gas Flow	15.50 L/min
Nebulizer Gas Flow	0.85 L/min	Lens Voltage	7.75 V
Auxillary Gas Flow	1.20 L/min	Sample Uptake Rate	1.0 mL/min
Data Acquisition Parameters			
Measuring Mode	Peak Hopping	Integration Time	2500 ms
Point per Peak	1	Replicates	3
Number of Sweeps	50	Internal Standard	¹¹⁵ In
Dwell Time	50 ms		

a period of at least one month. A 50-ppb calibration standard for Au was used for GF-AAS measurement. The ICP-MS instrument was calibrated with 10 ppb mixed standard solution.

Selection of CRMs

The chemical laboratories of the Geological Survey of India (GSI) use stream sediment and soil certified reference materials (CRMs) for calibration of various instruments for the determination of different elements. In this study, the following CRMs were used: GXR-2, GXR-5, and GXR-6 Soil Powder (U.S. Geological Survey, Denver, CO, USA), JSd-1, JSd-2, and JSd-3 Stream Sediment Powder (Geological Survey of Japan, Ibaraki, Japan), and GPt-1 Soil Powder and GPt-2 Sediment Powder (Institute of Geophysical and Geochemical Exploration, Hebei, P.R. China).

Procedure

An amount of 5 g powdered sample was placed into a porcelain crucible and roasted for 1 hour in a muffle furnace at 650 °C. The roasted sample was transferred to a 500-mL polytetrafluoroethylene (PTFE) pressure vessel and digested with 120 mL distilled 6N HCl for 10 hours. The content of the vessel was filtered through an ashless Whatman #41 filter paper in warm condition and rinsed several times with hot water to remove Pb as the soluble salt. Then the filtrate was discarded. The residue was dried and ignited in a porcelain crucible and transferred to the same PTFE pressure vessel. Freshly prepared 60 mL *aqua regia* was added, and the pressure vessel was closed with a screw cap and heated on a hot plate for four hours at ~200 °C. After the *aqua regia* digestion, the screw cap was removed, and the content evaporated to dryness. Any trace of nitric acid was evaporated by treating the mass twice with 10 mL concentrated distilled HCl.

Finally, the content was filtered and made up to volume with 1N HCl in a 50-mL volumetric flask. This solution was transferred into a 250-mL separating funnel, and 10 mL of pre-equilibrated MIBK was added. The solution was shaken for about three minutes, allowed to settle, and the aqueous layer collected in a beaker. The organic layer was again shaken with 10 mL of 0.1N HCl for about two minutes and allowed to settle. Then the aqueous layer was discarded, and the MIBK layer used for GF-AAS measurement of gold. Gold from this organic phase may also be stripped in 2N HCl and can be measured by GF-AAS. To the raffinate (residual aqueous solution), 2 mL of 10% (w/v) ascorbic acid solution was added, followed by the addition of 2 mL of 10% (w/v) NH_4SCN solution. The colorless solution was allowed to stand for five minutes, and was then quantitatively transferred to a 250-mL separating funnel. The funnel was shaken for five minutes with another 10 mL of pre-equilibrated MIBK, and allowed to settle. The aqueous layer was discarded, and the loaded organic phase was stripped with 2N HNO_3 . The final volume was adjusted to 5 mL with the same reagent and placed into a clean and dry graduated glass-stoppered culture tube for ICP-MS measurement of Pt and Pd.

RESULTS AND DISCUSSION

Choice of Sample Aliquot

Precious metals are generally found to be distributed heterogeneously in geological samples and tend to form discrete particles or mineral phases. As a result, a large sample aliquot is required to minimize the probability of getting an improper representation of such metals (caused by the 'nugget effect') in the samples under analysis. Although the fire assay technique offers the advantage of accepting large sample aliquots

(usually 20-50 g), it suffers from some potential disadvantages like requirement of large amounts of flux, which often results in high procedural blank and loss of analytes during the course of the entire process. Keeping this in mind, initially a minimum of a 5-g sample had been chosen for the development of this method; later, the method was tested with a 10-g sample. It was found that the method is equally operative with a negligible increase of procedural blank value, although the consumption of acid was increased by at least 20-30%. For a 10-g sample, 150 mL 6N HCl and 100 mL *aqua regia* are sufficient for this method.

Quantitative Analytical Results

Table II and Table III list the analytical results of the CRMs (GXR-2, GXR-5, GXR-6, JSd-1, JSd-2, JSd-3, and GPt-1, GPt-2) and the in-house generated samples, respectively. Each value is obtained from the average of six replicates prepared separately for each sample. It can be seen that the data of most elements are in reasonable agreement with the certified data, showing that the methodology proposed here is most useful for gold, platinum, and palladium determination.

