GC–MS Analysis of Silylated Amaryllidaceae Alkaloids

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Ph.D. Dissertation

This doctoral dissertation focuses on the gas chromatography–mass spectrometry (GC–MS) analysis of Amaryllidaceae alkaloids, with particular emphasis on the impact of silylation on their analytical characteristics, including volatility, thermal stability, chromatographic behavior and fragmentation patterns. The study aims to expand the applicability of GC–MS for both qualitative and quantitative analysis of structurally diverse and biologically active alkaloids in plant matrices.

A comprehensive approach was employed, including the development of an electron ionization mass spectra (EIMS) library of both underivatized and silylated alkaloids, as well as comparative analysis of their chromatographic and spectral properties. The results demonstrate that silylation significantly improves the detectability, peak symmetry and thermal stability of hydroxylated and thermolabile alkaloids, enabling more reliable identification and structural characterization, including differentiation of epimers.

Phytochemical investigations of selected Amaryllidaceae species revealed a high diversity of alkaloids, including the identification of several new natural compounds. In addition, a fast, sensitive and validated GC–MS method for the quantification of galanthamine in *Hippeastrum papilio* was developed. The method is based on direct injection of acidified aqueous extracts, eliminating the need for organic solvents and extensive sample preparation, thus aligning with green chemistry principles. The method demonstrated excellent linearity, precision, and sensitivity, with low limits of detection and quantification.

The obtained results confirm that *H. papilio* is a rich natural source of galanthamine, with potential for industrial application. Furthermore, the combined use of silylation and GC–MS analysis provides enhanced insight into alkaloid profiles and biosynthetic pathways in Amaryllidaceae plants.

The study highlights the effectiveness of silylation as a powerful analytical tool and establishes GC–MS as a versatile and reliable method for phytochemical, pharmacological, and chemotaxonomic investigations of Amaryllidaceae alkaloids.