Derivatization of Carbohydrates: Revised Approaches for GLC-MS Analysis

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Analysis of carbohydrates has been determined by several chromatographic methods such as TLC, HPLC, GLC and recently by CZE. The inclusion of MS coupled to GLC provides a great method better than the other techniques, giving rise to sharp chromatograms being easily to identify and quantify. Since monosaccharide and methylation analysis are essential to determine the structure of carbohydrates, GLC-MS analysis became the most employed technique. Many reagents have been used for derivatization, such as TMS, $(CH_3CO)_2O$, $(CF_3CO)_2O$ and $(CF_3CF_2CF_2CO)_2O$, and none was really better than the other, in this case the application determines the choice of the derivatizing reagent. Silylation of sugars is very the simple, being rapid and applicable for all carbohydrate classes. However silyl derivatives are instable and provide formation of many isomers, thus a second step of derivatization is necessary, of interest is the conversion up to alditol or dithiocetal silyl derivatives. The stable alditol acetates derivatives have been employed in monosaccharide composition of polysaccharides, since it generates a single peak for each sugar, differing from silylated or fluroacylatilated methods, although it is not applicable to uronic acids. Revising strategies of acid-base catalysis on the alditol acetate method, we performed the determination of neutral, amino and acidic sugars, without formation of lactone byproducts, and also, re-introduce the Me-acetates glycosides, as well as, amino acids and OH-fatty acids as Acetate-Me-Esters (AME) or Alditol-Acetates-Me-Esters (AAME) derivatives for analysis of polysaccharides, glycolipids, LPS and other glycoconjugates. GLC-MS analysis on DB-225 of GalA, ManA, GlcA, GulA and IdoA, gave a single peak for each sugar derivative and characteristic ions at 361 $[M-59]^+$, 143, 156 and 173 m/z. A similar approach were used to obtain 19 AME amino acids derivatives with controlled byproduct formation (azlactone), giving molecular ions and characteristic daughter ions of $[M-59]^+$ and $[M-43]^+$ on EI-MS, providing rapid identification of the amino acids derivatives.