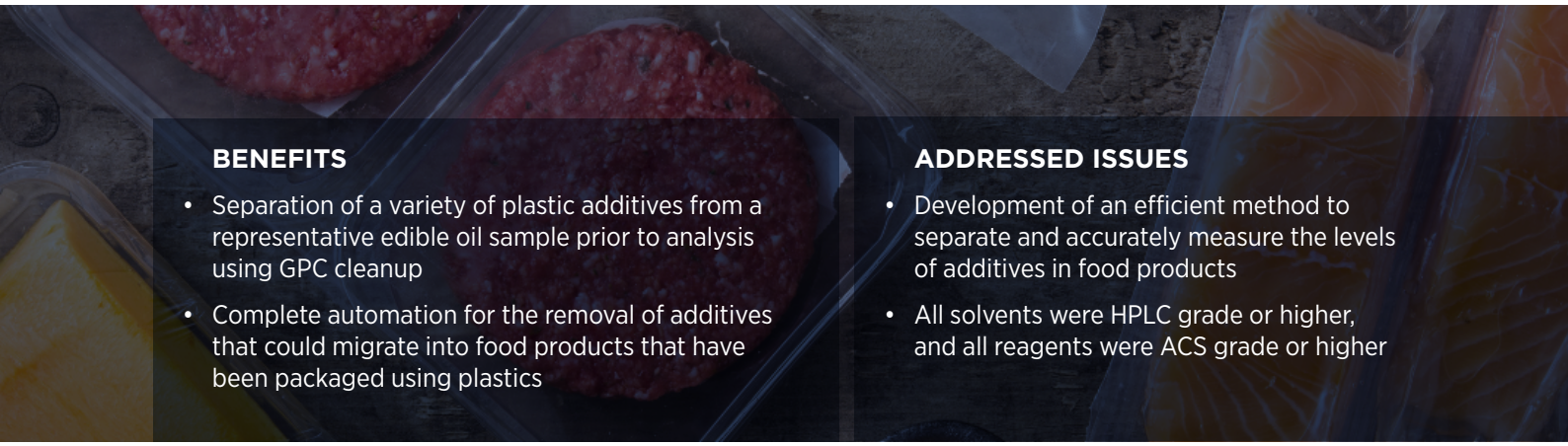
CLEANUP OF PLASTICS ADDITIVES FROM EDIBLE OILS USING GEL PERMEATION CHROMATOGRAPHY (GPC)

APPLICATION NOTE AN1052



BENEFITS

- Separation of a variety of plastic additives from a representative edible oil sample prior to analysis using GPC cleanup
- Complete automation for the removal of additives that could migrate into food products that have been packaged using plastics

ADDRESSED ISSUES

- Development of an efficient method to separate and accurately measure the levels of additives in food products
- All solvents were HPLC grade or higher, and all reagents were ACS grade or higher

GILSON APPLICATIONS TEAM

INTRODUCTION

Plastics are ubiquitous in our society and are extensively used in a large variety of packaged foods. A variety of chemicals that enhance the physical quality of plastics are often added as well. Some commonly used additives include plasticizers (such as phthalates) to improve flexibility, UV filters for protection from sunlight, brightening agents, coloring agents, and preservatives. Enhanced by a number of environmental factors, some of these additives may migrate into food products that have been packaged using plastics. There is currently a great deal of interest in measuring the migration of these compounds into food products, as considerable uncertainty exists regarding the human health risks associated with exposure to such compounds.[1]

Gel permeation chromatography (GPC) is a highly effective method for the removal of high molecular weight interferences such as lipids from a fatty food prior to analysis for the compounds of interest. GPC has been used to cleanup fatty foods contaminated with plasticizers and other plastics additives.

Figure 1
VERITY® GPC Cleanup System

Table 1
Plasticizer Additives Used in the Study.

