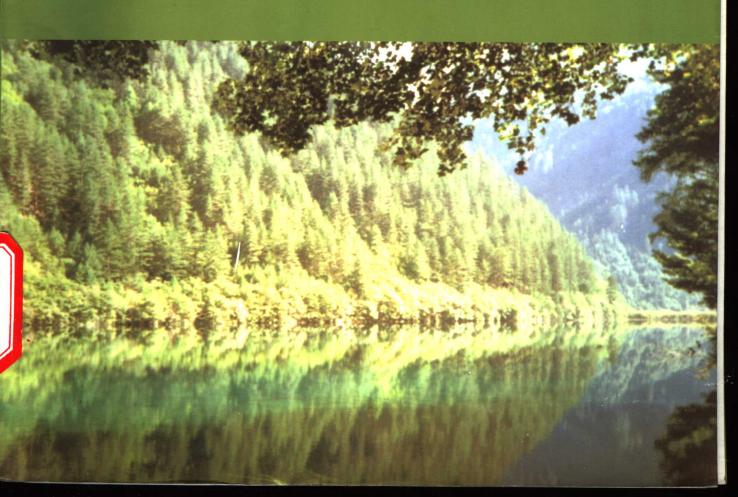
国家自然科学基金重大项目

施分析技术与旅游

"现代核分析技术及其在环境科学中的应用"项目组 著

原子能出版社



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核分析技术与环境科学

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内容简介

本书收录的 31 篇论文反映了分子活化分析、扫描质子微探针、高分辨率 X 射线谱学、加速器质谱学、核分析质量控制技术等现代核分析技术方法学及其在地球灾变环境、稀土元素生态环境、有机毒物体内环境及大气环境中应用研究的最新成果,是国家自然科学基金委员会"八五"重大研究项目"现代核分析技术及其在环境科学中的应用"的部分成果汇编。

本书可供科研院所、高等学校、科学技术管理部门从事核技术、环境学、生物医学和地学的广大专业人员和有关人员参考。

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前 言

本书是国家自然科学基金委员会"八五"重大研究项目"现代核分析技术及其在环境科学中的应用"(1993年10月至1997年12月,项目编号:19392100)的论文选编。4年来,参加本项目的中科院高能物理研究所、北京大学、中科院上海原子核所、复旦大学、中科院地理所、中科院地球化学所和中国原子能科学研究院的近百名科研人员兢兢业业、辛勤工作,取得了一系列富有创造性的成果。本论文集在一定程度上是项目组全体成员所获成果的一个缩影。

现代核分析技术是一门以粒子与物质相互作用、核效应及核谱学等为基础,由多种方法组成的综合技术,具有灵敏度高、准确度好、可同时测定多种元素、不破坏样品、基体效应小等特点,往往可以起到一般非核分析方法难以起到、甚至无法起到的作用。本书中收录的论文不仅反映了现代核分析技术的一般特点,而且体现出了当前国际上核分析技术的发展趋势:从微量(µg/g 量级)到超微量(ng/g 量级或更低),从总量到化学种态,从整体到微区,从单因子到多因子,以及从单一介质到多介质(包括界面)。对于当前环境科学研究中人们高度关注的生物标记物,本书中也有优秀论文予以介绍。

本重大研究项目设置了8个子课题:

- (1)分子活化分析研究;
- (2)扫描质子微探针方法研究;
- (3)新型 X 射线能谱分析研究;
- (4)加速器质谱分析研究;
- (5)现代核分析技术质控研究;
- (6)灾变环境研究;
- (7)稀土元素生态环境研究;
- (8)有机毒物分子与 DNA 的加合作用和大气甲烷来源的研究。

本书收录的 31 篇论文依次反映了上述 8 个子课题研究的部分研究成果。由于著者众多,且风格迥异,更由于上述 8 个方面的发展程度不同,因此要求全书在体例和格式上完全和谐一致是难以做到的,我们只力图保证每篇论文自成体系。书中涉及内容较多,读者完全可以按照自己的兴趣挑选有关论文阅读。

本书书末的论著目录列出了 4 年来项目组发表的论文以及在学术会议上特邀报告的目录。由于篇幅有限,不少优秀论文无法以其全貌展现在读者眼前,因此有兴趣的读者可查阅有关文献。

