GC-TOFMS Study on Genuine Scotch Whisky and Whisky Produced by Alternative Methods

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Key Words: GC-TOFMS, Food, Flavors

1. Introduction

A method has been developed in collaboration with Quadru, Environmentek, and NML, CSIR, South Africa—for the differentiation of genuine Scotch Whisky from lower grade material, produced by different manufacturing methods, and sold as Whisky.

Genuine Scotch Whisky is produced in accordance with strict manufacturing requirements to guarantee a drink of exceptional and consistent quality. As such it occupies an elite position in the liquor trade and consequently commands high prices. In South Africa unscrupulous manufacturers have developed alternative products which are marketed with names indicating a Scottish origin, and labeled as whisky. These products, while visually resembling genuine Scotch Whisky, are of inferior quality and lack the flavor components associated with the original.

A quick easy method of analysis was needed to rigorously differentiate acceptable product from the adulterated material.

2. Experimental Methods

GC Parameters: Agilent 6890N
Column:
DB-Wax; 20 m x 0.18 mm x 0.30 µm
Injector Temp: 200°C
Split Flow: Split 20:1
Oven Program:
50°C for 1.2 minutes, then to 150°C at 16°C/minute, then to 240°C at 66°C/minute, hold for 5 minutes
Flow Rate:
1.2 ml/minute Helium at constant flow

MS Parameters: Pegasus III GC-TOFMS
Mass Range: 30 to 350 amu
Acquisition Rate: 20 spectra/second
Ion Source Temp: 170°C
Transfer Line Temp: 170°C
Total Acquisition Time: 13.8 minutes

This method is a slightly modified version of a previous method used in the analysis of whisky and cognac.[1]

Six samples of commercially available whisky were obtained from Environmentek, Council for Scientific and Industrial Research, South Africa. Of these, three samples were genuine Scotch Whisky, two were believed to be cane spirit to which colorant had been added, and one was labeled as "a mixture of Scotch and South African whiskies."

3. Results and Discussion

Genuine Scotch Whisky Samples

1 µl of each of the three genuine Scotch Whisky samples were injected and run using the conditions described above. The chromatograms obtained for these three analyses are superimposed on each other and shown below, in Figure 1, plotting the total ion current (TIC).

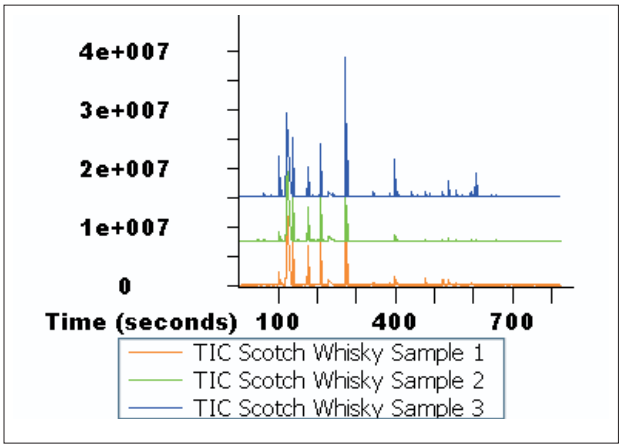

Figure 1. TIC Chromatograms of Genuine Scotch Whisky Samples.

These samples share many components; the main differences between the samples appearing after 400 seconds in the chromatograms. This region of the chromatogram contains small amounts of components which give the whisky its characteristic flavor1. It is of note that the most complex of the samples is sample 3, which is a Single Malt Whisky, and as such is reputedly of higher quality and complexity. The similarity of the samples indicates a common method of manufacture, with the differences perhaps arising from minor process modifications, differences in the origins of raw materials, or different aging protocols.

Adulterated Whisky Samples

1 µl of each of the two adulterated whisky samples were injected and run using the conditions described above. The chromatograms obtained for these two analyses are superimposed on each other and shown below, in Figure 2, plotting the total ion current (TIC).

