

UV-Vis 法を用いた食品添加物公定書塩化物試験法に関する検討

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Study on the chloride limit test for Japan's specifications and standards for food additives using the UV-Vis method

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Abstract

In the general test section of Japan's Specifications and Standards for Food Additives (9th edition), the chloride limit test, a visual inspection method, is "designed to demonstrate that the content of chloride in an additive does not exceed the acceptable limit specified in the individual monograph." Because the chloride limit test is a visual inspection test, it determines the concentration of chloride that can be distinguished visually.

The visual inspection method, on the other hand, makes it challenging to distinguish small differences in chloride concentration. The visual inspection method could not distinguish between adjacent chloride standard solutions unless there was a significant difference in concentration of more than 0.1 µg/mL for 0–0.5 µg/mL chloride standard solutions, more than 0.5 µg/mL for 0.5–1 µg/mL of chloride, and more than 1 µg/mL for 1–4 µg/mL chloride standard solutions. As a result, we examined the use of a UV-visible spectrophotometer to measure the absorbance of silver chloride (UV-Vis method). After the absorbance stabilized, a UV-Vis spectrophotometer was used to measure the absorbance at 600 nm after adding silver nitrate solution to either the test or chloride standard solution (approximately one hour). The calibration curve was linear ($R^2 > 0.99$) for concentrations ranging from 0.05 to 5 µg/mL, which are difficult to distinguish visually. By spiking samples with chloride equivalent to the criteria for three sample types: glycine, sodium dihydrogen phosphate, and sodium sulfate, this method was validated. After that, the chloride was quantified using the UV-Vis method. The UV-Vis method's accuracy and precision were comparable to that of ion chromatography for the direct detection of chloride ions, with satisfactory accuracy ($\geq 92.6\%$), precision ($\leq 3.7\%$) and intra-laboratory reproducibility ($\leq 7.3\%$). These results indicate that the UV-Vis method can discriminate against small differences in chloride concentrations that are difficult to determine using visual inspection methods. As a result of this study, it was demonstrated that the UV-Vis method is a simple and effective method for accurately determining chloride concentrations in test solutions.

Keywords : 食品添加物公定書、塩化物試験法、紫外可視吸光度計

Japan's Specifications and Standards for Food Additives, chloride limit test, UV-Vis spectrometer

I 緒言

第9版食品添加物公定書(公定書)¹⁾の塩化物試験法は、食品添加物中に混在する塩化物の限度試験法であり、検液と比較液中の水溶性塩化物を硝酸銀と反応させることで生じる塩化銀の濁度を目視により比較する方法(目視法)である。塩化物規格は、食品添加物の精製度

合を知る指標として、公定書の成分規格各条の純度試験に設定されている。

塩化物を測定する方法としては、目視法以外に、同様の原理を利用し、硝酸銀溶液で滴定する方法²⁾(モール法、フィヤンス法)、チオシアン酸アンモニウム溶液で滴定する方法²⁾(フォルハルト法)、塩化銀比濁法³⁾、イオンクロマトグラフィー^{4,5)}があるが、試料中の塩化