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The application of mass spectrometry to elucidate the biosynthesis of long-chain fatty acid amides

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Long-chain fatty acid amides are a family of endogenous signaling lipids that have various biological functions in mammals. The general structure of a fatty acid amide is R-CO-NH-X, where R is an acyl group and X is derived from a vast variety of biogenic amines; members of this family include the N-acylethanolamines, N-acylamino acids, the N-acylarylalkylamides, the N-monoacylpolyamides, and the primary fatty acid amides. These metabolites are lowly abundant in biological samples and are often difficult to identify and quantify. We employed LC/QTOF-MS analysis to study long-chain fatty acid amide metabolism in two model systems, mouse N18TG2 neuroblastoma cells and Drosophila melanogaster. The best studied long-chain fatty acid amide is the mammalian endocannabinoid, N-arachidonoylethanolamine (anandamide). However, many of the details of non-N-acylethanolamine long-chain fatty acid amide metabolism are not known in mammals or invertebrates. Full details of long-chain fatty acid amide metabolism will lead to the discovery of invaluable therapeutic targets. Herein, heavy-labeled precursor studies and siRNA mediated subtraction lipidomic analysis utilizing LC/QTOF-MS technology led to the elucidation of the enzyme responsible for N-acylglycine biosynthesis.

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Tandem mass spectrometry for end-group analysis of synthetic polymers: a useful guide to understand side-reactions in new polymerization processes

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Mass spectrometry has become a powerful tool for polymer end-group analysis, a key step in the investigation of polymerization mechanisms. Detection of individual polymer molecules as ionic adducts allows monomer(s) to be identified based on the repeating mass increment(s) within the distribution while the combined end-group mass is evaluated from the mass value obtained upon extrapolation to zero monomer. Because synthetic polymer samples can exhibit distribution of molecular weights, end-groups and architectures, the use of mass analyzers of high resolving power is mandatory to minimize peak overlaps and to determine ion elemental composition based on accurate mass measurements. Finally, each end-group can be individually characterized by studying MS/MS data with regard to dissociation rules reported for the studied polymer family. In the present study, this MS-based analytical methodology was implemented to investigate ring-opening polymerization of cyclic ketene acetals by nitroxide-mediated polymerization (NMP), an alternative strategy to synthesize linear polyesters. Since targeted polymers were expected to hold a nitroxide termination, electro spray ionization (ESI) was used as a soft ionization technique to ensure the integrity of original end-groups. The cyclic ketene acetal was 5, 6-benzo-2-methylene-1,3-dioxepane (BMDO), and prior to any MS/MS investigation of unknowns, dissociation rules had to be established for these specific polyesters. Beside the expected pBMDO species, three main by-products were structurally characterized. The nature of the end-groups in these polymeric impurities revealed the occurrence of specific side-reactions, found to account for the increase of the polydispersity index with monomer conversion, an unusual feature in the NMP process.

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