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Analytical Method for Structural Analysis of Isomaltooligosaccharides (IMOs) by GC-MS

Isomaltooligosaccharides (IMOs) are a form of oligosaccharide with a high proportion of (1,6)-linked glucose residues. Determining the composition and ratio of monosaccharides is important for understanding the structure and function of IMOs. Many of the currently available methods for determining the monosaccharide composition do not work well. For instance, they might only look at the carbohydrates that can be dissolved in water, or they might put harsh conditions on the carbohydrates, causing them to degrade. Although gas chromatography–mass spectrometry (GC-MS) is a great analytical instrument for the investigation of IMOs, it is important to prepare appropriate derivatives to ensure that the analyte is volatile so can be separated on a GC column. Methylation analysis for monosaccharide glycosidic linkage composition is a technique used to derivatize carbohydrates to make them GC-MS compatible. Methylation with GC-MS can be used to determine monosaccharide glycosidic linkage composition in oligosaccharides. The derivative process involves various reactions that result in the formation of partially methylated alditol acetates (PMAAs) from IMOs, thereby converting the monosaccharides in IMOs into volatile. Derivatization was performed by permethylating IMOs in dimethyl sulfoxide (DMSO) with sodium hydroxide and methyl iodide (CH3I). A volatile organic acid like trifluoroacetic acid (TFA) was then used to break down the glycosidic linkage. The sugar rings were then reduced with sodium borodeuteride (NaBD4) in ammonium hydroxide (NH4OH) to produce alditol, and the anomeric carbon atom was labeled with a deuterium. For GC-MS separation, the partially methylated alditols were acetylated using acetic anhydride (Ac2O) to improve the volatility. The PMAAs are then injected directly into a GC-MS for analysis. The position of the glycosidic linkage between monosaccharide residues were then determined using GC retention times (Rt) and MS fragmentation patterns.

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