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Studies on Lead Pollution: Atomic Absorption Spectrophotometric determination of lead in hair and teeth samples

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A simple, rapid and reliable method for the determination of lead in hair and teeth samples has been described. The method incorporates digestion of the samples by nitric acid followed by atomic absorption spectrophotometric determination of lead using 283.3 nm wavelength.

KEY WORDS: Lead, atomic absorption spectroscopy, hair, teeth.

INTRODUCTION

During recent years attention has been focused on the accumulation of trace elements in the human body, caused by the increasing environmental pollution.¹ The universal interest in the trace elements has stimulated a number of studies of their concentration and distribution in human body, with the purpose of establishing the normal values and to detect illness, occupational disease and toxic effects. Teeth and hair samples are useful indicators of past exposure. These are suitable biopsy materials due to their physical stability.²

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The proposed method would be of great advantage due to its speed, economy and simplicity.

EXPERIMENTAL

Equipment

A Pye Unicam SP 2900 atomic absorption spectrophotometer equipped with a lead hollow cathode lamp was employed for atomic absorption measurements. Graduated apparatus of standard calibration were used for measurements. Contamination from glassware, stoppers and pipette tips were scrupulously prevented. All glassware was washed with nitric acid (5% v/v), new glassware was allowed to stand for several hours in acid. After three rinses with deionized water and three rinses with further purified water, the glassware was stored under dust free conditions.

Reagents

All reagents used were of analytical grade of BDH or SM. Triple glass distilled water was used for solution preparation and other purposes.

Stock lead solution

1000 ppm, 1.6000 g lead nitrate was dissolved in 5 ml of 5 M nitric acid and made up to 1 litre. Working standards were prepared by diluting this solution.

Sampling

Human hair samples were collected and stored in clean paper bags. The sample was collected from one person on one cutting date. Human teeth samples were obtained from one of the local dental clinics. The teeth samples were caries free and were extracted for orthodontic reasons.

Sample preparation

Hair samples were washed for about 2 h in diethyl ether in a soxhlet

extractor and allowed to dry in dust free and lead free air. Teeth samples were rinsed with distilled water and then kept in a 30% solution of hydrogen peroxide for about $\frac{1}{2}$ hour. Organic materials such as blood etc. were cleaned carefully by scrapping and brushing. The teeth were dried at 100°C for about 2 h and finally, crushed manually in an agate mortar.

ANALYTICAL PROCEDURE

An accurately weighed (0.1–0.4 g) tooth powder or hair sample was transferred to 150 cm³ conical flask. It was ensured that the sample was thoroughly deposited on the base of the flask and none was left in the neck. 2–5 ml nitric acid were added to each flask. The flasks were placed on a hot plate with intermittent shaking. The solution was heated till dryness, the residue dissolved in 1–2 ml of nitric acid and 2–4 ml water were added. It was heated until a clear solution was obtained. The clear solution was transferred to a 5, 10 or 25 ml measuring flask depending upon the lead level being determined. A final volume of 10 ml is generally satisfactory. Final measurements of lead contents were made by atomic absorption spectrophotometer. Instrumental conditions were set up as recommended by manufacturer's manuals. Normal precautions for trace analysis were taken throughout.

RESULTS AND DISCUSSION

Method development

The decomposition of biological materials presents a special problem in securing complete sample decomposition and avoiding loss of element of interest. Generally, a simple and rapid method involving the use of small amounts of reagents for keeping the blank values low is preferred for trace analysis. The method proposed here, breaks down the organic matrix rapidly keeping the minimum contamination through reagents. The method involves dissolution of hair and tooth samples rapidly by hot nitric acid. It is noteworthy that the initial temperature should be low to prevent the violent boiling of the nitric acid. A ramp heating step was used at 60°C for 7–10 min followed by 140°C until complete dissolution.

Choice of decomposition procedure

The decomposition procedure is one of the most important steps as the quantitative results are influenced by inadequacy of decomposition steps. Heavy metals are generally acid extracted from biological materials by digestion with several different acid mixtures. The most popular combinations for digestion of biological materials are nitric-sulphuric-perchloric, nitric-perchloric and nitric-sulphuric acid mixtures.³ However, a simple and safe method is desirable for matrix analysis and it is preferable to obtain a procedure that avoids the use of troublesome and possibly hazardous reagents. The use of perchloric acid for routine digestion method is unattractive because of the explosion hazards.⁴ Attramadal and Jonsen⁵ employed hydrochloric acid for the digestion of tooth materials. With hydrochloric acid coloured residues were obtained indicating the incomplete digestion of the sample.⁶ The decomposition procedure outlined here, is simple, economical, safe and provides an effective means of determination in routine monitoring of a large number of samples and involves the use of only small amount of reagent to keep the blank value low.

Hair analysis

The analysis of hair has been done in toxicology, physiology, metabolism, pollution control and forensic studies.⁷ Concentration of lead in hair as a result of occupational exposure has been studied by various groups.^{8,9} Hasegawa *et al.*¹⁰ correlated concentration of lead in hair with the incidence of lead poisoning. Clarke *et al.*¹¹ described the method for preparation of hair for lead analysis.

Hair samples were successfully digested and analysed at least levels from 1 μg to 10 μg . The method for digestion was as described earlier. As atomic absorption methods are unable to give information about the normal lead content of human hair, standard lead solution was added to the hair samples and the procedure was applied. The recovery of added lead was satisfactory (Table I).

Teeth analysis

The main component of teeth is hydroxyapatite, minor constituents are proteins, amino acids and other organic compounds. Teeth

TABLE I
Determination of lead in hair samples by the
proposed method.

Lead added (μg)	Lead found ^a (μg)	Relative error (%)
1.0	0.93	-7.0
2.0	2.11	+5.5
3.0	3.15	+5.0
4.0	3.82	-4.5
5.0	5.35	+7.0
6.0	5.70	-5.0
8.0	8.16	+2.0
10.0	9.92	-0.8

Weight of the hair sample 0.1 g;

^aAverage of three analyses of sample; Hair samples with addition of considerable amounts of lead were analysed.

samples were also successfully digested by the proposed method. The results obtained in the analysis of teeth samples, presented in Table II are in excellent agreement with their actual values (i.e., standard added concentration).

This establishes the reliability and accuracy of the proposed method. The method is particularly suitable for routine work where a simple, rapid and accurate method is required. It is appealing to

TABLE II
Determination of lead in teeth samples by the proposed method

Sample no.	Weight of sample (g)	Lead added (μg)	Lead found		Lead concentration ($\mu\text{g/g}$)
			Total (μg)	in teeth sample (μg)	
1	0.2046	nil	1.10	1.10	5.38
2	0.4012	5.0	7.10	2.10	5.23
3	0.2218	5.0	6.15	1.15	5.18
4	0.2476	5.0	6.30	1.30	5.25
5	0.2842	5.0	6.40	1.40	4.93
6	0.4212	5.0	7.10	2.10	4.98
7	0.2980	5.0	6.50	1.50	5.03

the small laboratory since it allows the digestion of a large number of samples in a small working area.

Blank determination

The blank represents the total accumulation of metal from all steps in analytical procedure. In this work, the blank values were determined by carrying an aliquot of nitric acid through the whole analytical procedure. The maximum blank in the sample was between 5 and 10 ng. It is noteworthy that all experiments were carried out in an ordinary laboratory bench and somewhat high lead value in total blank was probably due to contamination from the laboratory air.

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