SHIMADZU APPLICATION NEWS

LAAN-A-G-E008

● GAS CHROMATOGRAPHY

No.G244

Improving the Repeatability of Aqueous Solution Analysis Performed with Capillary Columns

Sometimes it is difficult to attain a high level of repeatability when performing the split analysis of an aqueous sample with a capillary column. This is mainly because of the following factors:

Because water has a high latent heat of vaporization, instantaneous vaporization in the sample injection chamber does not take place properly and the behavior at the split point is unstable.

Because the vaporization volume is large, the volume of sample vapor exceeds the capacity of the glass insert.

With split analysis, because the sample passes through the glass insert relatively quickly, sufficient vaporization is not possible if heat is not exchanged efficiently. In order to perform analysis with a high level of repeatability, it is necessary to insert silica wool in the glass insert to increase the heat capacity and thereby increase the vaporization efficiency. The silica wool not only increases the heat capacity, it also helps evenly mix the vaporized sample and is indispensable for split analysis.

■ Analysis of Alcohol in Whisky

When using the silica wool provided with the GC-2010 or GC-2014 (P/N : 221-48600, 225-09147), although 10 mg is inserted in standard applications, the repeatability attained with aqueous solutions can be improved by adding more silica wool to increase the heat capacity.

The alcohol in whisky was analyzed with the amount of silica wool increased to 20 mg. The AOC-20i was used for sample injection. Increasing the amount of silica wool made it possible to attain a high level of repeatability for an aqueous solution.

Table 1 Analytical Conditions

Column	: Stabilwax 30 m × 0.25 mm I.D. df = 0. 25 µm
Column Temp.	: 70 ˚C (2 min) - 20 ˚C/min - 120 ˚C (1.5 min)
Carrier Gas	: He 30 cm/sec (103.6 kPa, 1.2 mL/min)
Injection port	: 250 ˚C
Split Ratio	: 1:30
Detector	: 280 ˚C FID - 2010
Injection volume	: 1 µL AOC-20i

