

Analysis of Potassium Bromate in Bread

Potassium bromate is permitted as a food additive in bread for improving a baking effect. However, from the viewpoint of consumers' safety, it must not remain in the final product. To comply with this requirement, it is essential to verify the absence of residual potassium bromate in the final product. In Japan's Health and Welfare Ministry Notification No. 119 "Analysis of Potassium Bromate in Bread" (September 11, 1997), the post-column derivatization HPLC coupled with o-

dianisidine as a reaction reagent is adopted as an authorized analytical procedure.

This Application News presents analyses of potassium bromate in bread using the Shimadzu LC-VP Bromate Analysis System in accordance with the ministerial notification. (Applications of this system for bromate and oxidized halogen ions in drinking water are presented in Application News L246.)

■ Analysis of Standard Sample

As standard solutions of potassium bromate, 1, 10, 50 and 100ng/mL of bromate ion solutions were prepared respectively. The calibration curve was created using these solutions, and analysis was performed under

the analytical conditions specified in the ministerial notification no. 119. Fig.1 shows the chromatogram of the 10ng/mL of standard solution, and Fig.2 shows the created calibration curve.

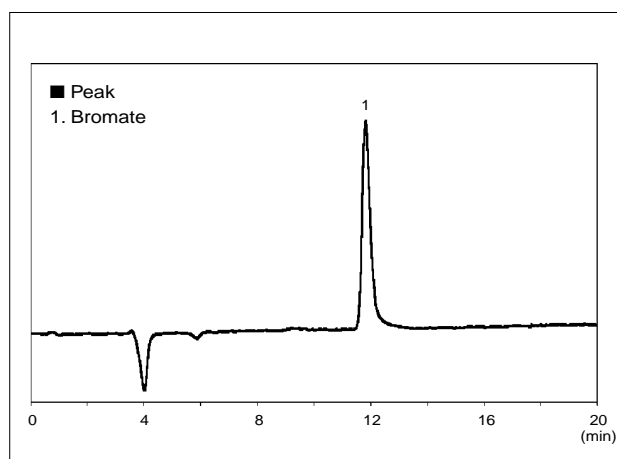


Fig.1 Analysis of Standard Sample (10ng/mL as Bromate)

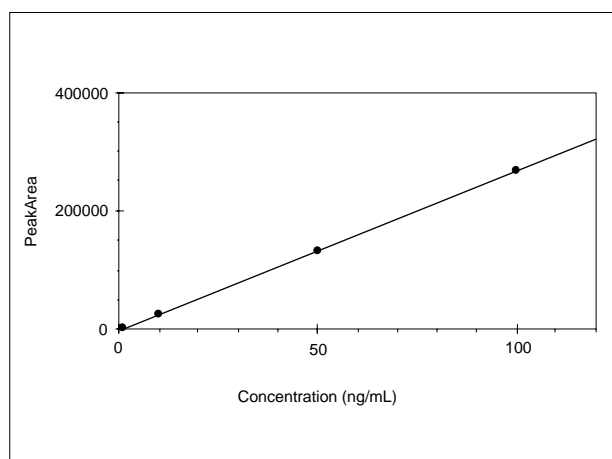


Fig.2 Calibration Curve

■ Analysis of Potassium Bromate in Bread

The lower chromatogram in Fig.3 shows the analysis of commercially available bread, performed with the pretreatment procedures indicated in Table 1 (according to above-mentioned notification no. 119). The upper chromatogram shows the analysis of the same bread, in which potassium bromate was spiked to make a content of 326 μ g of potassium bromate (equivalent to 250 μ g bromate ion) per 10g of bread sample just prior to pretreatment. The peak of bromate ion was not detected in the un-spiked bread sample. For the spiked sample, 325.4 μ g of potassium bromate (equivalent to 249.4 μ g bromate ion) per 10g of bread was determined, indicating nearly complete recovery of the spiked quantity.

