

Introduction

DEHP, bis-(2-ethylhexyl)phthalate, is one of the most prominent phthalate contaminants in drinking water. DEHP is also used as a plasticizer during plastic manufacturing. When ingested, it is a cancer-causing hazard and presents a high risk of liver function disorders. For this reason, DEHP is a banned substance during food production, under the Restriction of Hazardous Substances (RoHS) Directive by the EU¹, and levels must be monitored. Although a banned substance, commercial sport drink manufacturers have been known to substitute palm oil, a common emulsifier, for the more cost effective DEHP.

SCION Instruments developed a method for the identification of DEHP using HPLC and a Diode Array Detector (DAD).

Experimental

A SCION LC 6000 with DAD was used with a C18 reverse phase column for the detection of DEHP in mineral water and sports drink samples. A DEHP analytical standard was prepared at a concentration range of 0.1mg/L to 100mg/L. Standard addition was also performed on two negative water and sports drink samples to demonstrate the capability of the instrument. Samples were spiked with 1ppm and 10ppm of DEHP prior to analysis.

Table 1 details the analytical conditions of the HPLC-DAD system.

Table 1. Analytical conditions of HPLC-DAD

Conditions	
Column	C18 5µm x 4.6mm ID x 150mm
Column Temp	30°C
Mobile Phase	Water/Acetonitrile (2/98 v/v)
Flow Rate	1mL/min
Injection Vol	10µL
DAD	224nm

Results

The chromatogram of a 10mg/L DEHP standard can be observed in Figure 1 whilst Figure 2 shows the calibration curve of DEHP, over a range of 0.1mg/L to 100mg/L.

DEHP exhibits excellent linearity over a wide concentration range, as demonstrated in Figure 2. Retention time and peak area repeatability of the system was tested with six consecutive injections of a 10mg/L standard, the values of which can be found in Table 2.

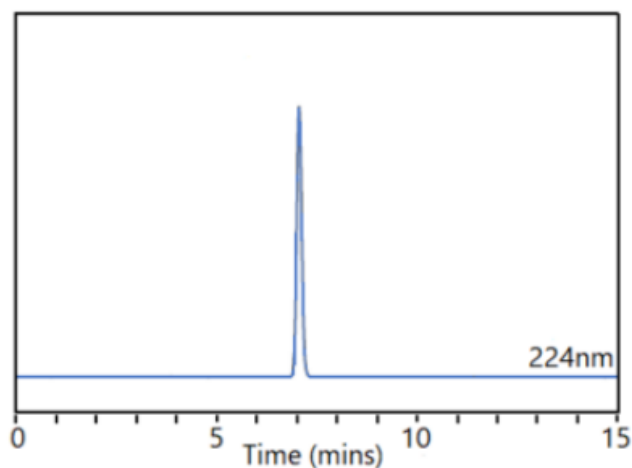


Figure 1. Identification of DEHP at 224nm (10mg/L)

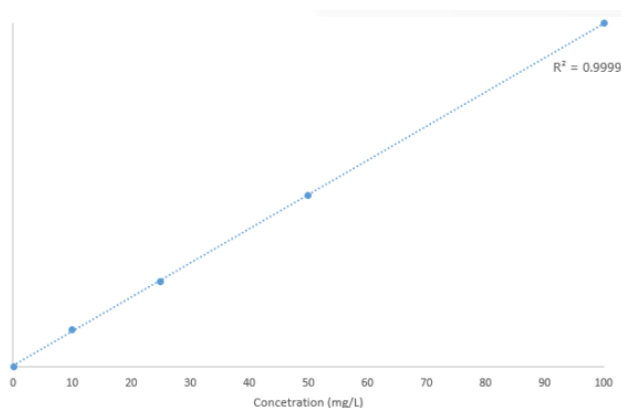


Figure 2. Calibration curve of DEHP; 0.1mg/L to 100mg/L

Table 2. Repeatability values of 10mg/L DEHP standard (n=6)

Run	RT (min)	Peak Area
1	7.058	131486
2	7.057	131742
3	7.056	131456
4	7.057	131769
5	7.056	131609
6	7.057	131743
Mean	7.057	131634
%RSD	0.011	0.11

Excellent repeatability of both retention time and peak value was observed, with RSD% values at 0.011 and 0.11, respectively, highlighting the robustness of the SCION HPLC-DAD system.

Mineral water and a sports drink were both analysed for the presence of DEHP. However, both samples were negative. Standard addition was performed on two separate samples of each sample type with an addition of 1ppm and 10ppm DEHP.

Figures 2 and 3 show the overlay chromatograms of both samples, including the initial blank results and both DEHP additions.

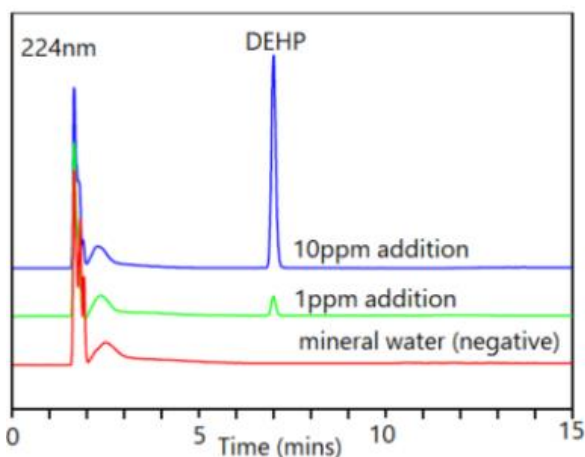


Figure 3. Chromatogram overlay of DEHP standard addition (mineral water)

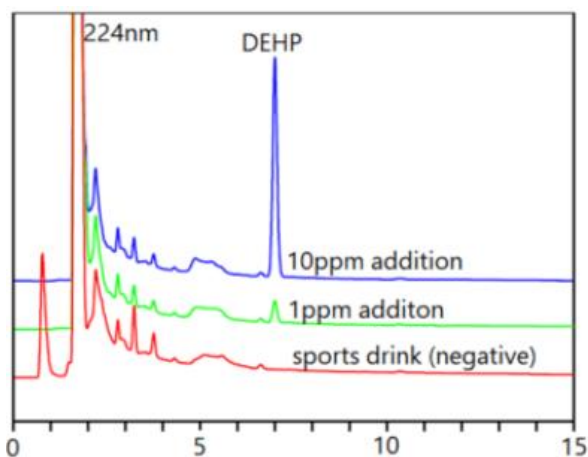


Figure 4. Chromatogram overlay of DEHP standard addition (sports drink)

The above figures demonstrate the excellent sensitivity of the HPLC-DAD system even at low concentrations such as 1ppm.

Conclusion

SCION Instruments offers the ideal solution for the identification of DEHP, a restricted contamination compound by HPLC-DAD. Excellent separation, linearity over a wide concentration range and system repeatability was observed for DEHP using a C18 column at 280nm wavelength.

References

1. European Commission (2011). Directive 2011/65/EU of the European Parliament and of the Council as Regards the List of Restricted Substances. European Parliament.

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