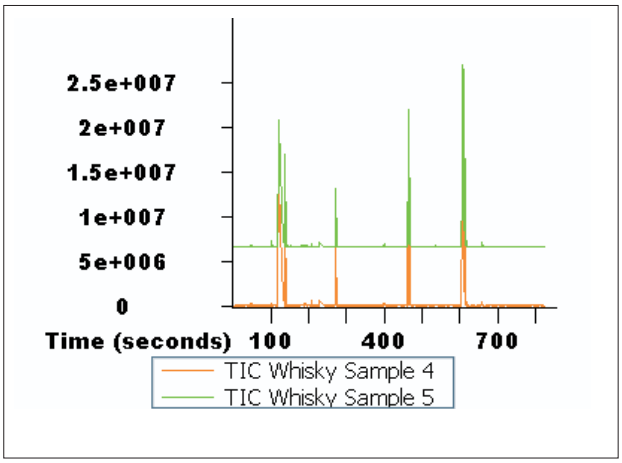

Figure 2. TIC Chromatograms of Adulterated Whisky Samples.

These samples are practically identical with no significant differences between them. The similarity of the samples again indicates a common method of manufacture. However, it is clear that they differ vastly from the genuine Scotch Whisky samples, as can be seen below, in Figure 3, in which the chromatogram of one of the adulterated samples is superimposed on the chromatogram of one of the genuine samples.

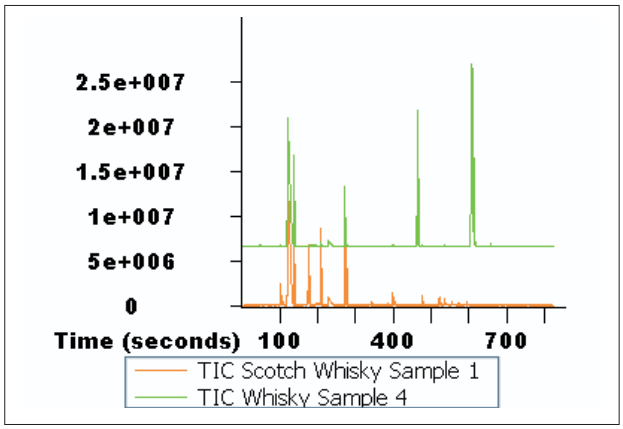

Figure 3. TIC Chromatograms of Adulterated and Genuine Whisky Samples.

Two major components are present in the adulterated sample but not in the genuine sample. On the basis of their mass spectra these compounds were identified as glycerol and propylene glycol. The recorded mass spectra for these compounds, together with the corresponding library matches are shown below, in Figures 4 and 5.

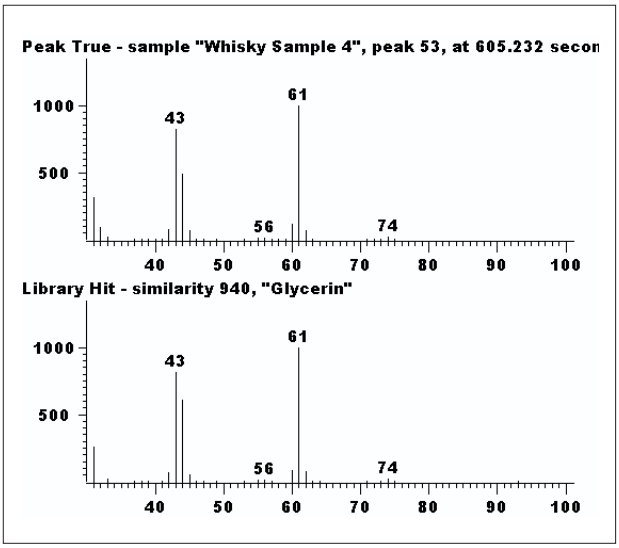

Figure 4. Mass Spectrum of Glycerol and Library Match.

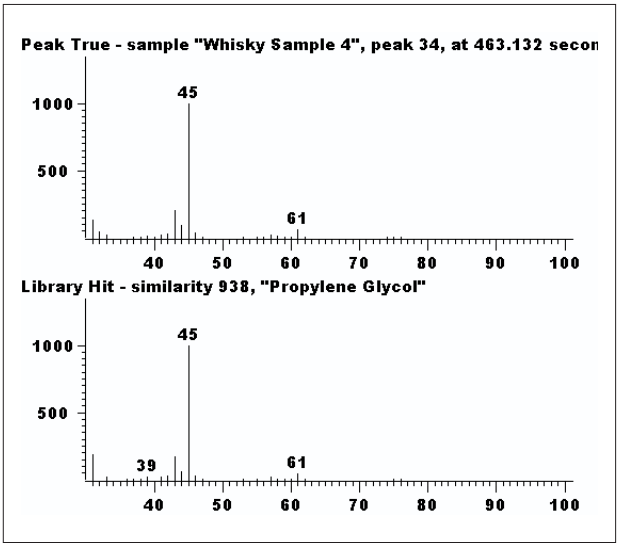

Figure 5. Mass Spectrum of Propylene Glycol and Library Match.

