# Determination of Trace Elements in Hair by ICP-MS

## Application Note
## Clinical

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**Abstract**

Human hair samples were analyzed using an 4500 ICP-MS system. Sample preparation was by microwave digestion. A Japanese standard reference material (NIES No.5 Human Hair) was also analyzed. For 11 elements including Hg, excellent agreement with the certified values were obtained, and the sensitivity of the 4500 ICP-MS enabled a very short acquisition time (24 seconds/sample) to be used. A recovery test was performed to check the effect on the sample preparation and measurement methodology. The pre-digestion spike gave recoveries in the range of 93.7 - 109%.

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## Introduction

The determination of toxic elements such as Hg, Pb and Cd in humans has been a critical issue in the field of clinical chemistry from the toxicology viewpoint. In addition, since recent biomedical research has shown that some elements at trace level have specific functions in the biochemistry of living organisms, the determination of trace element concentrations in human being has also become a major issue in the field of nutritional study. As a result, the analysis of toxic elements and also many trace elements in biological samples is required. The analyte concentration range is large, ranging from the trace levels normally found in the body, to the high levels resulting from industrial exposure. Since medical treatment regimes for hospital patients depend on the analytical results reported, the analysis of biomedical samples is critical. Therefore, the need for fast and reliable analytical methods and instrumentation is paramount.

4500 ICP-MS is a fast, sensitive analytical tool with the capability of multielement analysis. In addition, sample consumption is very low (in this case <1 mL of sample solution). These characteristics represent major advantages in a field where human life is directly involved.

Hair is a good index of exposure to elements and easily accessed. The levels of trace elements in hair also depend on race, age and gender of the patient. This application note describes the determination of several trace elements in hair by the 4500 ICP-MS.

## Sample Preparation

Samples analyzed were comprised of a Japanese standard reference material, NIES No.5 Human Hair, and a bulk sample of Japanese hair randomly obtained at a Japanese hairdressing salon. The samples were digested by weighing 0.1 g of each sample into a microwave digestion vessel with 5 mL of nitric acid (Tama Chemicals, Tokyo, Japan). They were placed in a microwave sample preparation system, MDS-2000 (CEM . Matthews, NC), and digested. The conditions for microwave system are shown in Table 1.

After digestion, the samples were diluted to 50 mL with DI water (Nihon Millipore, Tokyo, Japan).

Parameters for 4500 ICP-MS are given in Table 2.

Prior to quantitative measurement, a fast qualitative measurement for NIES No.5 was performed to select internal standard elements used for analysis. The spectrum obtained for NIES No.5 is shown in Fig. 1. Co, In and Tl were selected as the internal standard elements since these elements were not present in the sample.

| Stage | 1 | 2 | 3 | 4 |
|---|---|---|---|---|
| Power (W) | 378 | 378 | 378 | 378 |
| Pressure (psi) | 20 | 40 | 85 | 130 |
| Hold (min) | 5 | 5 | 5 | 5 |

**Table 1**
**Conditions for Microwave Digester**

| | |
|---|---|
| Plasma gas flow rate | 15.0 L/min |
| Aux. gas flow rate | 1.0 L/min |
| Carrier gas flow rate | 1.17 L/min |
| RF Power | 1300 W |
| Nebulizer | PEEK, Babington - type |
| Spray chamber | Glass, double pass |
| Spray chamber temp | 1°C |
| ICP torch injector | Quartz, 2.5 mm |
| Sample uptake rate | 0.4 mL/min |
| Sampler cone | Nickel |
| Skimmer cone | Nickel |
| Sampling depth | 8 mm |
| Points/mass | 3 |
| Integration time/mass | 0.3 sec |
| Total acquisition time/replicate | 8 sec |
| Replicates | 3 |
| Total acquisition time/sample | 24 sec |

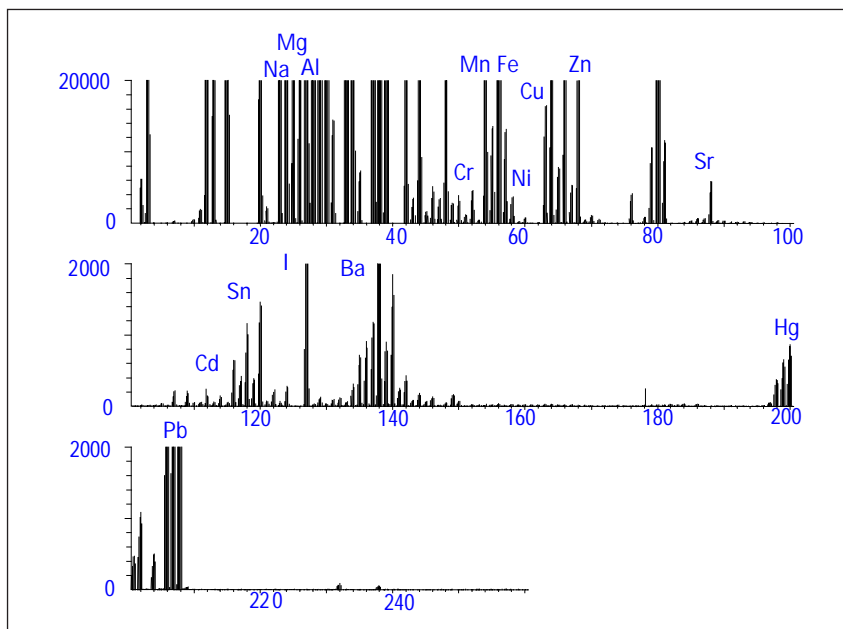**Table 2.**
**4500 ICP-MS Operating Parameters**

