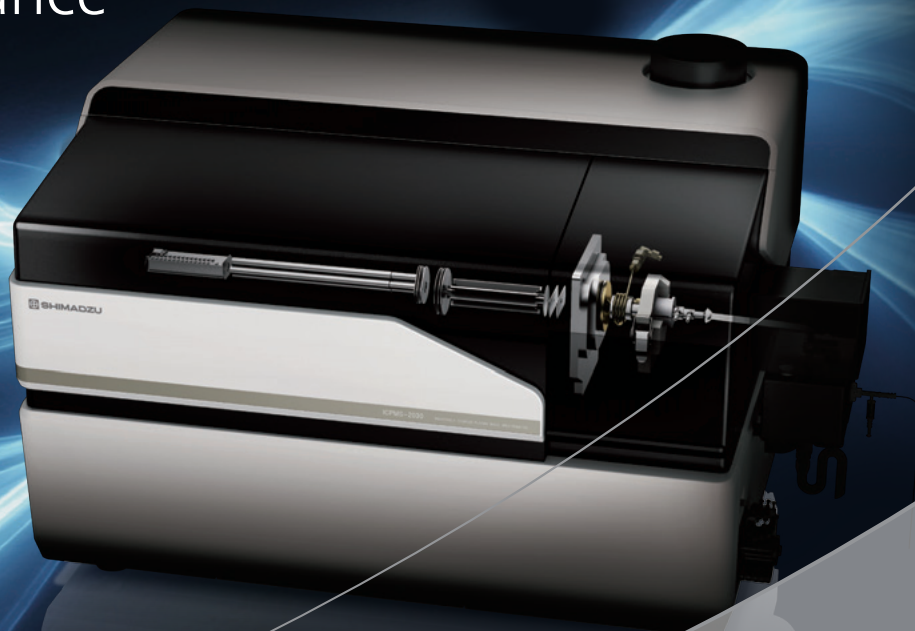


Accelerating Reliable Performance



Shimadzu ICPMS-2030 Inductively Coupled Plasma Mass Spectrometer

Quantitation of Arsenic Species in White Rice and Brown Rice Using LC-ICP-MS System

► Rice is one of the agricultural products that incorporates a relatively significant amount of arsenic in which the ratio of inorganic to organic arsenic is known to be high. Both inorganic and organic arsenic species are known for their potential for harm, however, inorganic arsenic is known to be more toxic than organic arsenic. As such, the Codex Alimentarius Commission held in July 2014 determined 0.2 mg/kg to be the maximum concentration of inorganic arsenic in processed rice.

An LC-ICP-MS system which connects an inductively coupled plasma mass spectrometer (ICP-MS) in-line with a high-performance liquid chromatography (HPLC) system allows high sensitivity and high accuracy measurement of arsenic species.

This report demonstrates the quantitation of inorganic arsenic species [As(III) and As(V)] and dimethylarsinic acid in white rice and brown rice that was accomplished by connecting the Shimadzu ICPMS-2030 in-line with a HPLC system.

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Samples

NMIJ White Rice : Certified Standard Substance (NMIJ CRM 7503-a)

NMIJ Brown Rice : Certified Standard Substance (NMIJ CRM 7532-a)

Sample Pretreatment (Extraction of Arsenic)

~ 0.5 g of sample was weighed into a 14 mL of centrifuge tube. 2 mL of 0.15M nitric acid solution was added to the centrifuge tube. Then the solution in the centrifuge tube was stirred. After the solution was heated in a heat block for 6 hours at 100 °C, pure water was added to the centrifuge tube to make 10 g of the solution. The solution was centrifuged at 3000 rpm for 20

minutes. The supernatant was filtered using a micro-membrane filter with 0.45 µm pore size. The filtrate was diluted twice with a mobile phase to make the measurement solution. The same pretreatment procedures were repeated three times for white and brown rice to obtain three measurement solutions for each.

Measurement and Results

The samples were measured with the ICPMS-2030 connected in-line with a HPLC system. An internal standard was added to samples and standards using a T-joint in the sample tubing. The quantitation of inorganic arsenic species [As(III) and As(V)] and dimethylarsinic acid (DMAA) was achieved by the calibration curve method (internal standard method). The measurement results are shown in Table 1. The concentration of inorganic arsenic species is

shown as the sum of As(III) and As(V). The results show that the arsenic concentrations of measured samples are less than the NMIJ certified values. For brown rice, six solutions were made according to the same pretreatment procedures were measured. The results show good reproducibility with RSD (relative standard deviation) of 1.1% to 2.6%. Chromatograms of inorganic arsenic species and dimethylarsinic acid are shown in Figure 1.

| LC: Measurement Conditions | |
|----------------------------|--|
| Column | Shiseido Capcell Pak C18 MG S5 |
| Mobile Phase | 10 mM Sodium 1-butanefulfonate 4 mM Tetramethylammonium hydroxide 4 mM Malonic acid (pH = 3) 0.05% Methanol |
| Flow Rate | 0.75 mL/min |
| Oven Temp. | 40 °C |
| Injection Volume | 20 µL |

| ICPMS-2030: Measurement Conditions | |
|------------------------------------|--------------------------------|
| RF Power | 1.2 kW |
| Plasma Gas Flow Rate | 8.0 L/min |
| Aux. Gas Flow Rate | 1.10 L/min |
| Carrier Gas Flow Rate | 0.60 L/min |
| Sample Introduction | Nebulizer 07 |
| Spray Chamber | Electronically-cooled cyclonic |
| Torch | Shimadzu mini-torch |

Table 1: Measurement Results for Arsenic Species

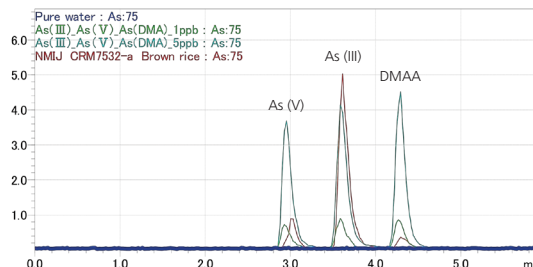
| Content in Sample (mg/kg) | | As (III) | As (V) | DMAA |
|---------------------------|------------------------------------|------------------------|--------|--------|
| White Rice | Quantitation Value (n=3) | 0.0649 | 0.0203 | 0.0138 |
| | Total of Inorganic Arsenic Species | 0.0852 | | - |
| | NMIJ Certified Value | 0.0841 As (III)+As (V) | | 0.0133 |
| | Expanded Uncertainty | 0.003 | | 0.0009 |
| Brown Rice | Quantitation Value (n=3) | 0.254 | 0.0498 | 0.0186 |
| | Total of Inorganic Arsenic Species | 0.303 | | - |
| | NMIJ Certified Value | 0.298 | | 0.0186 |
| | Expanded Uncertainty | 0.008 | | 0.0008 |
| | RSD (%) (n=6) | 1.1 | | 2.6 |

Fig. 1 Chromatograms of Arsenic Species

Green: 1 µg/L As (III), As (V) and DMAA

Blue: 5 µg/L As (III), As (V) and DMAA

Brown: Brown Rice



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