

# Determination of Phthalates in vegetables by GPC–GCMS

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### Introduction

Phthalates (PAEs) are a class of compounds which can be added to plastics to increase its flexibility, transparency, durability and longevity. They can be used in electronics industry, agriculture adjuvant, building materials, toys, food packaging materials and textiles etc. Because of its medium viscosity, high stability, low volatility, easily accessible, low cost and other features, they are currently the most widely used plasticizer.

In 2011 PAEs events broke out in Taiwan drinks, and in 2012 the same thing happened to a certain brand of liquor. And recently it was reported that “vegetables wrapped in tape” in the supermarkets may contain PAEs. And this caused more and more consumers pay great

attention to the PAEs.

PAEs were classified as one kind of suspected environmental hormone. Their toxicity is mainly estrogen and anti-androgen activity which can cause endocrine disorder and reproductive function hinder in the organism. Therefore, PAEs had been restricted used in the relevant national standards such as drinking water, toys, packaging materials and food etc.

In this report a method was developed using Shimadzu’s GPC-GCMS to determine 22 kinds of PAEs in vegetables. This method is sensitive, easy to operate and can be applied to quickly detect PAEs in vegetables.

### Experimental

**Instrument:**

Shimadzu GPC-GCMS



Figure 1 Shimadzu GPC-GCMS

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## Experimental conditions:

GPC conditions:	
Chromatographic column	: Shodex CLNpak EV-200 (2.1 mm×150 mm)
Mobile phase	: acetone/cyclohexane (3/7, V/V)
Flow rate	: 0.1 mL/min
Column temperature	: 40°C
Sample size	: 20 µL
GCMS conditions:	
Chromatographic column	: inert quartz tube: 5 m×0.53 mm
Precolumn	: WondaCap WAX, 5m×0.25 mm×0.25 µm
Analytical column	: WondaCap WAX, 25m×0.25 mm×0.25 µm
Column oven temperature	: 82°C(5min)_8°C/min_150°C(0min)_25°C/min_240°C(5min)
Injection temperature	: 120°C(5min)_100°C/min_280°C(15.8min)
Pressure	: 120kPa(0min)_100kPa/min_180kPa(4.4min)_(-49.8 kPa/min)_120kPa (15.9min)
Purge flow	: 5.0mL/min_(-10mL/min)_0 mL/min(6min)_10mL/min_5mL/min (15.1min)
Sampling time	: 7min
Solvent cut time	: 9.7min
Interface temperature	: 250°C
Ion source temperature	: 200°C
Acquisition mode	: SIM, acquisition conditions are shown in Table 1

## Sample pretreatment:

Weigh accurately 1.0 g grinded vegetables into 25 mL centrifuge tube, add 5 mL Water, mix and exact for 30 min. Then add 2 mL Hexane, mix and vortex for 3 min, then take the supernatant fluid for sample analysis.

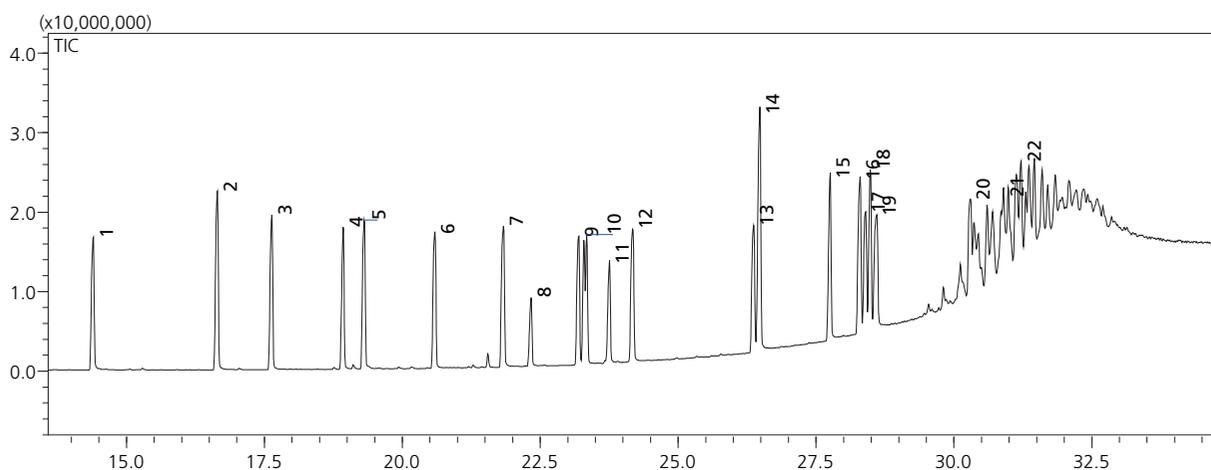


Figure 2 Total Ion Chromatogram of standard sample (1.0 mg/L)

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Table 1 Characteristic fragment ions of PAEs(m/z)

