WHISKY: TASTE, SMELL, AND MASS SPECTROMETRY

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INTRODUCTION

Whisky is Scotland's biggest export commodity

There are several different whisky regions each has characteristic flavours…

Whiskys from Islay are renowned for medical peatiness and maritime flavours

Speyside whiskys are renowned for elegance, exhibiting flowery, heathery-honey notes and a restrained, fragrant peatiness

Using a chemometric approach based upon principal component analysis (PCA) and the MarkerLynx application manager to mine exact mass UPLC– and GC-TOF/MS data we show that is it possible to distinguish between different whiskys.

This methodology could be for food quality, authenticity and traceability studies.

METHODS

GC/ToF MS

- CTC Analytics CombiPal autosampler with SPME
  - Fibre for flavours, volatiles & semi-volatiles
- Agilent 6890N GC
- GCT Premier
  - Electron Impact (EI+) *m/z* 50 → 500
  - lock mass = *m/z* 218.9856 Perfluorotributylamine,
  - Dynamic Range Enhancement (DRE) On
  - Analysis time = 30 min

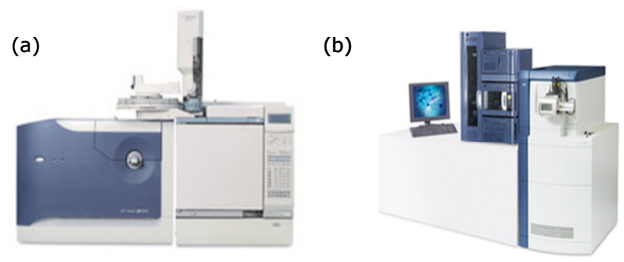

Fig 1a: GCT Premier Fig 1b: UPLC/Q-ToF Premier

UPLC/Q-ToF Premier MS

- Waters Acquity UPLC column
  - Acquity BEH C18 1.7 um 2.1 x 100 mm Column
- Q-ToF Premier
  - Electrospray (EI-) *m/z* 50 → 1000
  - lock mass = *m/z* 554.2615 Leucine Enk.
  - Dynamic Range Enhancement (DRE) On
  - Analysis time = 10 min