Effect of Open and Closed Vessel Digestion

For this study, closed vessel digestion with 6N HCl was used to remove the interfering matrix elements such as Fe^{3+} , Al^{3+} , Mn^{2+} , Ti^{4+} , Ni^{2+} , Co^{3+} , and Cu^{2+} . Since the thiocyanate ligand forms complexes with these metal atoms, complete removal of these metal ions facilitates the use of this ligand for complexation of Pt, Pd, and the subsequent extraction by MIBK.

In addition, the XRF analytical data of several hundred soil and stream sediment samples were studied. These samples were collected by GSI from different parts of the country using specific guide-

lines (22) and can provide a precise idea about the matrices present in such samples. It was found that the above-mentioned interfering elements were present as matrix in the following concentrations: Fe₂O₃ <10%, Al₂O₃ <20%, MnO <1%, TiO₂ <3%, Ni <60 ppm, Co <50 ppm, and Cu <50 ppm.

Digestion of the roasted sample with 6N HCl by both open and closed vessel methods was examined thoroughly. It was found that a better removal of the above-mentioned interfering elements was obtained by closed vessel. Although complete removal of the matrix was not achieved, the initial digestion with 6N HCl in closed vessel facilitated optimum utilization of ascorbic acid and thiocyanate ligand for complexation of Pt, Pd, and their subsequent extraction by MIBK (see Procedure section). Minimum procedural blank values achieved by this procedure also ensures the effectiveness of this process. In the thiocyanate extraction method, the results of Au estimation by ICP-MS are not reproducible.

In a separate experiment with 10 g stream sediment samples, 5 mg of pure metals (Au, Pt, and Pd) was added and the above-mentioned procedure performed with utmost care to verify the validity of the proposed method. The recovery of the pure metals (Au, Pt, and Pd) was found to be around 95%. Therefore, partial removal of the mega matrix in these samples by closed vessel digestion prior to *aqua regia* attack proved to be more beneficial for the following reasons: (a) relatively large sample amounts (5 to 10 g) can be used for analytical purposes, (b) partial matrix removal by closed vessel digestion is very simple, fast, effective, and safe, (c) procedural blank value is low, despite taking relatively large amounts of sample and subsequent acid treatment,

TABLE II
Analytical Results of Au Determination by GFAAS and Pt and Pd Determination by ICP-MS in CRMs

Sample No. CRM	C.V./ O.V.	GFAAS		ICP-MS			
		Au (µg/g)	Recovery (%)	Pt (µg/g)	Recovery (%)	Pd (µg/g)	Recovery (%)
1. GXR-2 ^a	C. V.	0.036	94.44	-	-	0.0001	-
	O. V.	0.034		<0.0005		<0.001	
2. GXR-5 ^a	C. V.	0.016	93.75	0.050	94.00	0.0004	-
	O. V.	0.015		0.047		<0.001	
3. GXR-6 ^a	C. V.	0.095	94.74	0.150	90.00	0.0022	95.45
	O. V.	0.090		0.135		0.0021	
4. JSD-1 ^b	C. V.	0.00093	-	-	-	0.0005	-
	O. V.	<0.001		<0.0005		<0.001	
5. JSD-2 ^b	C. V.	0.053	94.34	0.0167	95.21	0.0212	94.81
	O. V.	0.050		0.0159		0.0201	
6. JSD-3 ^b	C. V.	0.00566	95.05	0.0013	92.31	0.0032	93.75
	O. V.	0.00538		0.0012		0.0030	
7. GPt-1 ^c	C. V.	0.0009	-	0.00026	-	0.00026	-
	O. V.	<0.001		<0.0005		<0.001	
8. GPt-2 ^c	C. V.	0.01	95.00	0.0016	93.75	0.0023	91.30
	O. V.	0.0095		0.0015		0.0021	

O.V.: Observed value. Observed values are the mean of six determinations.
C.V.: Certified value.

^a GXR-2, GXR-5, and GXR-6 Soil Powder.

^b JSD-1, JSD-2, and JSD-3 Stream Sediment Powder.

^c GPt-1 Soil Powder and GPt-2 Sediment Powder.

TABLE III
Results of Au, Pt, and Pd Determination in Stream Sediment Samples by GF-AAS and ICP-MS (in ppb)

Sample No.	Topo Sheet No.	Sample No Details	GFAAS.	ICP-MS	
			Au	Pt	Pd
1.	47H/12	47H/12/043/CS/02	9	1.9	9.1
2.	47J/1	47J/1/53/S/03-04	4	6.8	9.4
3.	47J/2	47J/2/133/S/05-06	2	6.2	9.2
4.	47J/5	47J/5/033/S/02-03	2	5.6	8.9
5.	47J/6	47J/6/032/S/08	2	5.4	10.1
6.	47J/7	47J/07/14/08-09	<1	3.5	9.0
7.	55P/5	55P5/030/S/05	<1	7.0	11.2
8.	48I/1	48I01/147/S/12	14	<0.5	<1
9.	48I/1	48I01/03/S/12	18	<0.5	<1
10.	48I/1	48I01/165/S/12	24	<0.5	<1

(d) minimum use of reducing and complexing agents, which also contributes to lowering the blank values.