**Fig.1**
**Qualitative spectrum of hair (NIES No.5)**

## Results

Quantitative results and the certified values for NIES No.5 are given in Table 3. The 11 analytes values obtained were in excellent agreement for all the elements that had certified values. (There is no certified value for As.) Since microwave digestion was used, even the results for volatile elements such as Hg and Zn show good agreement. Using normal open vessel digestion with nitric acid, recoveries for these elements would be poor due to losses by vaporization during the digestion stage. Also of note was the good precision obtained on the analyzed values.

The three sigma detection limits for the analytes measured were calculated using the data obtained from the reagent blank (which had also been through the digestion step), and are also given in Table 3. The detection limits are expressed in the hair as received,

and demonstrate the excellent sensitivity of the 4500 ICP-MS, even though the total sample measurement time was only 21 sec. for three replicates.

A recovery test was performed using a bulk sample of Japanese hair. A multielement spike was added to portions of the sample both before and after digestion, to assess the validity of the digestion procedure.

The results are given in Table 4. As can be seen, both sets of results were excellent: the recoveries of the pre-digest spiked sample were 93.7 - 109%, and the recoveries of the post-digest spiked sample were 92.9 - 105%.

These results show there was no contamination or loss of analytes during sample preparation and also that the presence of sample matrix has no effect on the preparation and measurement methodology.

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| Element | m/z | Conc. (ug/g) | | D.L. |
| | | Measured | Certified | (ug/g) |
|---|---|---|---|---|
| Al | 27 | 220+/-6 | 240* | 0.003 |
| Cr | 52 | 1.72+/-0.07 | 1.4+/-0.2 | 0.004 |
| Mn | 55 | 5.47+/-0.13 | 5.2+/-0.3 | 0.001 |
| Fe | 56 | 219+/-5 | 225+/-9 | 0.9 |
| Ni | 60 | 1.87+/-0.06 | 1.8+/-0.1 | 0.004 |
| Cu | 65 | 16.7+/-0.6 | 16.3+/-1.2 | 0.002 |
| Zn | 66 | 171+/-4 | 169+/-10 | 0.004 |
| As | 75 | 0.18+/-0.02 | - | 0.02 |
| Se | 82 | 2.4+/-0.3 | 1.4* | 0.004 |
| Cd | 111 | 0.21+/-0.03 | 0.2+/-0.03 | 0.0002 |
| Hg | 202 | 4.52+/-0.15 | 4.4+/-0.4 | 0.003 |
| Pb | 208 | 5.98+/-0.11 | 6.0* | 0.0007 |

* : Not certified, information value only          - : Not certified

**Table 3**
**Quantitative results : NIES No.5**

| Element | m/z | Conc. in hair (ug/g) | Spiked volume (ug/g) | Recovery (%) | |
| | | | | pre-spiked | post-spiked |
|---|---|---|---|---|---|
| Al | 27 | 20.5 | 92.5 | 98.0 | 96.3 |
| Cr | 52 | 1.27 | 92.5 | 95.2 | 93.5 |
| Mn | 55 | 0.33 | 92.5 | 95.3 | 95.6 |
| Fe | 56 | 14.6 | 92.5 | 107 | 103 |
| Ni | 60 | 6.93 | 92.5 | 101 | 99.5 |
| Cu | 65 | 58.6 | 92.5 | 105 | 101 |
| Zn | 66 | 110 | 92.5 | 97.4 | 99.0 |
| As | 75 | 0.08 | 92.5 | 96.8 | 96.0 |
| Se | 82 | 0.70 | 92.5 | 109 | 105 |
| Cd | 111 | 0.023 | 92.5 | 103 | 103 |
| Hg | 202 | 1.18 | 9.25 | 99.1 | 104 |
| Pb | 208 | 2.54 | 92.5 | 93.7 | 92.9 |

**Table 4**
**Recovery test**

## Conclusion

The 4500 ICP-MS has been shown to give excellent results for the determination of trace level metals in hair.  In combination with microwave digestion, precise, accurate determinations for volatile elements including As, Hg and Zn, can be obtained.  Since the 4500 ICP-MS has excellent sensitivity, multielement data can be obtained with very short acquisition times.  Three replicates for 12 elements were performed in only 24 seconds, far outperforming the sample throughput available from even the latest multielement GFAA systems, and delivering the high sample throughput needed by busy clinical labs with fast sample turnaround demands.

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