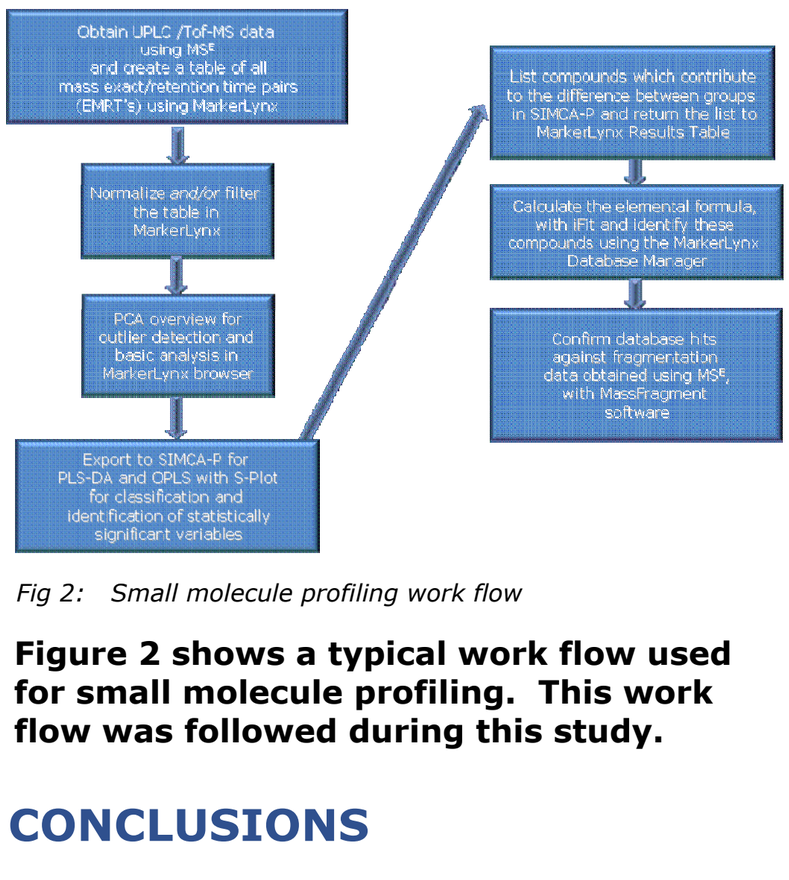

Fig 2: Small molecule profiling work flow

Figure 2 shows a typical work flow used for small molecule profiling. This work flow was followed during this study.

CONCLUSIONS

- **Using GC/MS and UPLC/MS different whiskys could easily be distinguished.**
- **The whiskys used in this study do not separate on geographical location alone.**
- **Whisky flavour is determined by many factors e.g. type of barrel it is matured in, type of still used *etc***
- **The application of UPLC/MS and GC/MS-chemometric analysis can be applied for:**
  - ⇒ **Monitoring of product quality**
  - ⇒ **Routine monitoring of raw material quality**
  - ⇒ **Detection of product adulteration, contamination & substitution**
  - ⇒ **Replacement or augmentation of sensory evaluation with analytical instrument systems**

RESULTS AND DISCUSSION

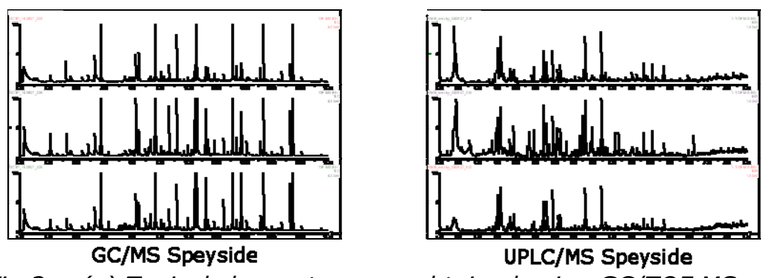

Fig 3: (a) Typical chromatograms obtained using GC/TOF MS
(b) Typical chromatograms obtained using UPLC/ TOF MS

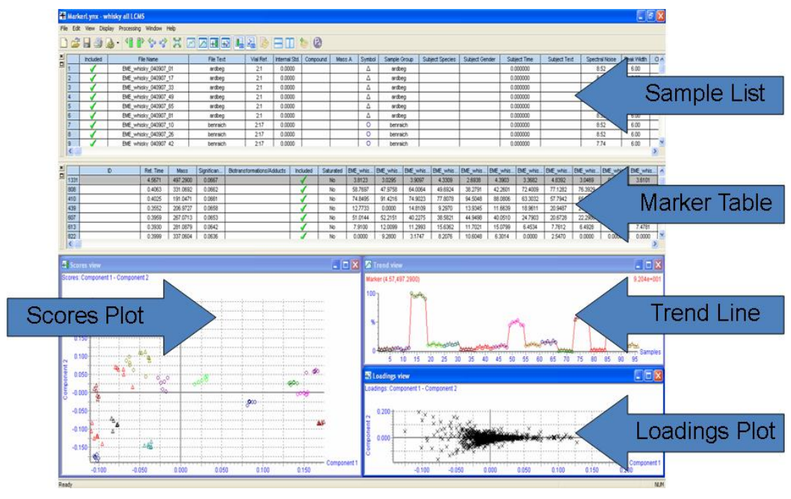

Fig 4: The MarkerLynx Browser containing, sample list, marker table, trend line, scores plot and loadings plot.