Choice of Thiocyanate as Ligand

In terms of Pearson's classification of "soft" and "hard" acids and bases with regard to the different polarizability of atoms, the thiocyanate ligand coordinated by a sulphur atom is a "soft" and coordinated by nitrogen atom is a "hard" base. Owing to its relatively low ionization energy, high polarizability, and available dative π bonds, sulfur as donor atom has, in comparison with the nitrogen atom, greater potential possibilities to affect the oxidation-reduction properties of the central atom. The coordination mode of the thiocyanate ligand in compounds depends on properties of the central atom and on properties of other ligands in the coordination sphere. In a simplified manner, the effect of the nature of the central atom on the coordination mode of the thiocyanate ligand can be shown on the homogeneous thiocyanates. The unidentate thiocyanate ligand is coordinated with Rh(III), Ir(III), Pd(II), Pt(IV), Pt(II), Ag(I), Au(III), and Hg(II) through the sulphur ("soft" donor) atom, and with the remaining transition metals through the nitrogen atom ("hard" donor) (23-27).

The most prominent feature of the aqueous chemistry of the platinum group metals in chloride solution is the strong tendency of the metals to form anion complexes with the chloride ion (23-25). Unlike HAuCl_4^- , chloro complexes of Pt and Pd are poorly extracted with MIBK. This was also examined from a mixed 10 ppb synthetic solution of metals under investigation in 1N HCl. It was found that only Au was extracted with MIBK above 95%, leaving the rest of the metals (>99%) in the raffinate. This restricts the direct extraction of PtCl_6^{2-} and PdCl_4^{2-} by MIBK and

demands any suitable complexing agent for extraction purposes. Palladium(II) in the presence of SCN^- undergoes a substitution reaction almost instantaneously, whereas owing to the kinetic inertness of PtCl_6^- , formation of the Pt(IV) thiocyanate complex is highly dependent on SCN^- and the acid concentrations. Since both metals prefer to coordinate most strongly with the polarizable sulphur atom of SCN^- , these complexes have been shown to be highly extractable into oxygen containing the organic solvents (28). Experiments with a mixed 10 ppb synthetic standard solution of Pt(IV) and Pd(II) in 1N HCl at room temperature results in incomplete extraction of Pt(IV), whereas almost 100% extraction of Pt(IV) was achieved after applying heat. When this synthetic, mixed standard solution of Pt and Pd was first allowed to react with ascorbic acid followed by the addition of SCN^- ligand, both metals were extracted almost 100% from the solution. Ascorbic acid reduces Pt(IV) to Pt(II), which is kinetically labile and forms a complex with SCN^- at normal conditions (29). In a geological matrix, ascorbic acid also reduces Fe^{3+} to Fe^{2+} , thus preventing the formation of the blood red Fe(III)-SCN complex. First, extraction of gold by pre-equilibrated MIBK is advantageous in the sense that during this extraction a significant amount of other metals like Fe^{3+} , Co^{3+} , and Ni^{2+} are co-extracted with gold, thus further reducing interfering elements before the addition of ascorbic acid and ammonium thiocyanate. The analytical results of the CRMs and in-house generated samples are listed in Tables II and III, respectively.

CONCLUSION

In this study, a combined and modified method was developed to quantify Au, Pt, and Pd in soil and stream sediment samples by closed

vessel digestion. The GF-AAS and ICP-MS techniques were employed for the quantitative determination of Au and Pt and Pd, respectively. The entire process can be subdivided into the following integral parts: (a) Partial removal of the matrix in PTFE pressure vessels, (b) dissolution of residue by *aqua regia* in the same vessel, (c) pre-concentration of gold by MIBK, (d) pre-concentration of Pt and Pd by MIBK as thiocyanate complexes from the raffinate, (e) determination of Au by GF-AAS, and (d) determination of Pt and Pd by ICP-MS. Since a single decomposition procedure is involved for the estimation of three precious metals simultaneously, the proposed method is simple, quick, economical, and safe. The reagents used are also readily available. Although the thiocyanate complexes of Pt and Pd are well documented, extraction of these complexes were never exploited for the ICP-MS estimation of these metals up to the 0.5 and 1.0 ng/g levels, respectively, in soil and stream sediment samples.

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