## B029 Total metabolite profiling of *Matricaria recutita* L by high field $^1$H NMR spectroscopy - the effect of origin and extraction methods

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Phytomedicine has been used for many centuries, and its use continues to increase. Quality control of phytomedicines is currently under intense scrutiny in the commercial sector, by academic researchers, and by regulatory authorities. QC methods can be subjective, and revision of the techniques currently used is required in order to meet the increasing demands of accuracy and reproducibility. Variations in both the origin and the preparation methods of phytomedicines can potentially contribute to inconsistent quality of the final products, even from batch to batch. It is therefore desirable to establish an analytical tool for profiling plant extracts which addresses the totality of the chemical profile, thus providing a means for controlling the quality of a phytomedicine without reference to active molecules or sometimes arbitrarily chosen marker compounds. High-resolution $^1$H NMR spectroscopy combined with chemometric analysis offers an innovative way to analyse metabolic changes. Here we have demonstrated an application using a combination of $^1$H NMR and chemometric methods and applied them to German chamomile (*Matricaria recutita* L.). The $^1$H-NMR profiles of chamomile from three different origins, each prepared with three different extraction methods were recorded at 600 MHz. Data were processed and analysed using principal components analysis. The major differences due to the origins and extraction methods can be identified. NMR analysis of the whole extract showed Egyptian chamomile had higher glutamate and proline contents together with a lower sugar content compared to Slovakian and Hungarian sources. PCA score plots also showed separations between the both the extraction and drying process of the samples, thus facilitating the ability to highlight sampling handling issues

## B030 Quantitative analysis of steroidal sapogenins in various fenugreek extracts (T*rigonella foenum-grae - cum* L.) by GC-MS

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Steroidal sapogenins, especially diosgenin, are widely used as precursors for semi-synthesis of numerous pharmaceutical drugs, such as contraceptive hormones, anti-inflammatory agents or steroidal diuretics (1,2). Fenugreek (*Trigonella foenum-graecum* L.) has been reported to contain up to 2.2 % (dry weight) of these compounds, mainly diosgenin (3), and thus represents a potentially useful commercial source.

Due to their low volatility, steroidal compounds usually require derivatisation before GC analysis. But due to irregular derivatisation process, this procedure may considerably complicate the interpretation of chromatograms, especially in the case of plant extracts.

In this contribution, we present a quantitative GC-MS method, using the SIM mode without derivatisation. This method allowed to identify and quantify unambiguously three steroidal sapogenins, namely diosgenin (D), smilagenin (S) and tigogenin (T). Analysis were performed using a HP-5 MS column (30 m x 0.25 mm x 0.25 µm), with helium as carrier gas at a flow-rate of 1 mL/min. The injector (splitless, 1 µL) was heated at 280 °C. The temperature program was initial 190°C (1.5 min), from 190 to 310 °C at 7 °C/min and hold at 310 °C for 10 min. For each compound, a quantification ion was selected, as well as two confirmation ions. Target ions were m/z = 271 for diosgenin, and m/z = 273 for both smilagenin and tigogenin. Confirmation ions were m/z = 300 and 342 for diosgenin and 302 and 287 for both smilagenin and tigogenin.

The developed method allowed a sensitive and selective determination of the compounds in seed, leaf and root extracts of fenugreek obtained by microwave-assisted extraction. Typical measured concentrations were for seeds: 0.150 % D, 0.072 % S and 0.068 % T; for leaves: 0.048 % D, 0.038 % S and 0.031 % T; for roots: 0.023 % D, no S detected and 0.006 % T.

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