- The whiskys were analysed using GC/TOF MS and UPLC/TOF MS.
- Figure 3 shows some typical examples of the chromatograms obtained
- Data were processed using Waters application manager MarkerLynx, which enables comparison of data sets.
- Figure 4 shows the MarkerLynx browser.
- MarkerLynx incorporates a peak deconvolution package which allows detection and retention time alignment of the peaks in the eluting chromatograms.
- Data is collected into a single matrix by aligning peaks with the same mass/retention time along with their normalised intensities.
- PCA is subsequently performed.

STATISTICAL ANALYSIS

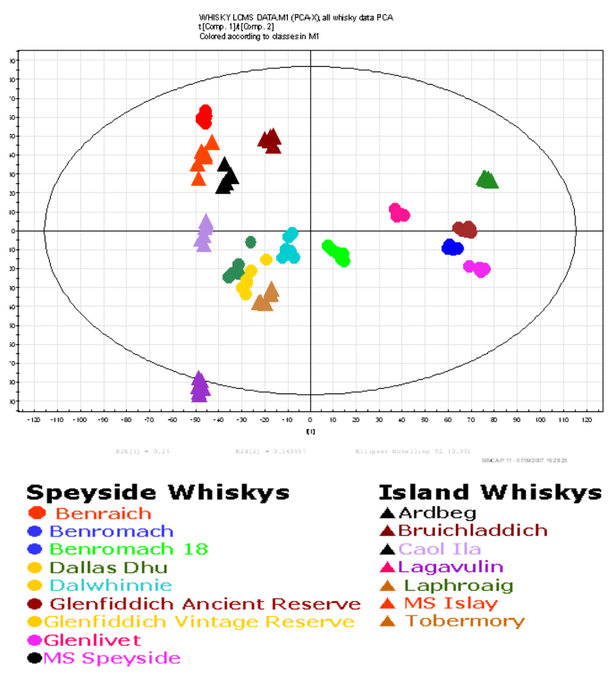

Fig 5: The score plot obtained on analysis of the UPLC/TOF MS data

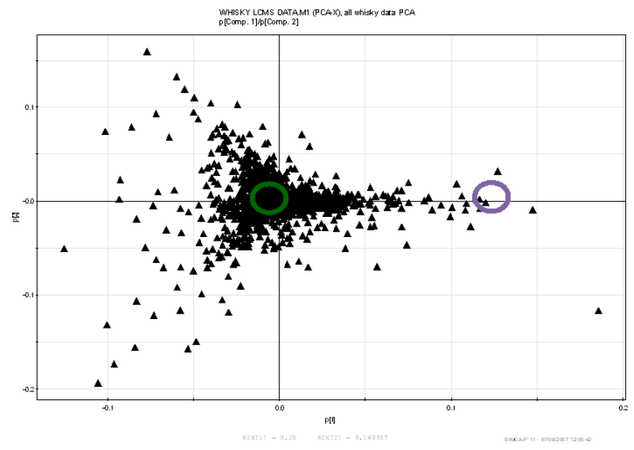

Fig 6: The loadings plot obtained on analysis of the UPLC/ TOF MS data

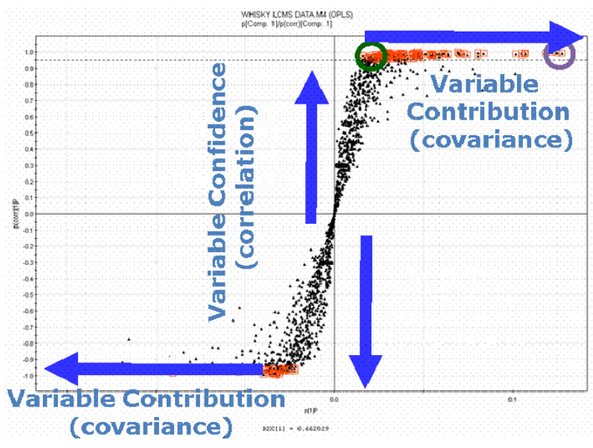

Fig 7: The score plot obtained on analysis of the UPLC/TOF MS data

- The scores plot (fig. 5) provides information about the similarity of each sample. Each symbol represents a single injection.
- The loadings plot (fig. 6) shows which exact mass retention time pairs (EMRTs) are responsible for the clustering observed in the scores plot.
- More detailed information can be obtained if only two variables are compared using OPLS within Unimetrics' SIMPCA-P software package. For example Glenfiddich Ancient and Vintage reserves (fig. 7). The confidence in each EMRT along with its contribution can be easily be assessed. Interesting results can be extracted and used to populate the results table in MarkerLynx