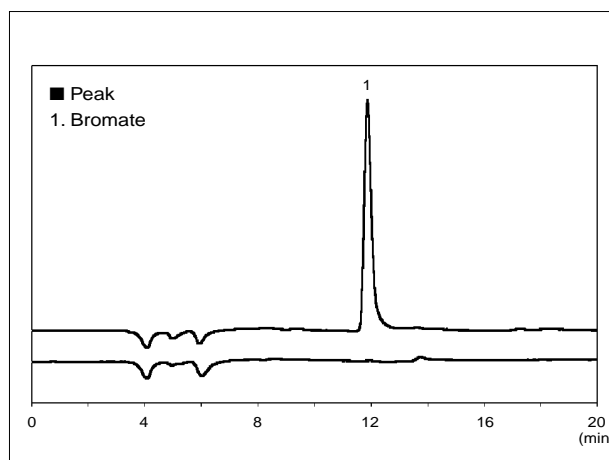


Fig.3 Analysis of Potassium Bromate in Bread
Upper: Potassium bromate-spiked sample
Lower: Un-spiked sample

Table 1 Pretreatment Procedures

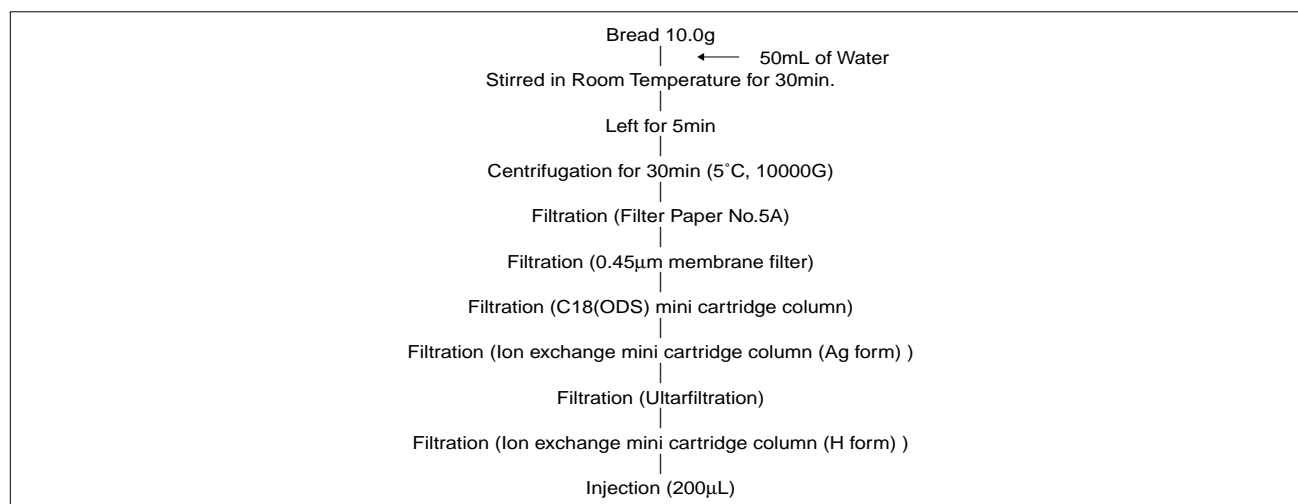


Table 2 Analytical Conditions

Instrument	: Shimadzu LC-VP Bromate Analysis System
Column	: Shim-pack VP-ODS (4.6mmi.d.×250mmL.)
Mobile Phase	: 100mL of methanol, 2.0g of acetic acid and 19g of tetrabutylammonium hydroxide were added to 700mL of Water, and pH of solution was adjusted to 6.3 - 6.5. And then, this solution was diluted to 1000 mL with Water.
Flow rate	: 1.0mL/min
Temperature	: 40°C
Injection Volume	: 200 μ L
Reaction reagent	: A ; 60mL of nitric acid (70%), 10.0g of potassium bromide was added to 700mL of Water. B ; 500mg of o-dianisidine dihydrochloride was added to 200mL of methanol. Solution A and B were mixed, and diluted to 1000 mL with Water.
Reaction unit	: Piping Kit for Bromate Analysis
Temperature	: 60°C
Detection	: Absorption (450nm)