5. 固相抽出—LC-MS/MS 法による食品中の甘味料 12 種および保存料 9 種の一斉分析

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A rapid and simple method for the simultaneous determination of twelve sweeteners and nine preservatives in various foods by liquid chromatography-tandem mass spectrometry (LC-MS/MS) was developed. The sweeteners and preservatives were extracted from solid samples with 80% and 50% methanol and from liquid samples with 80% methanol, followed by Oasis WAX cartridge cleanup. The LC separation was performed on a XSelect CSH Phenyl-Hexyl column (5 m, 2.1 mm ×150 mm) with a mobile phase of 10 mmol/L acetate buffer (pH 4.0) –acetonitrile and MS detection with negative ion electrospray ionization. The quantification limits of acesulfame K (AK), alitame (AL), aspartame (ASP), cyclamic acid (CYC), neotame (NEO), saccharin Na (SAC), p-hydroxybenzoic acid methyl (PHBA-Me), p-hydroxybenzoic acid ethyl (PHBA-Et), p-hydroxybenzoic acid isopropyl (PHBA-iPr), p-hydroxybenzoic acid isobutyl (PHBA-iBu) and p-hydroxybenzoic acid butyl (PHBA-Bu) were 0.001 g/kg, those of dulcin (DU), glycyrrhizic acid (GLY), neohesperidin dihydrochalcone (NHDC), rebaudioside A (REB), stevioside (STV), sucralose (SUC) and benzoic acid (BA) were 0.005 g/kg, and those of sorbic acid (SOA) and dehydroacetic acid (DHA) were 0.02 g/kg. The mean recoveries from ten kinds of foods fortified at the levels of 0.02 and 0.2 g/kg were 70.9–119.0%, and their relative standard deviations were 0.1–11.7%.