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Validation of the testing method for the determination of dibutyltin compounds in food utensils, containers, and packaging products made from polyvinyl chloride using gas chromatograph-mass spectrometry with nitrogen as a carrier gas

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Abstract

We validated an alternative testing method for the determination of dibutyltin (DBT) compounds in food utensils, containers, and packaging products made from polyvinyl chloride using gas chromatograph-mass spectrometry with nitrogen (N_2) as a carrier gas. The retention times, mass spectra, ion intensities, and signal-to-noise-ratios (*S/N*) of a DBT derivative were compared using both helium and N_2 as the carrier gas. The retention times were almost equal under the same flow-rate condition, as were the mass spectra. In contrast, the ion intensity with the N_2 carrier gas decreased to around 3/4, and the *S/N* decreased significantly to 1/10. This might be due to the increase in background noise level. We validated the performance in terms of a limit testing method that assess its suitability by comparing the peak area values of the DBT derivative in the test solution and a standard solution at a concentration corresponding with the acceptance criteria and a quantitative testing method with N_2 carrier. All parameters corresponding to the trueness, repeatability, and reproducibility as intermediate precision, satisfied the target values in both cases, indicating that both approaches demonstrate good performance as a testing method.

Keywords : nitrogen carrier gas, GC-MS, dibutyltin compound, food utensil, food container, food packaging, validation

I Introduction

A gas chromatography (GC), such as GC-flame ionization detector (FID), GC-flame thermionic detector (FTD), GC-nitrogen phosphorus selective detector (NPD), or GC-mass spectrometer (MS) is widely used for the semivolatile or volatile chemical substances analysis. Helium (He) gas is generally used as a carrier gas for most GCs because He is easy to handle, and the range of optimum linear velocity is wide. However, He gas supplies have frequently diminished in recent years, and this problem is expected to continue in the future.

Nitrogen (N_2) and hydrogen (H_2) are well known as alternatives of He in GC analyses. Differences in GC analysis using He and N_2 or H_2 have been reported by some scientists[1, 2), *1] and also by the instrument companies, such as, Shimadzu[*2], Agilent Technologies[*3], and Perkin Elmer[*4]. According to these reports, N_2 can be handled safely, but changing the carrier gas from He to N_2 might cause a decrease in separation capability. And the sensitivity decreased significantly, especially in GC-MS analysis. Conversely, the H_2 carrier gas has demonstrated good separation and sensitivity similar to He. However, H_2 is a dangerous gas because it can easily explode; therefore, H_2 must be handled with extreme caution. Consequently, H_2 cannot be applied for GC-FID or GC with a headspace sampler.

In the Food Sanitation Act (FSA) of Japan, GC-FID, GC-FTD, GC-NPD, or GC-MS have been designated to be used

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*1 Nahoko Haneishi, Effect of changing carrier gas on the testing method using gas chromatography, Reports of Research Grant from the Ministry of Health Labour and Welfare in Japan (Grant number: H25-food-003 for Mutsuga Motoh), 2014, 95-111 (2015).

*2 https://www.shimadzu.com/an/service-support/technical-support/technical-information/alternative_carrier_gas/index.html (Last access, Dec 9, 2020).

*3 https://www.agilent.com/cs/library/slidepresentation/Public/ASTS-2013_Helium_Conservation.pdf (Last access, Dec 9, 2020).

*4 https://www.perkinelmer.com/lab-solutions/resources/docs/TCH_010288_01-HydrogenCarrierGasGC.pdf (Last access, Dec 9, 2020).