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Determination of Impurities in Whiskey Using a Gas Chromatograph

Introduction: The purpose of this lab is to separate and identify six different compounds commonly found as impurities in both American and Scotch Whiskey. Ethanol and water are the primary components in all alcoholic beverages. However, it is the presence of other compounds in much smaller quantities that yields the distinctive taste for each beverage. Fermented beverages such as beer, wine and sake tend to produce sulfur-containing compounds which give them their "yeasty" smell. Distilled beverages (such as whiskey, vodka, rum and tequila) tend to have alcohols other than ethanol and esters present, which are characterized by strong smells. For most distilled beverages, the presence of these other alcohols is considered a "fault", but the taste of whiskey is characterized by larger quantities of these strong-smelling derivatives.

Students will initially inject each impurity separately into the GC to determine its retention time and then prepare a standard solution containing all impurities to demonstrate compound separation in a complex mixture. Four unknown samples of whiskey will then each be injected on the GC and students will be able to identify the presence and relative quantities of each impurity present in the whiskey. Students can then make a final ranking of whiskey "quality" based on the presence of impurities.

Materials:

- Standard impurities: Methanol, Acetaldehyde, Ethyl Acetate, 1-Propanol,
- 1-Butanol, 1-Pentanol
- Standard solution= 40% ethanol in water to simulate whiskey matrix
- 4 Whiskey brands known to contain impurities to be used as unknowns.

Pre-Lab:

1. Describe how gas chromatography works including a description of the stationary and mobile phases.
2. Students look up boiling points for impurities and ethanol to predict the relative elution order/retention times for compounds moving through the column.
3. Both Ethyl Acetate and 1-propanol have very strong and distinctive smells. Why do you think it is good to have strong smelling compounds as part of a beverage?

Procedure (abbreviated):

1. Students inject 0.2 microliters of each impurity one at a time onto SeaPort GC system. PC-based software will be used to show compound peaks as sensors detect the compound exiting the chromatography column. Students record retention time for each peak to determine where each impurity is expected to appear.
2. Students prepare mixture of impurities in solution of 40% ethanol. Demonstrate that each compound yields a separate peak even when in a complex mixture. Students identify the peaks on a printed chromatogram based on their measured retention times. (Graphs can be produced using either Excel or another graphing program).
3. Inject 0.2 microliters of each unknown whiskey sample on SeaPort GC system. Identify the impurities from a printed graph by comparing these chromatograms to the calibration standard chromatogram (from steps 1 and 2).

4. Optional Procedure: On your GC-MS, inject unknown samples. Your system may be able to identify the impurities based on its spectra library. If not, we can also use the standard sample for comparison. Also, your system can likely calculate peak area. We can spike each sample used for your GC with a known quantity of Hexanone. By comparing relative peak areas, students can calculate the relative concentrations of impurities in ppm.

Post-Lab Questions:

1. Present the chromatograms for the calibration mixture and for each Unknown Whiskey with peaks identified. Are the impurities in the Unknowns in the same order as the calibration standard? Are there significant differences in the retention times?
2. Rank (highest to lowest) the Unknown Whiskey samples based on their relative impurity concentrations. For example:

Impurity 1	*Impurity 2*	*Impurity 3*	*Impurity 4*
Whiskey 1	W2	…	…
Whiskey 3	W3	…	…
Whiskey 2	W1	…	…
Whiskey 4	W4	…	…

3. Rank your Unknown Whiskey samples based on "quality". Does one impurity standout as a marker of "quality"? (The instructor can give students direction as to whether higher "quality" whiskeys have fewer impurities or more.)
4. If Optional Procedure is followed…calculate quantities of impurities present in samples.