在项目研究和本书编写过程中,我们得到了国家自然科学基金委员会数理学部、化学部、地学部、生物学部和重大项目处以及承担本重大项目的 7 个单位领导和职能部门的支持。著者谨向他们表示衷心的感谢。原子能出版社的袁祖伟同志和高能所的王晓光同志,为本书的编辑出版尽心尽力,著者亦向他们致以谢意。

柴之芳 1997 年 9 月 10 日于北京

Molecular Activation Analysis for Iridium

CHAICF KONGP MAOXY and MASL

Institute of High Energy Physics and Laboratory of Nuclear Analysis Techniques, the Chinese Academy of Sciences, P. O. Box 2732, Beijing, 100080, China

Abstract

Molecular activation analysis (MAA) of iridium in geological samples, based on a combination of a newly-developed chemical sequential dissolution method and radiochemical and instrumental NAA, was described to study the distribution pattern and chemical species of iridium in various fractions (soluble carbonate, metal, sulfide, oxide, silicate and acid-insoluble residue) of geological boundary samples, meteorites, ultrabasic rock and volcanic lava. The correlations of Ir with Au, Os, siderophile, chalcophile and lithophile elements were discussed. In addition, the role of kerogen and noble-nugget in the Ir enrichment was scrutinized. The MAA results of Ir favor a mixed effect of asteroid impact, volcanic eruption and post-depositional redistribution to interpret the extant Ir anomaly at Cretaceous-Tertiary boundary layer.

Molecular activation analysis (MAA) has been widely used in environmental and biomedical fields to study the chemical species of trace elements in various tissues, body fluids, soil and water samples^[1-4]. The main trace elements of interest are selenium^[1], chromium^[4], arsenic^[5], REE^[6], mercury^[7], etc. However, little work was done in MAA of geological samples, mainly because of difficult separations for them. More than 10 years have passed since ALVAREZ et al. ^[8] first found the Ir anomaly at two Cretaceous-Tertiary(K-T) boundary clay samples from Denmark and Italy. However, although a large number of experimental results have been accumulated, none of them, including shock-metamorphic quartz, microspherules, isotopic composition, amino acid, etc., is exclusive. Currently, there are still several different models to interpret the origin of anomalous Ir. One typical example is that Van Valen^[9,10] cited 10 pieces of evidence for the extraterrestrial impact model, but also listed 15 pieces of evidence against it. At present there are 3 main models about Ir anomaly, i. e. extraterrestrial, volcanic and geochemical. Due to the relationship of the origin of anomalous Ir with its chemical species, we attempted to reveal the Ir species in samples of interest by using a newly-developed molecular activation analysis procedure for Ir.

1. Basic thoughts of Ir-MAA

The Ir-MAA is based on the following five possible iridium species in nature according to the known chemical behaviors of Ir. (1) A compound of Pt-group elements. In some cases Ir in extraterrestrial matter may exist in a form of noble nugget[11], which consists of Ir as a pure metal or with other noble metals in a binary or polynary alloy. If the extraterrestrial impact model is valid, Ir may have been enriched in such a nugget via a series of physical and chemical differentiation process at high temperature and pressure produced at the time of the entry of extraterrestrial matter into the atmosphere and its collision with the earth. (2) Due to its siderophile nature, Ir may exist in a Fe-Ni metal phase, a main host of Ir in meteorites. (3) Also, being chalcophile, Ir is present as sulfide. (4) Ir forms soluble complex ions. Ir belongs to the third long-period transition elements, owing to an incompletely filled d-orbit with strong complexing ability. In the marine system, Ir is easily able to form complex ions with chloride ions. (5)Combined with organic matter, Ir is present in kerogen, as reported by KUCHA[12] in the Cu-bearing shale at Zechstein . DISSANAYAKE and KRISTOTAKIS[13] determined the high content of noble metals at $\mu g/g$ levels from recent algae mats. Recently, SCHMITZ et al. $^{[14]}$ found about 50 % Ir enriched in the organic matrix. In case of the volcanogenic origin of Ir, sulfide is likely the main species. If the geochemical enrichment of Ir is valid, it should predominantly exist in soluble ions, sulfides and /or organic matter. If anomalous iridium mainly originates from the extraterrestrial matter, its chemical species should be the first one, i.e. a compound of Pt group metals. Ir would exist in the acid-insoluble phase, because of its high chemical inertness and strongly acid-resistant properties. Thus, Ir-MAA will, doubtlessly, help us elucidate the origin of anomalous Ir.