No.	Compound name	CAS	R.T.	Target Ion	Ref. Ion 1	Ref. Ion 2
1	DMP	131-11-3	14.400	163	133	194
2	DEP	84-66-2	16.642	149	177	176
3	DIPRP	605-45-8	17.633	149	209	150
4	DAP	131-17-9	18.925	149	104	189
5	DPRP	131-16-8	19.308	149	209	191
6	DIBP	84-69-5	20.583	149	167	205
7	DBP	84-74-2	21.833	149	205	223
8	DMEP	117-82-8	22.333	149	104	176
9	DIPP	605-50-5	23.192	149	219	237
10	BMPP	146-50-9	23.342	167	149	251
11	DEEP	605-54-9	23.750	149	104	176
12	DPP	131-18-0	24.175	149	219	237
13	DHXP	84-75-3	26.367	149	233	251
14	BBP	85-68-7	26.475	149	91	206
15	DBEP	117-83-9	27.750	149	101	193
16	DCHP	84-61-7	28.292	149	167	249
17	DHP	3648-21-3	28.392	265	149	247
18	DEHP	117-81-7	28.483	279	149	167
19	DPHP	84-62-8	28.592	225	104	153
20	DNOP	117-84-0	30.292	149	261	279
21	DINP	68515-48-0	30.908	293	149	127
22	DIDP	26761-40-0	31.217	307	149	141

## Result

### Calibration curve & Repeatability

Dilute the standard stock solution into 0.005, 0.05, 0.1, 0.5, 1.0 µg/mL. Some of the calibration curves obtained are shown in Figure 2. The correlation coefficients are >0.999, the RSD% of 7 consecutive tests of 0.05 µg/mL

standard samples are less than 5 % and the detection limit calculated according to the data of 0.005 µg/mL standard sample (3 S/N) are below 3 µg/mL.

### Sample & recovery results

Add PAEs standard (0.1, 0.2, and 0.4 mg/kg) into sample (lettuce, canola and celery) before sample pretreatment in accordance with the processing steps and calculate recovery rate. The results were between 60 % ~ 130 %.

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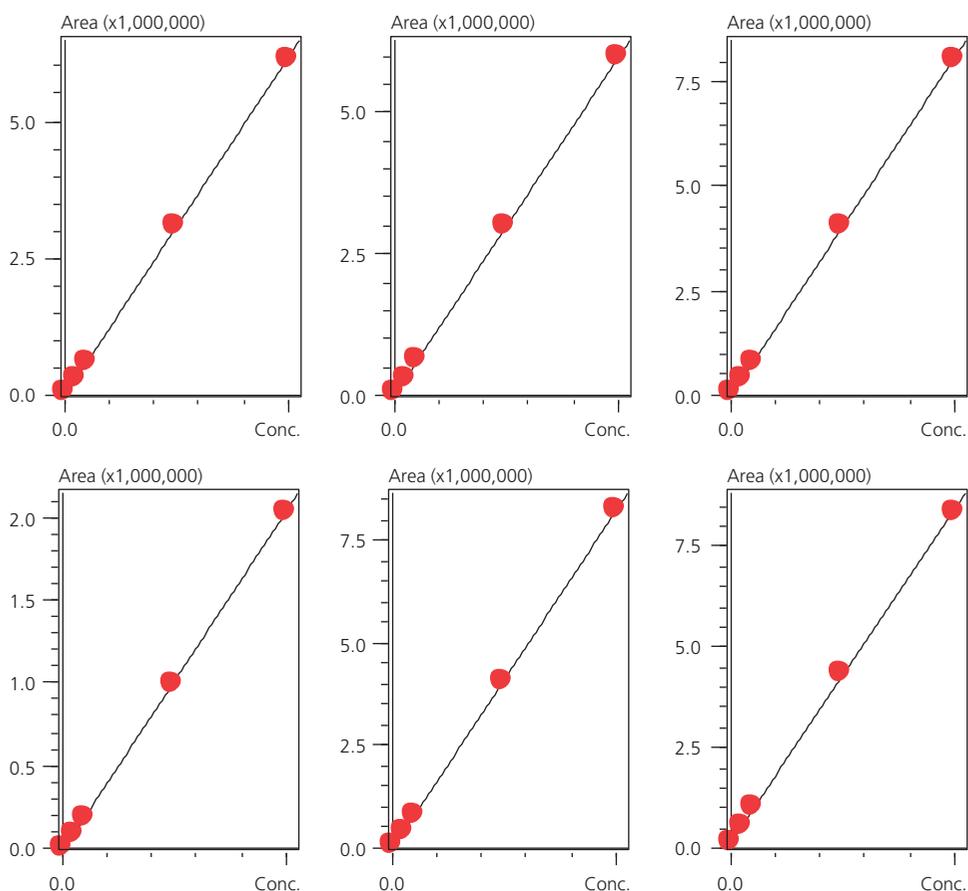


Figure 3 Calibration curve of some compounds

## Conclusions

A quick, easy and reliable method for determination PAEs in vegetables by Shimadzu's GPC-GCMS is developed. This method is sensitive, easy to operate and can be applied to quickly detect PAEs in vegetables.

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