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Silyl Sulfinate: Effective Reagent for Qualitative and Quantitative Analysis of Polyhydroxylated Compounds

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Many compounds that contain polar and reactive groups are not sufficiently volatile and stable for direct analysis by gas chromatography. The most extensively used derivatization procedure to reduce polarity, increase volatility and thermal stability of polar and non-stable organic compounds is silylation [1]. Mass spectra of silyl derivatives are useful for structural characterization and quantification of certain compounds which are analyzed by GC-MS.

In 2002, trimethylsilyl 2-methylprop-2-ene-1-sulfinate was produced in sila–ene reaction between methallylsilane and sulfur dioxide in the presence of Lewis acid [2, 3].

The present work deals with improved preparative procedure towards silyl sulfinate and with use of it as derivatization reagent in qualitative and quantitative analysis of non-volatile polyols, carbohydrates, carboxylic and hydroxycarboxylic acids. Furthermore, obtained silyl sulfinate can be used in derivatization and analysis of mixtures of polyhydroxylated compounds.

For instance, silylation and GC analysis of carboxylic and *n*-hydroxycarboxylic acids is demonstrated by examples of salicylic, stearic, palmitic, oleic, malic, mandelic, cinnamic, benzoic acids and others. The mixture of benzoylaminocaproic, tartaric, malic, malonic phenoxyacetic, mandelic, cinnamic and citric acids was analyzed by GC after derivatization. The results are shown in Fig.1.

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2. Bouchez, L. C.; Vogel, P. *Synthesis* **2002**, 225.
3. Bouchez, L. C.; Reddy Dubbaka, S.; Turks, M.; Vogel, P. *J. Org. Chem.* **2004**, *69*, 6413.

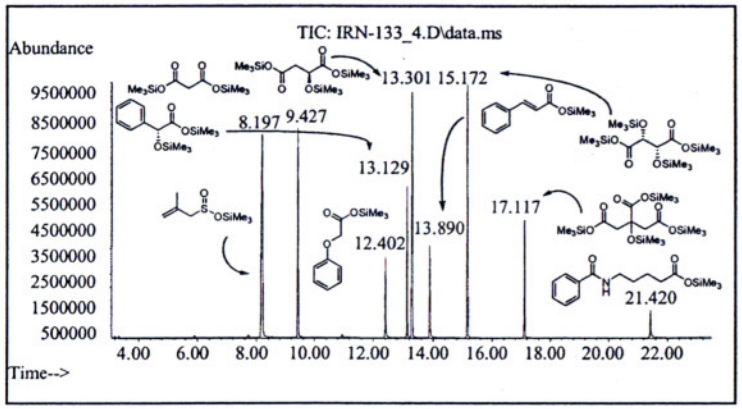

Fig.1 GC-MS traces of silylated mixture of acids