The Analysis of Hand Sanitiser by Gas Chromatography with Flame Ionisation Detector

Advanced Projects and Custom Solutions Team, SCION Instruments NL BV, Amundesweg 22-24, 4462P Goes, The Netherlands.

Since the global outbreak of the Corona Virus, hand sanitiser has become part of a daily routine for us all. Alcohol-based hand sanitiser containing between 60% to 95% alcohol is recommended by the World Health Organisation (WHO). Typically, hand sanitiser contains ethanol, isopropyl alcohol (IPA) and/or n-propanol, the main active components responsible for the removal of viruses and bacteria from hands. The concentration of these active ingredients is vital in determining the effectiveness as a disinfecting agent. Methanol is a contamination by-product, commonly found in hand sanitisers. Hand sanitisers also often contain Glycerin, a moisturising agent used to protect skin from the alcohol.

International Standard Test methods such as ASTM D3695 and USP 611 define standard procedures for the qualitative and quantitative analysis of alcohols by Gas Chromatography (GC). For the detection of volatile alcohols in water, a GC with Flame Ionisation Detector (FID) is recommended. GC also provides critical information such as alcohol identity using retention time indicators; ensuring that the hand sanitiser is of high quality and has not been contaminated.

SCION Instruments developed a quick yet effective method for the identification of Methanol, Isopropyl Alcohol (IPA), Ethanol, n-propanol and Glycerin in hand sanitiser by GC-FID, as recommended by ASTM and USP methods.

Experimental

The below method is applicable to both SCION 436GC and 456GC instruments. The 4X6 was equipped with a FID and 8400 Autosampler, using a plunger in needle syringe. *Figure 1* showcases the SCION 4X6 Gas Chromatographs

Figure 1. SCION Instruments 4X6 Gas Chromatographs

Particular care needs to be taken when selecting injection and inlet volumes to prevent backflush. Backflush occurs when the solvent and sample flow back up into the inlet resulting in gas lines becoming contaminated. This can be observed when the chosen liner volume is too small for the expansion of water in the sample. In certain conditions, 1.0µL of water can expand up to 1400 times its volume. When choosing a liner for analysis, this expansion must be considered. A 4mm liner with glass wool is recommended and was implemented throughout this analysis. The glass wool provides better RSD% values compared to a liner without the glass wool.

The experimental conditions for the analysis of hand sanitiser can be found in *Table 1*.

Table 1. GC-FID Method Parameters

Parameter	Setting
Injector	S/SL, 20:1 split, 250°C, 0.2µL
Liner	4mm, Glass Wool
Column	SCION Wax MS 30m x 0.53mm x 1µm
Oven	50°C (5 min), 30°C/min to 230°C (3 min)
FID	250°C
Software	CompassCDS

Calibration standards were prepared for each of the target analytes, Methanol, Isopropyl Alcohol (IPA) and Ethanol were prepared at a concentration range from 1% to 5% (v/v), whereas n-propanol were prepared at concentration ranges of 0.1% to 1.5% and 0.092% to 0.138% (v/v), respectively. It is vital to use an internal standard (IS) when analysing hand sanitiser due to the high viscosity of the samples; acetonitrile was added to all calibration standards and samples. A Quality Control sample (QC) was prepared comprising of Ethanol at 2% (v/v) and IPA at 2.5% (v/v), with the addition of internal standard. Two hand sanitiser samples, one gel and one rub, were prepared by diluting in demineralised water, before being injected into the GC.

Results

All calibration standards were analysed, with the retention times used for peak identification and confirmation. Post identification, the precision of the method was obtained by analysing ten consecutive injections of the 1 or 1.5% (v/v) calibration standard (containing internal standard). *Figure 2* details the chromatogram used for peak identification but also highlights the precision of the method.

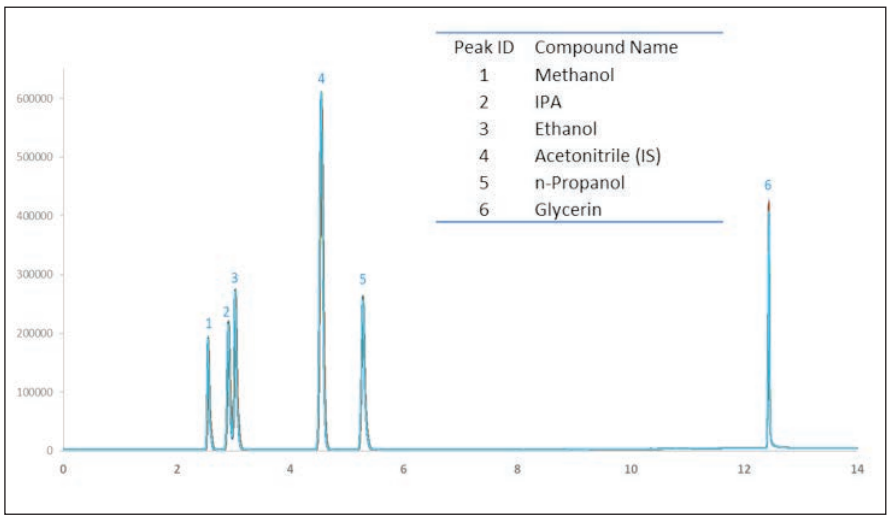

Figure 2. Chromatogram overlay of repeatability standards (n=10).

The calibration curve for each of the target compounds can be found in *Figure 3*.

With the exception of Glycerin, all target compounds exhibited a correlation coefficient (R2) value of >0.999. The R2 value for Glycerin was slightly lower at 0.9961.

