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# Determination of trace elements in medicinal activated charcoal using slurry sampling electrothermal vaporization inductively coupled plasma mass spectrometry with low vaporization temperature

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## ARTICLE INFO

## Article history:

Received 7 June 2014

Received in revised form

10 August 2014

Accepted 12 August 2014

Available online 22 August 2014

## Keywords:

Inductively coupled plasma mass spectrometry

Ultrasonic slurry sampling

Electrothermal vaporization

Medicinal activated charcoal

Multielement determination

## ABSTRACT

The determination of Cd, Sb, Te, Hg, Tl and Pb in medicinal activated charcoal by ultrasonic slurry sampling electrothermal vaporization inductively coupled plasma mass spectrometry (USS-ETV-ICP-MS) was described. EDTA was used as the modifier to enhance the volatility of elements studied. The influences of instrument operating conditions and slurry preparation on the ion signals were studied. A relatively low vaporization temperature of 1000 °C was used, which separated the analyte from the major matrix components that improved ion signals. The method has been applied to determine Cd, Sb, Te, Hg, Tl and Pb in an NIST SRM 1633b Coal Fly Ash reference material and three brands of medicinal activated charcoal capsules using isotope dilution and standard addition calibration methods. The concentrations that are in ng g<sup>-1</sup> levels were in good agreement between different calibration methods. The precision between sample replicates was better than 7% with USS-ETV-ICP-MS technique. The method detection limit estimated from standard addition curves was 0.4, 0.3, 0.3, 0.3, 0.04 and 0.9 ng g<sup>-1</sup> for Cd, Sb, Te, Hg, Tl and Pb, respectively, in original medicinal activated charcoal.

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## 1. Introduction

Inductively coupled plasma mass spectrometry (ICP-MS) is a powerful technique for trace multielement and isotopic analysis. It has been applied to a wide range of samples. The majority of analyses by ICP-MS are carried out on solutions using a conventional pneumatic nebulizer. Hence, solid samples need sample preparation prior to analysis.

The activated charcoal is a difficult to dissolve sample. The medicinal activated charcoal is used as medicine for many purposes, for instance to release toxic effects of organic and inorganic pollutants [1,2]. However, environmental pollution and the contamination during processing can cause the activated charcoal to contain toxic elements which on consumption will produce detrimental effects on human health. Thus, the determination of trace elements in medicinal activated charcoal is essential in order to know the level of toxicity. ICP-MS has been successfully applied to the determination of trace and minor elements in carbon samples

[3,4]. Most of the analyses need tedious sample dissolution and pretreatment steps [3]. Furthermore some of the volatile species might be lost during sample pretreatment. However, direct solid analysis is a simpler alternative for the graphite powder analysis. Solid sampling graphite furnace atomic absorption spectrometry has been successfully applied for the determination of trace elements in graphite samples [5,6]. ETV method has been used for the sample introduction of difficult to dissolve samples such as ceramic alumina, silicon nitride etc. [7,8]. The ETV-ICP-MS method has the advantages of multielemental capability and better detection power over the GFAAS method. Ultrasonic slurry sampling electrothermal vaporization is one of the methods for solid sample introduction in ICP-MS, which has been successfully applied to determine trace elements in various powder samples [9–13]. Compared to traditional sample preparation methods such as acid digestion and dry ashing, slurry sampling offers several benefits including reduced sample preparation time, less possibility of sample contamination and decreased possibility of analyte loss prior to analysis. Moreover, ETV sample introduction method has been employed to eliminate most of the oxide and hydroxide ion interferences in ICP-MS analysis [14,15]. In ETV-ICP-MS analysis, in order to alleviate non-spectroscopic interference, a relatively low vaporization temperature could be used that separates volatile

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analytes from major matrix components. This strategy has been applied to the determination of various trace elements in several previous studies [15,16]. Addition of certain organic compounds promote atomization of the analyte prior to vaporization of the bulk matrix, thus facilitating temporal separation of the analyte and the matrix and leading to less interference effects of sample matrix. For example, EDTA was used as the chemical modifier to improve the signals of Cd, Hg and Pb in previous ETV-ICP-MS application [15].

In the present work, ultrasonic slurry sampling electrothermal vaporization inductively coupled plasma mass spectrometry (USS-ETV-ICP-MS) is proposed as an alternative technique for the simultaneous determination of Cd, Sb, Te, Hg, Tl and Pb in medicinal activated charcoal samples. The optimization of the USS-ETV-ICP-MS technique and its analytical figures of merit, as well as its applications for the determination of Cd, Sb, Te, Hg, Tl and Pb in medicinal activated charcoal capsules, are described in this paper.

## 2. Experimental

### 2.1. Apparatus and conditions

A Perkin-Elmer SCIEX (Concord, Ontario, Canada) ELAN 6100 DRCII ICP-MS equipped with HGA-600MS electrothermal vaporizer was used for studies and analysis. Pyrolytic coated graphite tubes with same material platforms were used throughout. The transfer line consisted of 90-cm-long, 6-mm-i.d. PTFE tubing. The sample introduction system included a Model AS-60 autosampler equipped with an USS-100 ultrasonic slurry sampler. Teflon autosampler cups were used throughout. The USS-100 was set at 10 W, and a 5-s mix time was used to mix slurries before injection of 20- $\mu$ L sample aliquots for analysis.