2. Chemical procedure for Ir-MAA

WOLBACH et al. [15] and SCHMITZ et al. [14] studied the HF-insoluble residue by simple treatment of K-T boundary samples with HCL and HF to remove carbonates and silicates in them. As mentioned above, we attempted to study the chemical species of Ir by the MAA procedure. Evidently, our purpose is different from theirs. For that reason we had to develop a chemical procedure of Ir MAA without disturbing primary Ir species in the samples. Indeed, some authors introduced physical methods for it, but none of them are appropriate for very fine clay samples, thus we have developed a new chemical method with a series of leachants with various concentrations under different conditions. As a principle, a leachant must be capable of selectively attacking and quantitatively extracting elements present in one particular phase regardless of other phases present. While a particular phase of interest in a multiphase system is completely dissolved, the rates of dissolution of other phases present must be negligible. Its basic steps are as follows. (1) Remove carbonate and other soluble components in geological samsic steps are as follows. (1) Remove carbonate and other soluble components in geological samsic steps are as follows.

ples by using citric acid and ascorbic acid at 30 °C. (2) Dissolve metal phase with hydroxylamine and citric acid at pH=5 and reflux for 8—10 min. (3) Extract sulfide by Br-saturated water at room temperature. (4) Leach oxide by 6 mol/L HCl and reflux for 20 min. (5) Finally, dissolve silicate by 40 %HF at 120 °C. The remaining fraction is called the residue phase. The detailed Ir-MAA procedure has been published elsewhere^[16].

The radiochemical NAA based on a long-chain primary amine extraction^[17] and instrumental NAA were used to determine the contents of Ir and other trace elements in various phases of the samples of interest, including some K-T boundary samples at Stevns Klint, Denmark, Montana and Colorado, USA, and Morgan Creek, Canada, meteorites, ultrabasic rock and volcanic lava at Kilauea, Hawaii. The analytical error for the Ir determinations ranges from 10 to 20 %, while it is 5 %—10 % for other elements. The DINO-1 prepared by the Lawrence Berkeley Laboratory, USA, was used for analytical quality control. The contents of light elements were analyzed by a combustion method and X-ray diffraction and ECAX spectrometry were used to investigate the mineral and chemical composition of minute particles in the residue phase.

3. Results and discussion

3. 1 Phase distribution of Ir and other trace elements in K-T boundary,

meteorite, ultrabasic rock and volcanic lava

We determined the phase distribution patterns of Ir and other trace elements (Au, Ni, Co, Fe, As, Sb, Se, Zn, Cr, Sc and Th) in the K-T boundary samples at Stevns Klint, Morgan Creek, Montana and Colorado, the Ningqiang carbonaceous chondrite (CV3), the Baoxian chondrite (LL4) and the Shuizhou chondrite (L5), an ultrabasic rock from Xingjiang, and volcanic lava from Kilauea, Hawaii, by our MAA procedure. Table 1 lists the results of the Stevns Klint K-T boundary. It can be seen from Table 1 that the Ir content increases from 13.8 ×10⁻⁹g/g in the bulk rock to 162 ×10⁻⁹g/g in the residue phase, i.e. Ir enrichment factor of 11.7 relative to the bulk rock, being in good agreement with 11.8 of SCHMITZ et al. [14]. Furthermore, the relative percent of Ir in the residue phase is 54.5 %, also similar to their 47 %. Comparing with their work, we not only studied the distribution patterns of Ir and other elements in the residue phase, but the distributions in other phases. It is evident from Table 1 that Ir in the Stevns Klint sample is mainly present in the residue phases, followed by metal, silicate, oxide, carbonate and sulfide. Interestingly, no Ir exists in the sulfide phase at all.

