Development of a stable isotope dilution analysis for the quantification of the Bacillus cereus toxin cereulide in foods

Abstract: An increasing number of severe food borne intoxications are caused by a highly stable depsipeptide, named cereulide, which is produced by emetic Bacillus cereus strains. As cereulide poses a health risk to humans, the development of an appropriate method for the analysis of this toxin is mandatory. Therefore, the reference material of cereulide as well as its (13)C(6)-isotopologue was prepared by means of a biosynthetic approach using a B. cereus culture, followed by a rapid but efficient downstream purification. After structure confirmation by means of liquid chromatography (LC)-time-of-flight mass spectrometry, LC-tandem mass spectrometry, and one-/two-dimensional NMR spectroscopy, a stable isotope dilution analysis (SIDA) was developed for the quantification of cereulide in foods using the (13)C(6)-cereulide as the internal standard. Validation experiments were performed, and the data were compared to the quantitative analysis using the structurally related valinomycin instead of the (13)C(6)-cereulide as an internal standard. Trueness, repeatability, and reproducibility expressed as relative standard deviation showed values \textless{}10 or \textless{}8\% for valinomycin or \textless{}8\% for (13)C(6)-cereulide, respectively. Furthermore, the MS response of the valinomycin was found to be significantly influenced by the food...
matrix, thus leading to rather low recovery rates of 91(%) from boiled rice and 80(%) from boiled rice supplemented with 10(%) sunflower oil. In comparison, the use of (13)C(6)-cereulide as an internal standard gave good recovery rates of 104 and 111(%) from both matrices, thus demonstrating the robustness and accuracy of the developed SIDA.

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