The ICP conditions were selected to maximize Mg, In and U ion signals while a solution containing 1 ng mL<sup>-1</sup> of these elements in 1% HNO<sub>3</sub> was continuously introduced with an ARIDUS membrane desolvation sample introduction system (Cetac, Omaha, NB, USA) to produce dry aerosol. The ICP-MS and ETV operating conditions used throughout this work are summarized in Tables 1 and 2, respectively. Integrated analyte signals (peak area) were measured throughout at transient peak hopping mode.

### 2.2. Reagents

The reagents and standard solutions were procured from following sources. Suprapur HNO<sub>3</sub> was from Merck (Darmstadt, Germany);

**Table 2**  
HGA-600MS temperature programming<sup>a</sup>.

Step	Temperature (°C)	Ramp time (s)	Hold time (s)	Gas flow rate (mL min <sup>-1</sup> )	Read
Dry1	80	5	10	300	-
Dry2	100	5	10	300	-
Pyrolysis	150	10	10	300	-
Cooling	20	1	5	300	-
Vaporization	1000	0	4	0	On
Cooling	20	5	5	300	-
Condition	2500	1	5	300	-
Cooling	20	5	10	300	-

<sup>a</sup> Slurry volume: 20  $\mu$ L.

Triton-X 100 and ammonium pyrrolidinecarbodithioate (APDC) were from Fluka (Buchs, Switzerland); 8-Hydroxyquinoline (8-HQ) was from JANSSEN CHIMICA (Geel, Belgium); Disodium ethylenediamine tetraacetic acid (EDTA) was from Fisher (Fair Lawn, NJ, USA); Cd, Te and Hg element standard solutions were from Merck; Sb and Mo element standard solutions were from Fisher; Tl and Pb element standard solutions were from Accustandard (New Haven, USA); W element standard solution was from Spex (Edison, NJ, USA). Enriched isotopes were procured from the following sources, <sup>111</sup>CdO (96.4%) was from the Oak Ridge National Laboratory (Oak Ridge, TN, USA), <sup>123</sup>Sb (97.1%), <sup>125</sup>TeO (92.6%), <sup>201</sup>HgO (80.5%) and <sup>203</sup>Tl<sub>2</sub>O (98.4%) were from Cambridge Isotope Laboratories (Andover, MA, USA) and <sup>206</sup>Pb(NO<sub>3</sub>)<sub>2</sub> (99.7%) was from Merck (Darmstadt, Germany). Activated charcoal was from Sigma-Aldrich (St. Louis, Mo, USA).

### 2.3. Slurries preparation

The applicability of the method to real samples was demonstrated by the analysis of Cd, Sb, Te, Hg, Tl and Pb in three brands of medicinal activated charcoal capsules and an NIST SRM 1633b Coal Fly Ash reference material (National Institute of Standards and Technology, USA).

The analytes in various medicinal activated charcoal samples were determined by the isotope dilution method and standard addition method. The slurries were prepared using the following procedure. A 1-g portion of the powder material was transferred into a 10 mL flask and diluted to mark with pure water. 1-mL aliquot of the stock slurry solution was transferred to 10 mL flasks. Suitable amount of EDTA was added to make the final solution containing 1% m/v charcoal powder and 1% m/v EDTA. Since the concentrations of Pb and several other elements in SRM 1633b Coal Fly Ash were quite high, to determine the elements studied simultaneously, an 0.01% m/v coal fly ash slurry was prepared for the analysis. After addition of suitable amount of enriched isotope or various amounts of Cd, Sb, Te, Hg, Tl and Pb element standard solutions (0, 0.5, 1, 2, 4 and 8 ng mL<sup>-1</sup> in the final solutions); these slurries were made up to the mark with pure water. To correct any analyte contamination in the reagent used, a blank solution containing 1% m/v EDTA was also prepared. There was no significant amount of the analyte in the blank. The concentrations of analytes in the samples were calculated by the equation described in a previous paper [17] and/or from the standard addition calibration curves. To study the effects of ETV conditions and slurry preparations on the ion signals, a Sigma-Aldrich activated charcoal slurry sample prepared using the procedure described above and spiked with 0.5 ng mL<sup>-1</sup> of Cd and Te was used for studies. Spiking of other analytes of interest was not carried out as they are present above detectable levels in the sample.

**Table 1**  
Equipment and operating conditions.

ICP-MS instrument	Perkin-Elmer Elan 6100 DRCII
Plasma conditions	
RF power (W)	1300
Outer argon flow rate (L min <sup>-1</sup> )	15
Auxiliary argon flow rate (L min <sup>-1</sup> )	1.13
Carrier argon flow rate (L min <sup>-1</sup> )	0.95
Data acquisition	
Dwell time (ms)	10
Scan mode	Peak-hopping
Sweeps/reading	2
Reading/replicate	130
Signal measurement	Integrated
Isotopes monitored	<sup>111</sup> Cd, <sup>113</sup> Cd, <sup>121</sup> Sb, <sup>123</sup> Sb, <sup>125</sup> Te, <sup>126</sup> Te, <sup>201</sup> Hg, <sup>202</sup> Hg, <sup>203</sup> Tl, <sup>205</sup> Tl, <sup>206</sup> Pb, <sup>208</sup> Pb

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