



ANALYSIS OF DEHP IN DRINKING WATER BY HIGH PERFORMANCE LIQUID CHROMATOGRAPHY – DIODE ARRAY DETECTOR

Introduction

DEHP, bis-(2-ethylhexyl)phthalate, is a phthalate widely used in various industries, particularly in cosmetics, packaging and more critically as a plasticiser in toy manufacturing. DEHP is also one of the most prominent phthalate contaminants in drinking water. Phthalates are not irreversibly bound to a polymer matrix so can easily leach into the environment, contaminating water supplies. Additionally, when used in food and water containers, the phthalates can be consumed by the end user due to leaching from the plastic material into the product. 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 EU[1]. These restrictions require the levels of DEHP to be continuously monitored during the manufacturing process and quality control procedures. Although a regulated compound, 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 the SCION Instruments LC6000 HPLC equipped with a Diode Array Detector. Figure 1 highlights the SCION Instruments LC6000 HPLC.

Experimental

A SCION Instruments LC6000 HPLC with a Diode Array Detector was equipped with a C18 reverse phase column (150mm x 4.6mm x 5µm) for the detection of DEHP in mineral water and sport drink samples. DEHP analytical standards were prepared at a concentration range of 0.1mg/L to 100mg/L, in acetonitrile. Standard addition was also performed on two negative water and sports drink samples to demonstrate the capability of the instrument in detecting low levels of DEHP, commonly found in drinking water and sports drinks. Samples were spiked with 1ppm and 10ppm of DEHP prior to analysis. The column was kept at a constant 30°C temperature. The analysis was performed under isocratic conditions with water/acetonitrile (2/98 v/v) as the mobile phase. The flow rate was 1mL/min with a 10µL injection of all analytical standards and samples. The DAD was set at a single wavelength of 224nm.

Results

The calibration curve of DEHP, at a concentration range of 0.1mg/L to 100mg/L can be found in Figure 2.

DEHP exhibits excellent linearity over a wide concentration range, as demonstrated in Figure 2. DEHP was identified using the calibration standards with a retention time of 7.05 minutes.

Retention time and peak area repeatability of the LC6000 was tested with six consecutive injections of a 10mg/L standard, the values of which can be found in Table 1.

Excellent repeatability of both retention time and peak area was observed, with RSD% values at 0.011 and 0.11 respectively; highlighting the robustness of the SCION Instruments LC6000 HPLC with Diode Array Detector.

A mineral water and a commercially available 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 to each sample. Figures 3 and 4 show the overlay chromatograms of both samples, including the initial blank results and both DEHP additions.

The above figures demonstrate the excellent sensitivity of the SCION Instruments LC6000 with Diode Array Detector even at low concentrations such as 1ppm. The limit of detection was calculated using the criteria of signal to noise ratio of ten ($S/N = 10$). The limit of detection for DEHP was 0.10mg/L. The accurate identification and quantification of DEHP in drinking water is critical, especially during water quality analysis. The rapid method, developed by SCION Instruments provides a quick and easy solution for the rapid screening of water sources to ensure the drinking water is safe.



Figure 1. SCION Instruments LC6000

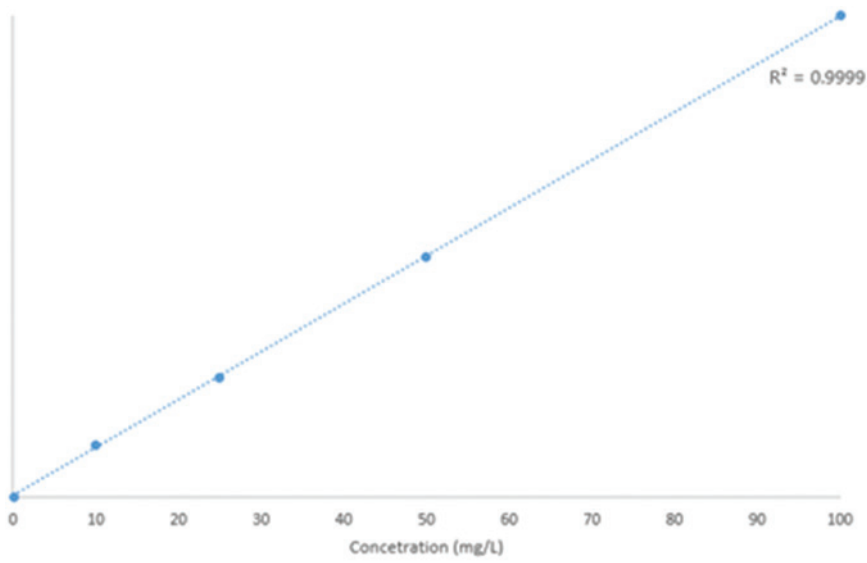


Figure 2. Calibration curve of DEHP; 0.1mg/L to 100mg/L

Table 1. Repeatability values of DEHP (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

Conclusion

SCION Instruments offers the ideal solution for the identification of DEHP, a restricted contaminant commonly found in drinking water. Excellent separation, linearity over a wide concentration range and system repeatability was observed for DEHP using a C18 column at wavelength 224nm. With an analysis time of only seven minutes, this application allows a high throughput of analysis for the most demanding water quality control laboratories and food manufacturers.