Because the Stevns Klint K-T boundary is a marine sediment, we have also studied several continental K-T boundaries. Fig. 1 compares the distribution patterns of 3 continental K-T boundaries, i. e. Montana, Colorado and Morgan Creek. It is clear from Fig. 1 that the acid-insoluble residue phase is the main host for all K-T boundaries, no matter whether they are

Table 1 Distributions of Ir and other elements in the Fish Clay K-T boundary, Stevns Klint. Denmark

Ir /10-9 Ir /% Au /10-9 Au /% Ni /10-6 Oo /10-6	13.8			Suma	Oxide	Cilicate	Kesidue
Ir /% Au /10-9 Au /% Ni /10-5 Ni /% Co /10-6		2.23	31.6		9, 1	6.6	162
Au / 10 ⁻⁹ Au / % Ni / 10 ⁻⁶ Ni / % Co / 10 ⁻⁶	100	4.0	21.5	0	9.4	12.0	7.47
Au /% Ni /10-6 Ni /%	9.2	17	52.0		. :	1	OS S
Ni /10-6 Ni /%	100	49.9	15.5	0	C	C	80 80 80 80
Ni /%	875	366	5541	374	92.4	40.8	÷ :
ال، /١١١-و	100	28.0	59.4	4.6		1.7	c
27 / 22	129	147	850	41.6	24.5	0.543	>
%/ °O	100	28.2	61.8	7.1	2.7	0.1	C
Fe /10-6	006 68	!	496 000		68 400	26 600	449 000
Fe /%	100	0	51.8	0	10.8	25.8	21.6
As /10-6	9.57	8.63		307	:) 1
As /%	100	26. 1	0	73.9	0	С	c
Sb /10-6	6.93	3.60	31.3	6.26	10.9	1.37	»
%/ qs	100	13.9	39.7	24. 1	16.1	6.1	C
Se /10-6	29. 5	49.2	1	54.7	4, 4,	11.6	>
% / 3	100	44.6	0	41.5	1.7	12.3	c
% /10−6	225	275	543	383	. 00	° 6	·
Zn /%	100	32.6	28.2	39. 1	0.2		c
)r /10-6	141	1	540		1250	165	>
Cr /%	100	0	28.2	0	35. 3	36.5	c
ic /10-6	9.9	2.99	34.6	i	37.3	8, 68	•
%/ 3S	100	8.2	30.6	0	33.8	27.4	c
h /10-6	2.51	7.11	5.72	ı	0.71	.	> }
Th /%	100	75.8	20.0	0	3.9	0	0
Phase Volume / %		24.7	0	ć	•		

Notes: The data in the first row for each element are its content in the samples, while the data in the second row are its relative percent.

- means too low of an element content to be detected.

marine or continental. It indicates that the anomalous Ir has no relation to the sedimentation environment, i. e. the geochemical factor does not play any important role in the Ir enrichment.

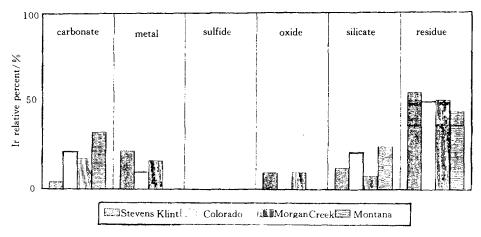


Fig. 1 Distribution patterns of Ir in K-T boundary samples

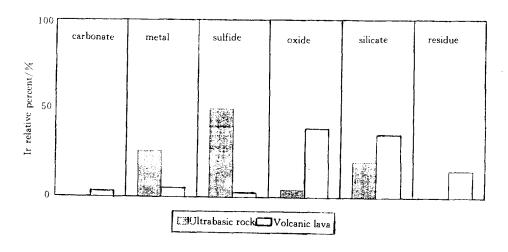


Fig. 2 Distribution patterns of Ir in ultrabasic rock and volcanic lava

Figs. 2 and 3 show the results for 3 types of meteorites, an ultrabasic rock and a volcanic lava. It can be seen that Ir is mainly distributed in the sulfide phase in the ultrabasic rock, a representative of the upper mantle, then in the metal and silicate in decreasing order. As mentioned above, the K-T boundary samples have no Ir in their sulfide phase. Furthermore, the percent content of Ir in the residue of the ultrabasic rock is as low as 5 %, which is also remarkably different from the K-T. As to the Hawaii volcanic lava, its Ir mostly exists in oxide and silicate phases (>70 %), while only 10 % of Ir is in the residue phase. These results do not support the volcanic origin of anomalous Ir. In our previous paper^[18], the difference between ultrabasic rock and volcanic lava has been discussed.