It is apparent that most of the flavor components, which appear in the chromatograms of the genuine Scotch Whisky samples after 400 seconds, are lacking in the adulterated samples.

On the basis of these results it can be suggested that the adulterated samples were prepared by addition of commercial glycerol and colorants to a distilled cane spirit. The propylene glycol may be present as an impurity in the commercial glycerol. Sale of this mixture as "whisky" would then be fraudulent and subject to legal action.

Mixed Scotch and South African Whisky Sample

1 µl of the mixed "Scotch and South African" whisky sample was injected and run using the conditions described above. The chromatogram obtained for this analysis is shown below, in Figure 6, plotting the total ion current (TIC).

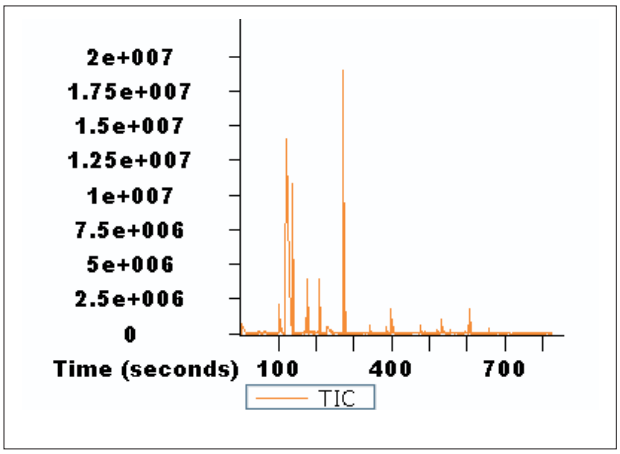

Figure 6. TIC Chromatogram of Mixed Whisky Sample.

When the chromatogram of this sample is superimposed with a chromatogram of genuine Scotch Whisky, as is shown below in Figure 7, it can be seen that the samples are quite similar.

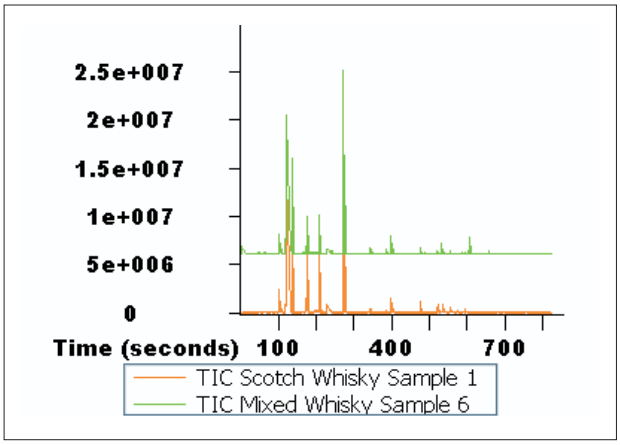

Figure 7. TIC Chromatograms of Mixed and Genuine Whisky Sample.

There is no evidence of dilution using glycerol or propylene glycol, and the mixed sample contains flavor components to be expected in genuine Scotch Whisky. There is no evidence to suggest that it is anything other than what is claimed on the label, a mixture of Scotch and South African whisky. As such it may be legally sold within South Africa.

4. Conclusions

The described work demonstrates the use of GC-TOFMS to differentiate genuine Scotch Whisky from cheaper, and sometimes illegal, products. The use of a Time-of-Flight mass spectrometer in this work demonstrates a number of advantages over other types of mass spectrometers, specifically the ability to do faster chromatography for increased sample throughput with no sacrifice in the quality of the results obtained.

The strength of the Pegasus GC-TOFMS for the analysis of these sample types lies in its automated data handling capabilities. Peak finding, spectral determination (deconvolution), and library searching are all automatic. This is possible due to the high degree of spectral continuity generated as well as the large data density allowed by the Pegasus GC-TOFMS system—up to 500 full mass spectra per second. Peaks are located and full range mass spectra obtained for all components, even when peaks coelute completely beneath other peaks, allowing confident structural determination and quantitation. Samples can then be compared, either by using the sophisticated sample comparison software1 or visually as in the above study. Differences can be rapidly located and identified to unequivocally indicate product origin.

5. References

[1]Qualitative Comparison of Whisky Samples Using Fast GC-TOFMS; LECO Corporation Application Note, No. 203-821-200.

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