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DETERMINATION OF LIMONENE IN RECYCLED HDPE PELLETS

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INTRODUCTION

Over four and a half million tonnes of plastic is produced annually in the UK alone, with over 85% of this going to landfill[1]. Over 45% of plastic bottles are sent to recycling with the number expected to significantly increase in the coming years. Recycled plastics have a variety of uses from new bottles/ food trays to underground drainage products and soft furnishing materials [1].

During the recycling process, clear and mixed colour plastics are separated before they are granulated and separated. The granulated plastics are then thoroughly cleaned and dried before being tested in quality control and sent to be remanufactured. The plastics are turned into high density polyethylene (HDPE) pellets.

It is vital that the pellets produced from the recycling are deodorised from contaminants and flavour compounds especially when they are manufactured into food packaging.

Limonene is a cyclic monoterpene and is the major component in the oil of citrus fruits. Limonene contaminates recycling materials and the amount of contamination needs to be reduced from the HDPE pellets, done via deodorisation during recycling [2]. During quality control testing, the pellets must have a limonene concentration of less than 20ppm.

AIM

To design and implement a method for the determination of low concentration limonene in HDPE pellets.

To demonstrate the high sensitivity of the SCION Single Quad MS.

METHOD

Instrumentation: SCION Single Quad Scion GC-MS(436) with 8400 Autosampler

Software: Scion Mass Spectrometry Work Station

Table 1. Analytical Conditions of GC-MS

Conditions	
Injector	S/SL at 250°C
Columns	30m x 0.25mm x 1.4µm (Scion624-MS)
Oven Conditions	55°C (hold 1 minute) 220°C at 20°C/min (hold 1 minute)
Carrier	Helium 1mL/min
MS	Full Scan
Peak Identification	Limonene (Ions 93/68)

Calibration standards were prepared in Carbon Disfulfide (CS_2) at a range of 0ppm, 5ppm, 10ppm, 20ppm and 40ppm.

0.1g of HDPE pellets were extracted using 1mL of CS_2 and incubated at room temperature for 60 minutes. The extracts were then injected into the GC using a 8400 autosampler. The HDPE pellets were analysed in triplicate. Figure 1 displays the solvent extraction.

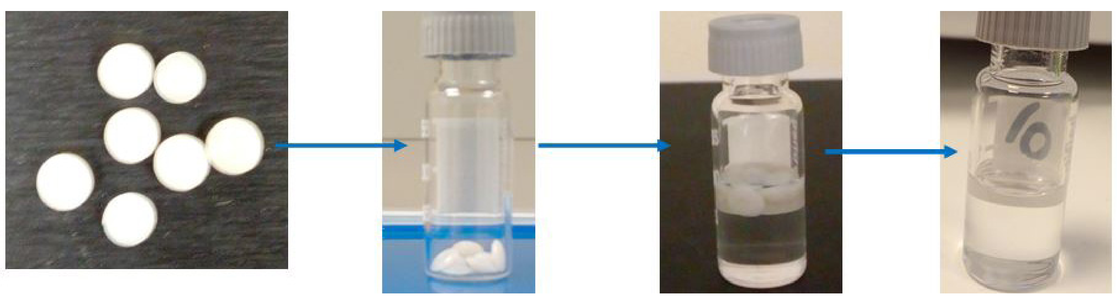

Figure 1. A simple solvent extraction of limonene from HDPE

A full scan method was used to identify m/z 93, the quantifier ion of limonene with m/z 68 as a qualifier ion. The TIC was subjected to a NIST library search for confirmation. Figure 2 shows the SCION instrumentation.

Figure 2. SCION Single Quad GC-MS with 8400 Autosampler

RESULTS

The limonene concentration of the HDPE pellets was 0.01µg/g. Figure 3 shows the limonene peak identified using the extracted ion function on the mass spectrometry software.