6

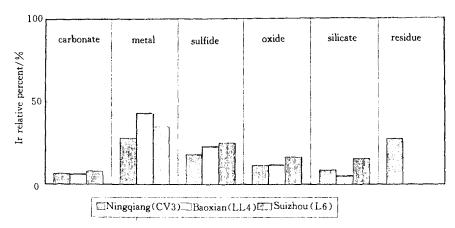


Fig. 3 Distribution patterns of Ir in three types of chondrites

Significantly, the Ir distribution pattern of the Ningqiang chondrite is quite similar to the K-T, while different from ultrabasic rock and volcanic lava. This result favors the model of the extraterrestrial impact on the earth causing the Ir anomaly. However, it can be found by careful comparison that the content of Ir in the K-T residue is 20 % higher than that of Ningqiang, while the sulfide and metal phases show the contrary tendency. A possible interpretation is that during the high temperature and pressure caused by the impact event, Ir may have been migrated from the sulfide and metal phase into the refractory phase via oxidation^[19].

As for the two ordinary chondrites (Baoxian and Suizhou), their relative percent of Ir in the residue phase is below 3 %, which is significantly different from the K-T. Based on the Ir species obtained by the Ir-MAA procedure, it seems to indicate that carbonaceous chondrites might be a source matter resulting in the Ir anomaly of the K-T boundary, if the impact model is valid.

3. 2 Correlation of Ir with Au and Os

The ratios of Ir/Au and Ir/Os can be used as indicators of extraterrestrial matter^[20]. Table 2 lists the ratio values of Ir with Au and Os in various phases of the K-T boundary at Stevns Klint, Denmark. It can be seen that the K-T bulk rock has the Ir/Os of 1.39 and the Ir/Au of 1.5—3.3. Significantly, its residue phase has the similar ratios (1.02 of Ir/Os and 2.03 of Ir/Au). It is well-known that the solar Ir/Os and Ir/Au ratios are 1.0 and 3.3, respectively^[21], while the terrestrial ratios are 0.04 and 0.02^[22]. The Ir/Os and Ir/Au ratios of the residue phase of the K-T boundary

Table 2 Ratios of Ir/Os in various phases of K-T boundary, Stevns Klint

Phase	Ir/Au	Ir/ Os
Bulk	1.50 -3.3	1.39
Carbonate	0.13	
Metal	0.61	
Oxide		
Sulfide		
Silicate		
Residue	2. 03	1.02
SCHMITZ et al.	2.57	-
Solar	3. 3	1.0
Terrestrial	0.02	0.04

Note: - means no data.

are close to the solar ones, which states that these elements are present in the same species and supports the cosmogenic origin of anomalous Ir.

FISENKO et al. [22] reported the Ir/Au ratios in the range of 0.75 to 330 in the acid-insoluble phase from the Efremovka carbonaceous chondrite, being in accordance with ours.

The Ir/Os and Ir/Au ratios in other phases (carbonate, metal, oxide, sulfide and silicate) differ from the solar ones, which indicates their terrestrial source.

3. 3 Correlation of Ir with siderophile, chalcophile and lithophile elements

In our previous paper^[24], we mentioned the positive relationship of Ir with siderophile and chalcophile elements in the K-T boundary bulk samples at Stevns Klint. However, by our new MAA procedure we found no correlation between their chemical species (see Fig. 4). It is evident that the siderophile elements, e. g. Co, mainly exist in the metal phase, while the chalcophile elements, e. g. Sb, are present in the metal and sulfide phases. As to the lithophile elements, the main host is oxide. The unique distribution pattern of Ir implies that it may exist as a compound with other Pt-group elements, favoring the cosmogenic origin of anomalous Ir in the K-T boundary.

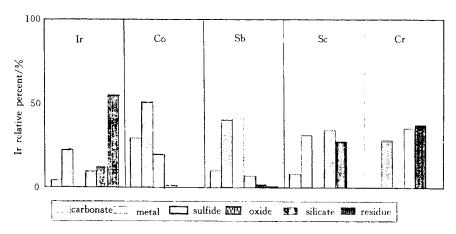


Fig. 4 Elemental phase distribution patterns of the Fish Clay K-T boundary samples. Stevns Klint, Denmark

3.4 Ir and kerogen

In his study of the anomalous Ir in the Fish Clay K-T boundary, SCHMITZ et al. [14] claimed that the remaining Ir is mostly present as a metallic organocomplex after the strong acid treatment. But they did not analyze the residue phase. Thus, we used a combustion method to determine the contents of C, H and N in the residue phase of the Danish K-T boundary samples, Concentration of 33 %—35 % for C, 2.2 %—2.6 % for H and 0.9 %—3.8 % for N in the Fish clay K-T residue phase lie well in the range of the kerogen [25]. But

the contents of C, H and N for the Montana K-T residue are 3.3 %—5.0 %, 1.9 %—2.0 % and 0.3 %, respectively. Such a low carbon content indicates little or no relation between the residue and kerogen. Thus, kerogen is probably not required for Ir enrichment, i.e. the Ir anomaly is not necessarily associated with the organic matter^[18].