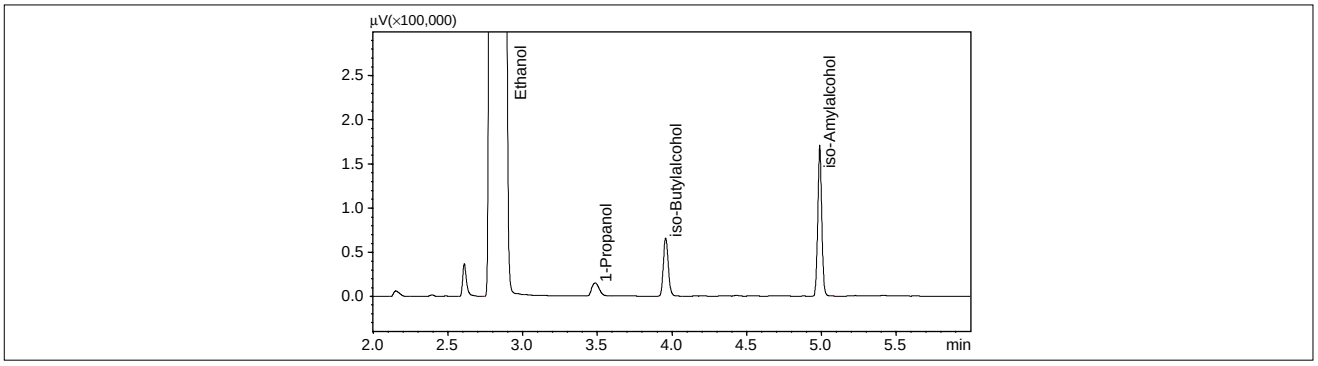

Fig.1 Analysis of Alcohol in Scotch

Table 2 Repeatability

Retention time	1	2	3	4	5	Average	C.V.%
Ethanol	2.854	2.854	2.854	2.854	2.854	2.8540	0.000
1-Propanol	3.489	3.488	3.488	3.487	3.487	3.4878	0.024
iso-Butyl alcohol	3.959	3.959	3.959	3.959	3.958	3.9588	0.011
iso-Amyl alcohol	4.991	4.991	4.991	4.990	4.990	4.9906	0.011
Peak area	1	2	3	4	5	Average	C.V.%
Ethanol	75689200	75504352	75404960	75311975	75309397	75443976.8	0.210
1-Propanol	50548	50371	50296	50357	50324	50379.2	0.196
iso-Butyl alcohol	151177	150702	150407	150394	150309	150597.8	0.237
iso-Amyl alcohol	317582	316458	316064	316149	315915	316433.6	0.212
Peak height	1	2	3	4	5	Average	C.V.%
Ethanol	21989563	21988433	21965308	21966209	21989623	21979827.2	0.058
1-Propanol	14721	14670	14401	14541	14506	14567.8	0.884
iso-Butyl alcohol	65899	65396	65000	65647	64954	65379.2	0.624
iso-Amyl alcohol	170626	170230	168745	169388	168850	169567.8	0.492

■ Packing Method of Silica Wool

In order to improve the repeatability, the glass insert must be packed evenly with silica wool. The packing position also significantly influences the repeatability.

In the standard silica-wool packing position for the GC-2010 split insert (when using an AOC-20i), the top of the wool is 25 mm away from the top of the insert. This position is directly under the tip of the syringe needle at the time of injection. Making the top surface of the silica wool flat so that the discharged sample makes contact with the wool in the same way each time may help to further improve the repeatability.

In the standard silica-wool packing position for the GC-2014 split insert, the top of the wool is 20 mm away from the top of the insert. At the time of injection, the tip of the syringe needle penetrates approximately 5 mm into the silica wool. In analysis using an organic solvent with a small heat capacity as the sample solvent, although the repeatability may be slightly inferior to that attained when the wool is directly under the tip of the syringe needle (i.e., in the standard packing position for the GC-2010 split insert), analysis is less affected by the state of the needle tip (e.g., clogging). If the wool is packed in this position, immediately after replacing the insert, injection must be repeated until the condition of the silica wool stabilizes.

In the analysis of aqueous solutions with large heat capacity, the repeatability may be improved by packing approximately twice the standard amount of silica wool at the 20 mm position. Because the tip of the syringe needle penetrates the silica wool, the uniformity of the silica wool in the region approximately 25 mm away from the top of the insert is very important. If the wool is not packed uniformly in this region (i.e., if there are large cavities), vaporization may occur unstably and this may adversely affect the repeatability. Furthermore, with samples that contain high numbers of nonvolatile constituents, residual nonvolatile constituents may be concentrated, push open the wool, and create gaps. The packing volume must be sufficient to prevent this. The optimal packing volume varies with the type of silica wool used and so, when switching to a different type of silica wool, the ratio of the wool fiber thickness (which is related to the surface area) to the weight must be taken into consideration.

In order to ensure that the silica wool is packed uniformly and that the top surface is flat, it is recommended that Shimadzu's simple insert-wool packing tool (P/N : 092900, Shimadzu GLC) is used to pack the silica wool

Fig.2 Insert-Wool Packing Tool

■ Syringe for Aqueous Solution Analysis

If an aqueous solution is analyzed with a standard auto-injector syringe, plunger movement may become heavy during operation and this may adversely affect the repeatability. Sticking of the plunger in aqueous solution analysis can be reduced by using the AOC elastic syringe (P/N : 221-49548), which has a titanium plunger. This syringe incorporates a septum to prevent clogging in the needle tip (modified needle-tip shape) and is therefore effective for the analysis of other types of sample analysis.

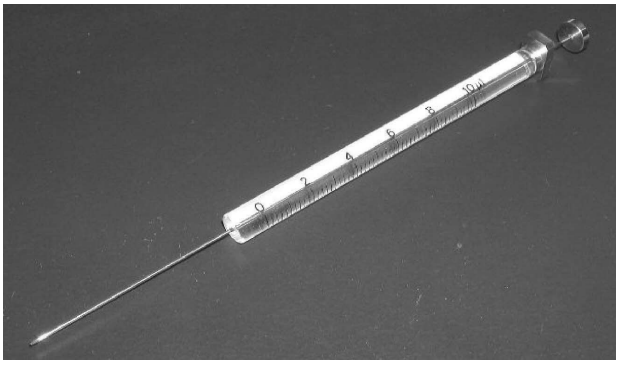

Fig.3 AOC Elastic Syringe (P/N : 221-49548)

NOTES:

✻This Application News has been produced and edited using information that was available when the data was acquired for each article. This Application News is subject to revision without prior notice.

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