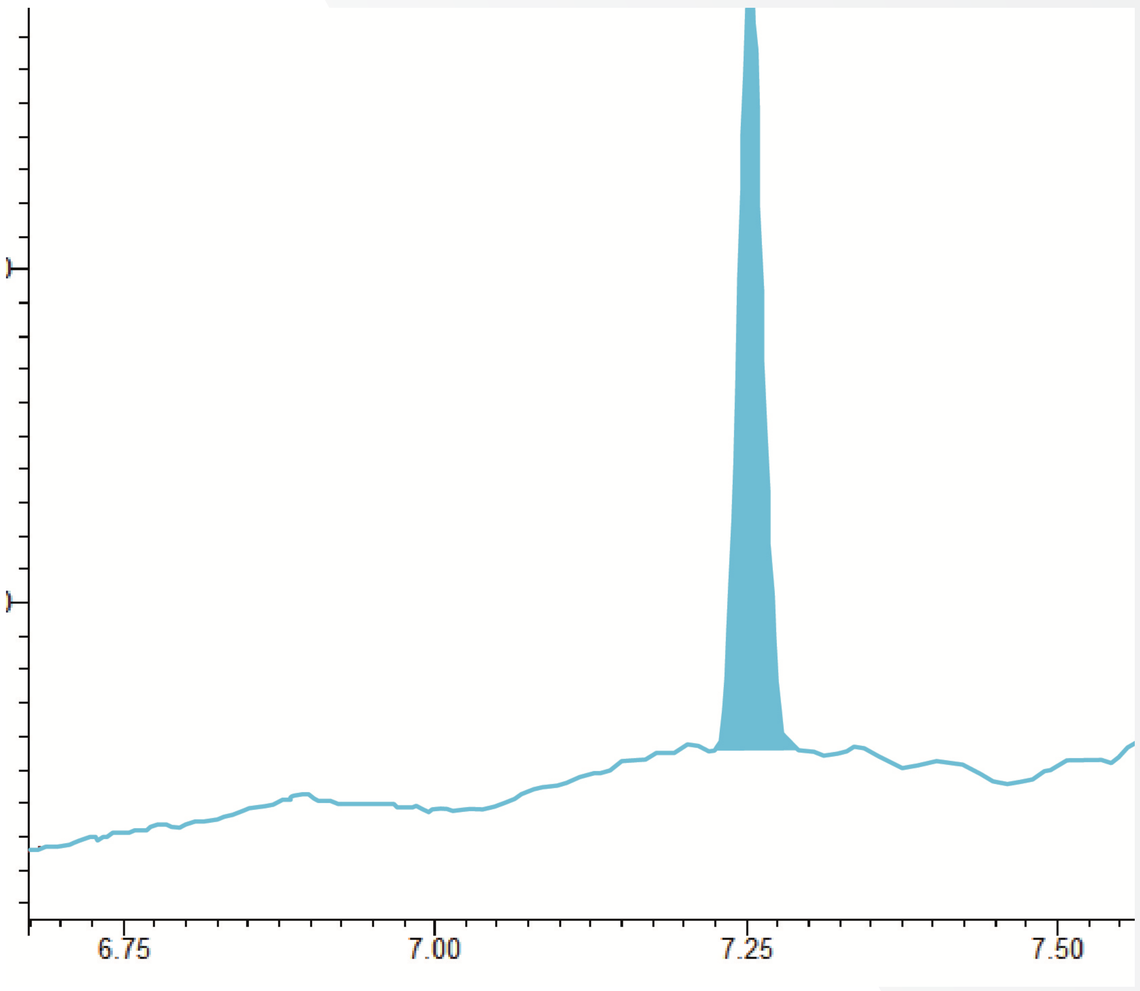

Figure 3. Ion 93 for the identification & quantitation of limonene

CONCLUSION

The SCION 436 GC coupled with SCION MS and 8400 Autosampler offers enhanced sensitivity for the detection of limonene at trace levels. A simple liquid extraction with direct injection was implemented using HDPE pellets. The quick yet efficient extraction method ensures accurate quantification along with reproducibility.

References

[1] Viridor.co.uk. (2018). Plastic Recycling. [online] Available at: https://viridor.co.uk/our-operations/recycling-and-resources/plastic-recycling [Accessed 28 Aug. 2018].

[2] Welle, F. (2005). Develop A Food Grade HDPE Recycling Process. Oxon: The Waste & Resources Action Programme, p.4.