3. 5 Ir and the noble nugget

We found a spherule with a 0.1 μ m diameter whose compositions were Ir (5 %), Os (5 %) and Rh (90 %) in one K-T residue phase by the method of transmission electron microscopy and EDAX^[18] and calculated the probability of finding this kind of spherule. We suggest that the spherule of noble metals was formed under the conditions of high temperature and pressure caused by an impact of extraterrestrial matter on the target. This provides further evidence for the cosmogenic origin of anomalous Ir.

4. Summary

The conclusions drawn by our results on the basis of the new molecular activation analysis of Ir and other elements are as follows.

- (1) The anomalous Ir of the K-T boundary mainly originates from extraterrestrial matter, likely from a body having the chemical composition of carbonaceous chondrite, e.g. type C asteroid. About 74 % of the asteroids in the asteroid belt belong to this class.
- (2) A volcanogenic origin of the excess Ir at the K-T boundary seems to be rejected, although volcanic activity must have participated in the extinction event at the end of the Cretaceous.
- (3) The organic matter in microorganisms, such as algae and bacteria, certainly played a role during the enrichment of Ir at the K-T boundary in marine sediments, but their role may have been insignificant in continent sediments. Moreover, this Ir enrichment by secondary geochemical processes probably mainly derives from decomposed extraterrestrial debris and dust formed at the impact event.

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铱的分子活化分析

柴之芳 孔屏 毛雪瑛 马淑兰

中国科学院高能物理研究所,核分析技术开放研究实验室

摘要

本文叙述了一种基于新发展的化学逐步溶解法和放射化学中子活化及仪器中子活化的分子活化分析法,可用于研究地质界线样品、陨石、超基性岩和火山熔岩中铱的分布特征及化学种态。借此,讨论了 Ir 与 Os、Au、亲铁元素、亲硫元素和亲石元素的相关性,以及干酪根和贵金属微粒在铱富集过程中的作用。铱的分子活化分析结果有利于白垩纪/第三纪界线铱异常来源的混合模型,即由小行星撞击、火山喷发和沉积后再分配这些作用共同造成铱异常。

A New Preconcentration Method for Platinum and Gold

Based on a Macropore Anion Resin HHY-10A

LI Chunsheng^{a,b} CHAI Chifang^{a,b} YANG Xuefeng^c

HOU Xiaolina, and MAO Xueyinga, b

- a Institute of High Energy Physics, the Chinese Academy of Sciences, Beijing, 100080, China
 - b Laboratory of Nuclear Analysis Technique, the Chinese Academy of Sciences,

P.O. Box 2732, Beijing, 100080, China

c Institute of Chemical Engineering and Metallurgy, China Nuclear Industry General Co., Beijing, 101149, China

Abstract

The ion exchange behaviour of Au and Pt on a new China-made macropore anion resin was studied using radiotracer technique. The conditions for adsorption and desorption, such as acidity, flow-rate and cocentration of desorpting agents were studied carefully. Using the established method, the contents of Au and Pt in two Chinese ultrabasic SRMs were determined by ICP-MS and the results found in good agreement with the reference values.

Keywords: Radiotracer technique Au and Pt Adsorption and desorption HHY-10A macropore resin ICP-MS

1. Introduction

With an average concentration of $5 \times 10^{-9[1]}$ in the earth crust, platinum belongs to the category of the most rare naturally occurring elements. The omnipresent concentration of platinum in environmental and biological samples is estimated to lie in the 10^{-12} region, or even lower. There are only very few reliable data available about it, simply for the reason that no analytical procedures with sufficient detection sensitivity are yet existing.

Since the introduction of automobile exhaust catalytic cleaning, platinum has become a new environmental pollutant^[2]. The concentration of Pt in ocean water has been reported to be higher than that existing in fresh water^[3]. Thus the concentration of Pt in ocean orgnisms is likely very high. Perhaps more and more intake of seafood will lead to an unexpected effect. This potential new impact to human health has initiated research to develop analytical methods

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