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.

For more information and to download the application note, please visit <https://scioninstruments.com/> or contact Ashleigh Mellor, LC Product Manager.

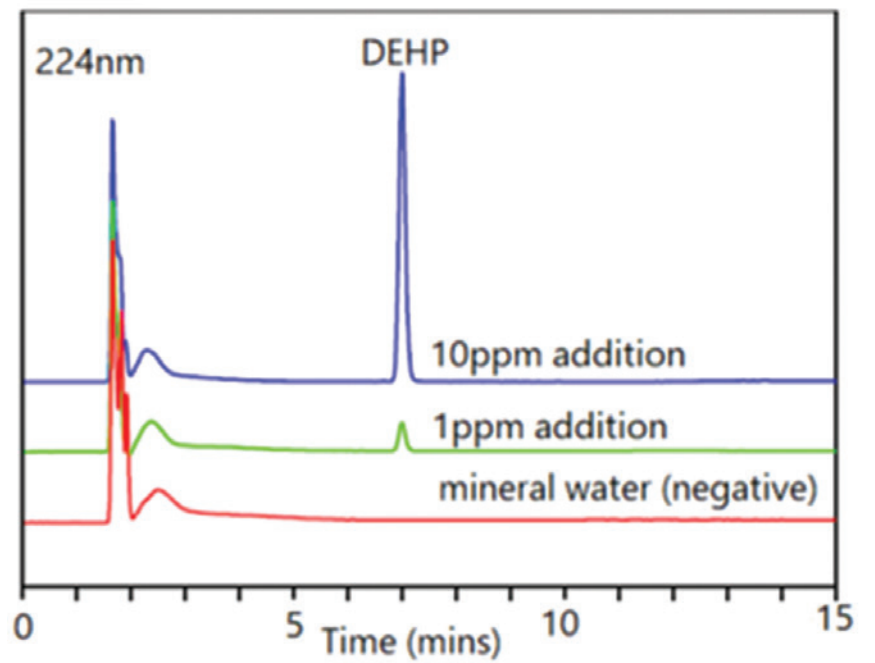


Figure 3. Chromatogram overlay of DEHP standard addition (mineral water)

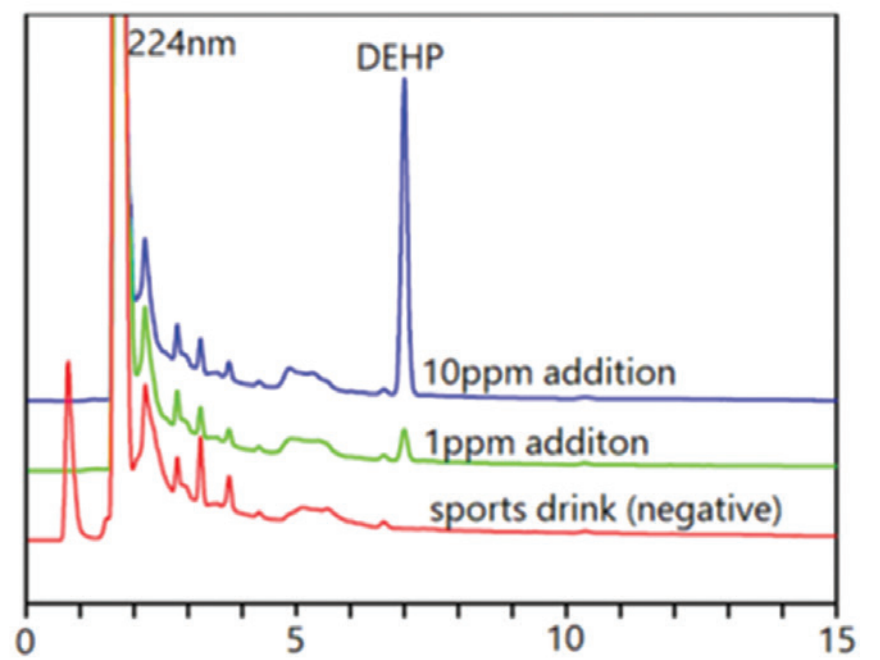


Figure 4. Chromatogram overlay of DEHP standard addition (sports drink)

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