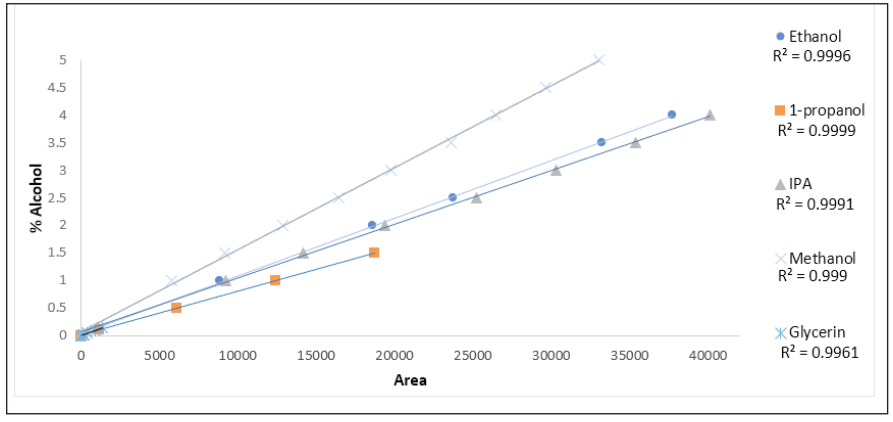

Figure 3. Calibration curve of target compounds with Acetonitrile as internal standard.

Standardised method USP 611 outlines a series of specifications that must be met for the developed method to be implemented. Specification one is that the RSD% of no more than 4%, for each compound, must be observed for the method to pass specification. The following RSD% values were obtained during the ten consecutive injections: Methanol 1.03%, IPA 1.04%, Ethanol 1.19%, n-propanol 1.05% and Glycerin 1.52%. All values were significantly lower than the specification limit of 4%, highlighting the precision of the SCION Instruments 4X6 GC. For successful quantification, USP 611 also states that the resolution of all alcohols must be >1; this was successfully achieved with all resolution values being >1.4 (IPA and Ethanol as shown in *Figure 2*). The third specification to be met is each target compound must have a tailing factor <2. Methanol exhibited a peak tailing factor of 1.68, IPA 1.09, Ethanol 1.29, n-propanol 1.14 and Glycerin 1.23. All target compounds exceeded the requirements for USP 611. Since this specification was met, the QC sample was then analysed.

The QC sample was analysed in three individual runs consisting of ten injections in each. The average concentration for the target compounds were; Ethanol 2.3% with an RSD% of 0.76%, IPA 2.5% with an RSD% of 1.37%, passing the QC check. The two hand sanitiser samples were then analysed using the same conditions as above. According to the label of sample 1 (hand gel), the alcohol content was listed as >70%. The ingredients list also listed Glycerin and IPA but without a concentration. When analysed, it was determined that sample 1 contained 73.4% (v/v) Ethanol, 1.93% (v/v) Methanol, 1.6% (v/v) IPA and 1.13% (v/v) Glycerin, thus exhibiting what was described on the label. *Figure 4* highlights the chromatogram obtained from the analysis of sample 1.

The ingredients list for sample 2 (hand rub) stated that the concentration of alcohol was 70% with no other ingredients listed. When analysed, sample 2 contained 67% (v/v) Ethanol and 0.54% Glycerin; this failing to meet to concentration described on the label; although it was still within the recommended concentration of the WHO.

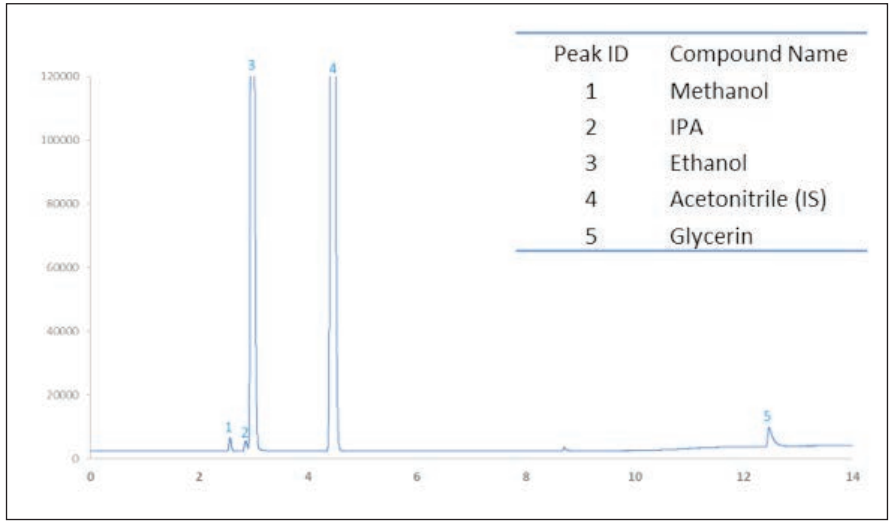

Figure 4. Chromatogram of hand sanitiser sample 1 (hand gel).

In order to maintain the quality of this analysis, it is recommended to run blank samples in between each hand sanitiser injection and QC sample. Traces of the analysed samples can accumulate within the GC system due to the viscosity of the original sample. If carry over is observed, thoroughly clean the syringe to prevent further contamination.

Conclusion

The SCION 4X6-GC analyser equipped with an FID was used to develop a method for the analysis of hand sanitiser, an every-day necessity during the Corona Virus pandemic. In under 15 minutes the key active ingredients (Ethanol, IPA and n-propanol) contaminant (methanol) and added ingredients (Glycerin) were separated, identified and quantified.)

Although the developed method is fully automated and exceeded the international standard test method requirements, it is important for operators to keep an eye on the quality, by using a quality control standard with each analysis. Due to the challenging matrix, the RSD% values should also be carefully monitored. When an increase is observed, cleaning the syringe and changing the linear is recommended.