

大気圧サンプリング法-レーザーアブレーション ICP 質量分析法による固体試料の直接微量元素分析法の開発 Development of atmospheric pressure sampling device for direct elemental analysis using LA-ICP-Mass Spectrometry

平田 岳史^{1*}, 坂田周平¹, 西口講平², 宇谷啓介²

Takafumi Hirata^{1*}, Shuhei Sakata¹, Kohei Nishiguchi², Keisuke Utani²

¹ 京都大学大学院理学研究科地球惑星科学専攻, ² 株式会社ジェイ・サイエンス・ラボ

¹Department of Earth and Planetary Sciences, Kyoto University, ²J-Science Laboratory Co. Ltd.

Combination of laser ablation sample introduction technique and ICP-mass spectrometry (LA-ICPMS) has been widely used for element and isotopic analyses for various geochemical and biochemical samples [1]. The laser ablation utilising shorter wavelength or shorter pulse duration can minimise the elemental fractionation during both the laser ablation and ionisation processes, and the resulting precision for elemental and isotopic ratio measurements has been successfully improved. One of the great advantages to use the LA-ICPMS technique is that the analysis can be made under the atmospheric pressure, and therefore, this technique can be applied for the elemental analysis of the wet samples including cell or tissue. However, it should be noted that the sample was located inside the cell to improve the transmission efficiency of the sample aerosol from the sample to ICP ion source. This suggests that the elemental analysis could not be achieved for the large-sized sample or continuously delivering samples. To overcome this, we have developed direct sampling technique using the laser ablation under the atmospheric pressure conditions using a gas exchange device (GED) [2,3]. Laser ablation was carried out under the atmospheric air, and the laser induced sample aerosols were extracted and collected with air using a diaphragm pump. The carrier gas was converted from the air (N₂ and O₂) to Ar through the semipermeable filter, and the sample aerosol was introduced into the ICP in the same manner with the conventional laser ablation setup. To minimize the pulsation of the gas flow, which causes serious reduction in the analytical sensitivity of ICPMS, the glass capillary tubing (i.d. 0.4 mm) was adopted between the diaphragm pump and gas exchange tubing.

In this study, we have monitored the signal intensities of ²⁰⁸Pb, ²³²Th, ²³⁸U in order to test the collection yield of the sample aerosols. Signal intensities of ²³²Th¹⁶O and ²³⁸U¹⁶O were also monitored to test the production ratio of oxide signals, which could reflect the amount of residual O₂ in Ar carrier gas through the gas exchange process. With the simple extraction setup using a cut-off tubing and diaphragm pump, the resulting signal intensities for Pb, Th and U were almost one order of magnitude lower than those achieved by the conventional laser ablation setup (i.e., cell configuration). This indicates that the laser induced sample particles could not be collected by the simple extraction using the cut-off tubing. Although He was flushed onto the ablation pit toward the collection tubing (push-pull setup) in order to improve the collection efficiency, the signal intensities for Pb, Th and U became rather lower than the simple extraction setup. This could be due to deterioration in the evacuation rate of the diaphragm pump because of lower viscosity of He. In contrast, the production ratios for ThO⁺/U⁺ and UO⁺/O⁺ became lower when the higher flow rate of He flushing was employed. The lowering of the oxide production could be explained by the possible dilution of O₂ in air by He flushing. Principles and advantage of the present laser sampling technique and versatility in elemental mapping analysis will be demonstrated in this presentation.

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