

# Iodine screening of tomato, melon, ginger, ha-shoga, Japanese ginger and chestnut by applying an analytical method for bromine using wavelength-dispersive X-ray fluorescence spectrometry

(Received October 25, 2012)

(Accepted February 14, 2013)

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## Abstract

Methyl iodide is a fumigant for which the maximum residue level (MRL) was set in September 2009. Because the usage of methyl iodide as an agrichemical resembles that of methyl bromide, it is widely used as a substitute, and the amount of residue in agricultural products is strictly controlled by the Japanese Food Sanitation Law. The residual level of bromine derived from methyl bromide is also regulated in Japan, and bromine analysis is performed for its surveillance. Therefore, if residual methyl iodide could be evaluated concomitantly with bromine analysis, the efficiency of this analysis could be improved. However, the volatility and reactivity are markedly different between bromine and methyl iodide, and simultaneous analysis is not possible. When methyl iodide is used as a soil fumigant in the United States and Australia, no MRL is set because it does not remain in agricultural products. Although residual testing of crops carried out in Japan also gives results less than the limit of quantification (LOQ) (0.01 ppm), the decomposition product iodine is detected. Furthermore, methyl iodide can be detected in chestnuts that are fumigated directly, and more iodine is detected than methyl iodide. Therefore, we investigated a method to easily screening for the presence or absence of residual methyl iodide by means of its decomposition product, iodine. We attempted to adapt the method of analyzing bromine using a wavelength dispersive X-ray fluorescence (WDXRF) spectrometer for simultaneous analysis of iodine. Iodine and bromine compounds were extracted from samples using distilled water, and extracts from sugar-rich samples were further purified using a PSA mini-column. A portion of the test solution was dried on to filter paper, and iodine and bromine were measured using a WDXRF spectrometer. When potassium iodide was added to agricultural products as iodine at 5.0  $\mu\text{g/g}$ , the recovery and coefficient of variation were 70-98% and 2.0-6.9%, respectively, and the limit of detection (LOD) and LOQ were 0.7 and 2.2  $\mu\text{g/g}$ , respectively. Because the iodine content of agricultural products is generally 0.5  $\mu\text{g/g}$  or lower, detection of iodine at a level higher than the LOD suggests that the parent compound methyl iodide was used and iodine was retained. When methyl bromide was added to agricultural products as bromine at 5.0  $\mu\text{g/g}$ , the recovery and coefficients of variation were 76-102% and 0.6-8.6%, respectively, and the LOD and LOQ were 0.4 and 1.3  $\mu\text{g/g}$ , respectively. Although it was clear that an adequate recovery rate could be obtained in recovery tests by adding 5.0  $\mu\text{g/g}$  of iodine and bromine to various agricultural products, if the MRL for methyl iodide is not set and a uniform standard (0.01 ppm) is applied, it is difficult to check for residual methyl iodide from the analysis results for iodine using this method. This method is, therefore, thought to be usable for screening methyl iodide in tomato, melon, ginger, ha-shoga, Japanese ginger (myoga), and chestnuts where the form of the residue has been clarified by crop residue testing in the past and the current reference value is set to 0.05 to 0.5 ppm.

**Keywords :** wavelength-dispersive X-ray fluorescence (WDXRF) spectrometer, methyl iodide, iodine, methyl bromide, bromine

## I Introduction

Methyl iodide is widely used as a fumigant insecticide

substituting for methyl bromide, which will be prohibited by 2015 based on the Montreal Protocol on Substances that Deplete the Ozone Layer. In Japan, the maximum residue level