DATA BASE AND LIBRARY SEARCHING

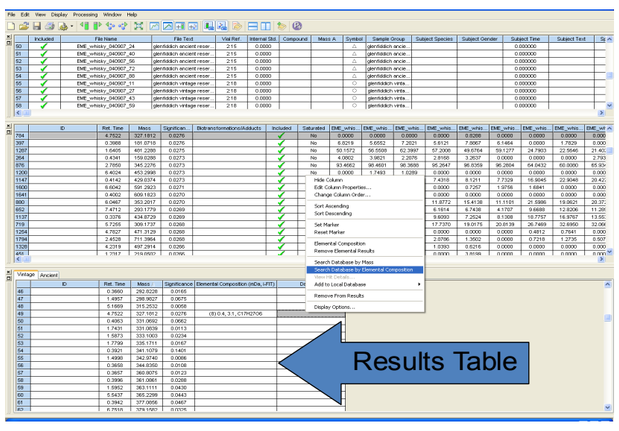

Fig 8: Screen shot showing the UPLC/TOF MS results table and the option to database search

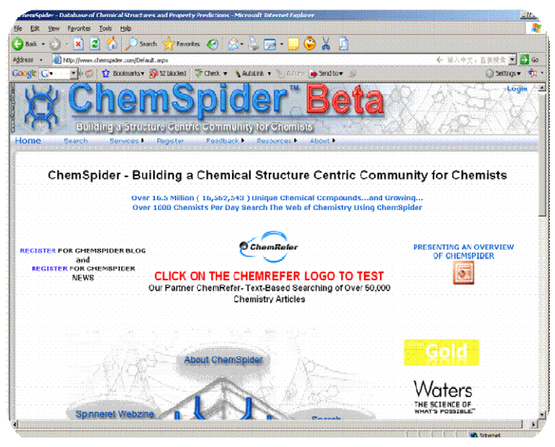

Fig 9: Online database search engine:ChemSpider

- Within the results table (fig. 8) the elemental composition of the EMRTs can be calculated. Both accurate mass and elemental composition can be used in a database search to enable identification of the compounds.
- Local databases and on-line searching can be performed. ChemSpider (fig. 9) is a free online chemistry search engine that accesses over 80 different databases.
- Identification of EMRTs identified from the GC-MS analysis can be performed by library searching (fig. 10).

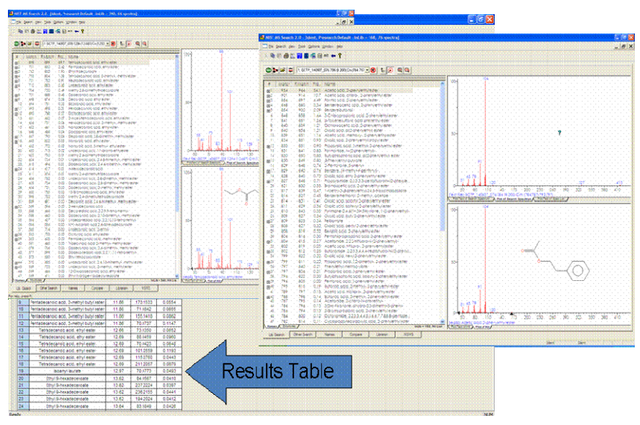

Fig 10: Screen shot showing some of the resultd obtained fron searching the NIST library and the GC/TOF MS results table,

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