Compound	Trade name or Abbreviation	Formula Weight
Dimethyl phthalate	DMP	194.19
Diethyl phthalate	DEP	224.24
Dibutyl phthalate	DBP	278.35
2-ethyl hexyl phthalate	DEHP	390.56
Butyl benzyl phthalate	BBP	312.36
Diisobutyl phthalate	DIBP	278.35
Diisononyl phthalate	DINP	418.61
Diphenyl phthalate	DPP	318.33
Di-n-octyl phthalate	DNOP	390.56
Octadecyl 3- (3,5-di-tert-butyl-4-hydroxyphenyl) proprionate	Irganox® 1076	530.88
2-Hydroxy-4 (octyloxy)-benzophenone	Chimassorb® 81	326.4
2,5 - Bis (5-tert-butyl-2-benzoxazolyl) thiophene	Uvitex® OB	430.57

This note describes the use of GPC cleanup to separate a variety of plastics additives, such as phthalates, Chimassorb® 81 (a UV absorber and stabilizer), Irganox® 1076 (an optical brightener) and Uvitex® OB (an antioxidant and thermal stabilizer) from a representative edible oil sample prior to analysis.

MATERIALS AND METHODS

All solvents were HPLC grade or higher. All reagents were ACS grade or higher. Phthalates and other plasticizer additives were obtained from Sigma-Aldrich (Table 1), and corn oil was obtained from a local market. A Phenomenex EnviroSep-ABC column was used with a mobile phase of 1:1 ethyl acetate/cyclohexane.

Methods – Sample Preparation

Standards were prepared in 1:1 ethyl acetate/cyclohexane. GPC calibration standards were prepared according to US EPA Method 3640A in 1:1 ethyl acetate/cyclohexane.

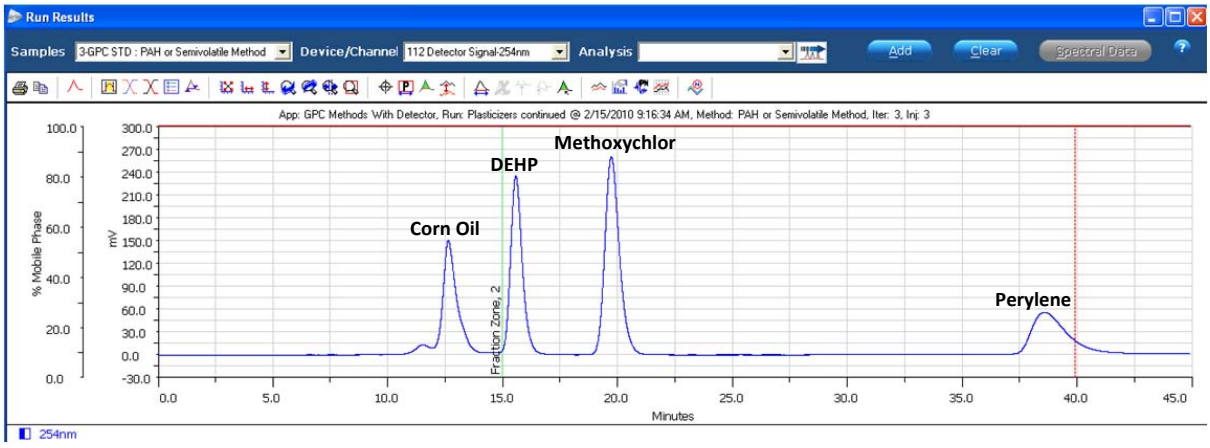


Figure 2
Chromatogram of a US EPA Method 3640A Calibration Standard.
Retention Times: Corn Oil = 12.6 min, DEHP = 15.6 min, Methoxychlor = 19.7 min and Perylene = 38.6 min.

Methods – GPC Cleanup Protocol

GPC separation was achieved using the VERITY GPC Cleanup System and a Phenomenex EnviroSep-ABC column for GPC with a mobile phase of 1:1 ethyl acetate/cyclohexane at a flow rate of 5 mL/min. The injection volume was 1 mL. The column was calibrated using a GPC calibration standard (described above), a Gilson 112 UV Detector set at 254 nm and Gilson TRILUTION® LC software to check for resolution and retention time values (Figure 2).

RESULTS

Chromatograms showing retention times of a common phthalate, DINP (Figure 3), and a UV absorber, Chimassorb 81 (Figure 4). These chromatograms were representative of the results observed for all plastics additives measured using the GPC cleanup protocol described.

All plastic additives separated from the representative edible oil (corn oil), as shown below in Table 2.

Table 2

Retention Times for Plasticizer Additives, Corn Oil and GPC Standards (For Abbreviation Key See Table 1).

Compound	RT (min)
Corn Oil	12.6
Irganox 1076	13.7
DINP	15.1
DNOP	15.5
DEHP	15.6
DIBP	17.1
Uvitex OB 17.5	17.5
DBP	17.6
Chimassorb 81 17.7	17.7
BBP	18.5
DEP 19.3	19.3
Methoxychlor (GPC Std)	19.7
DPP	19.9
DMP	21
Perylene (GPC Std) 38.6	38.8

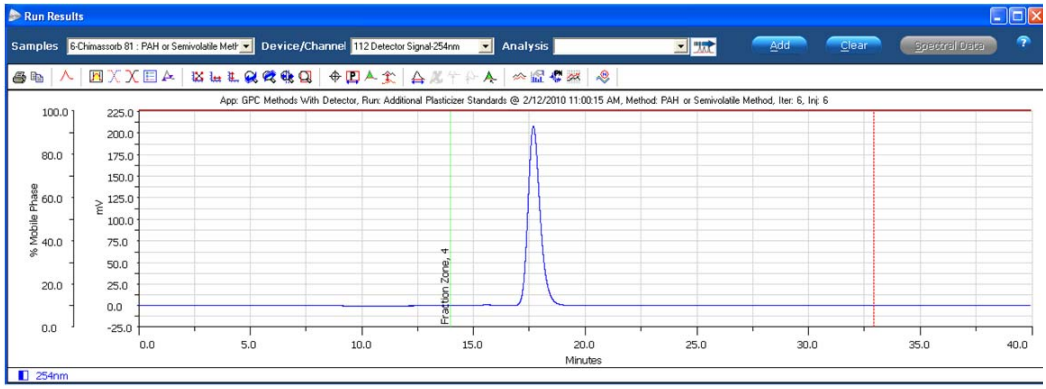


Figure 3

Chromatogram Showing Retention Time of Chimassorb 81.

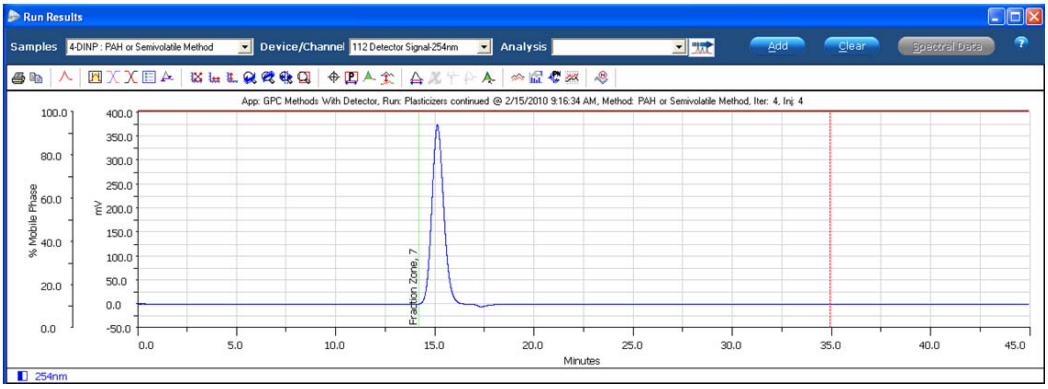


Figure 4

Chromatogram Showing Retention Time of Diisononyl phthalate (DINP).

SUMMARY

Gel permeation chromatography using the VERITY GPC Cleanup System is an efficient cleanup technique to separate phthalates and other plastics additives from fatty foods including edible oils, as shown in this application note. As concern over the potential toxicity and adverse human health effects associated with inadvertent exposure to chemicals compounded with plastic food packaging grows, so does demand for the development of efficient methods by which to separate and accurately measure the levels of such additives in food products.

REFERENCES

1 Meeker, J.D., Sathyanarayana, S., Swan, S.H. Phthalates and other additives in plastics: human exposure and associated health outcomes. Phil. Trans. R. Soc. B (2009) 364: 2097-2113.