

Fast Analysis of Phthalates

Using the Agilent 8850 GC/5977C GC/MSD with
hydrogen carrier gas and midcolumn backflushing



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Abstract

An Agilent 8850 GC system coupled with an Agilent 5977C Inert Plus GC/MSD, configured with hydrogen carrier gas and the Agilent Inert Plus source, was applied to the fast analysis of 19 phthalates. The high oven temperature programming rates available with the 8850 GC provided flexibility to run both conventional and rapid screening methods with the same hardware setup. With the appropriate choice of column stationary phase and oven ramp rate, the phthalates can be separated in 4.5 minutes. Midcolumn postrun backflushing was used to remove higher boiling matrix components in 0.75 minutes instead of using an extended bakeout time at the end of the run, which resulted in a total run time of 5.25 minutes. A conventional method with increased chromatographic resolution used the same hardware setup and a slower oven temperature program, with a total run time of 12.25 minutes. Having both methods available with the same setup allowed for fast screening and/or higher resolution for confirmation in the same analysis sequence. Calibration was performed for 17 of the 19 phthalates from 1 to 1,000 pg. With the conventional method, 15 were linear and two required quadratic fits, with all but four compounds having 1 pg as the lowest calibration level. With the fast method, five were linear and 12 required quadratic fits, with 13 compounds having 1 pg as the lowest calibration level. Diisononyl phthalate (DINP) and diisodecyl phthalate (DIDP), both mixtures of isomers, were calibrated from 50 to 20,000 pg and required a quadratic fit with both methods.

Introduction

Plastics are ubiquitous in the modern world and are used in thousands of applications. To tailor mechanical characteristics of plastics, such as flexibility, transparency, and durability for specific uses, esters of phthalic acid are often added to their formulation. These compounds are not covalently bound to the plastics in which they are mixed, and thus are easily released into the environment. They are present in a wide range of products including wire insulation, children's toys, packaging, pens, tubing, and more. Many countries now regulate phthalate content in a wide range of products. The analytical requirements for the analysis of phthalates vary depending on the region and products being tested, but GC/MS is usually the preferred measurement technique.

Given the potentially large number of samples that laboratories could be tasked to analyze, it would be valuable to have a fast-screening method to help prioritize samples for more in-depth analysis. It would also be helpful to have a method with higher chromatographic resolution for identity confirmation and quantitation using the same instrument hardware configuration.

This application note describes the development of both fast screening and conventional methods for the analysis of 19 common phthalates. The parameters considered for increasing the speed of the analysis included:

- Oven temperature program
- Column dimensions
- Column stationary phase
- Carrier gas
- Midcolumn postrun backflushing

Oven temperature program

Increasing the rate at which the oven temperature is programmed can be used to significantly reduce the analysis time. The 8850 GC has a smaller oven than conventional instruments and provides significantly faster temperature programming rates. The key is to optimize the program rate in concert with other chromatographic parameters to achieve the required separation when using the faster method.

Column dimensions

The length, diameter, and film thickness of the column can be optimized to yield the same separation with reduced run time. For example, using a 20 m × 180 µm diameter × 0.18 µm film thickness column in place of a 30 m × 250 µm diameter × 0.25 µm column can give the same separation in reduced time, albeit with some loss of capacity. For the analysis of phthalates, the capacity of the 180 µm diameter column is usually sufficient.

Column stationary phase

Stationary phase chemistry can often be used to adjust the separation and relative elution order of analytes to achieve the desired resolution. In this case, the phase typically used for phthalates resulted in coelution when the oven ramp rate was increased. An alternative, higher polarity phase was found to overcome this.

Carrier gas

While helium is generally considered the best carrier gas for GC/MS analysis, its relatively high cost and reoccurring shortages have increased demand for applications using hydrogen as an alternative. One important advantage with using hydrogen as a carrier gas is its ability to decrease the analysis time while maintaining chromatographic resolution. For example, by changing from a 250 µm diameter column with helium to a 180 µm diameter column with hydrogen, the same separation can be achieved in approximately 50% less time. When adopting hydrogen for GC/MS analysis, there are several things to consider. The Agilent EI GC/MS Instrument Helium to Hydrogen Carrier Gas Conversion user guide¹ describes the conversion steps in detail. The use of hydrogen carrier gas for analyzing the 19 phthalates investigated in this application note has been demonstrated previously.²

Midcolumn postrun backflushing

Residual matrix components extracted with the phthalates from samples can contaminate the column and result in ghost peaks from carryover in subsequent runs. This would normally require an extended postrun bakeout time to remove them. The technique of backflushing can be used to address this problem. With midcolumn backflushing, a connection with a controlled purge gas is inserted in the middle of the GC column. After elution of the last analytes, the inlet pressure is reduced, and the purge gas pressure is raised at the midcolumn connection. This causes the carrier gas to flow backwards through the first half of the column and forwards through the second half at a significantly higher rate than the analytical flow. This process quickly removes the high boiling compounds from the column and eliminates the need for extended postrun bakeout.

Experimental

The system used in these experiments was configured to minimize potential problems with the hydrogen carrier gas in the analysis of the phthalates. The important parameters used were:

- **Hydrogen gas:** In-house hydrogen (99.9999% purity) with low water and oxygen specifications was used as a carrier gas.
- **Pulsed splitless injection:** Used to maximize the transfer of phthalates into the column.
- **GC column:** An Agilent J&W DB-EUPAH column, 20 m × 0.18 mm, 0.14 µm (p/n 121-9627) was used to obtain separation of the phthalates at high oven temperature ramp rates. The column was cut in the middle, and the ends were attached to an Agilent purged Ultimate union (PUU) (p/n G3186-60581) to provide backflushing. Purge gas was supplied to the PUU with a pneumatic switching device (PSD) module in the 8850 GC to control the flow.
- **Inlet liner:** The Agilent Ultra Inert inlet liner, low pressure drop with glass wool (p/n 5190-2295), was found to give good overall performance for the phthalates.
- **MSD electron ionization (EI) source:** When converting to hydrogen carrier gas, the choice of EI source hardware is an important consideration.¹ For analytes that are subject to hydrogenation, the Agilent Hydrolnert source is strongly recommended because it is constructed from a material that greatly reduces the catalytic activity with hydrogen often seen in metals typically used in EI sources.¹

If the method is used to identify unknown compounds by library searching scan data against, for example, the NIST23 library, the Hydrolnert source would again be the source of choice. However, a large percentage of compounds analyzed by GC/MS do not exhibit reactions with hydrogen and can be analyzed using the standard Inert Plus source fitted with the optional 6 or 9 mm extractor lens.¹

In this application there are 19 target phthalates. Experimental data comparing spectral fidelity (based on NIST library match scores) and quantitative performance² showed that the standard Inert Plus source with the 9 mm lens gave results comparable to the Hydrolnert source. Therefore, the Inert Plus source was chosen for the method.

The instrument operating parameters are listed in Tables 1 and 2. Figure 1 shows the system configuration used in this experiment.

Table 1. GC and MS conditions for the analysis of phthalates.

Parameter	Value
GC	
GC	Agilent 8850 GC with an Agilent 7693A automatic liquid sampler
Inlet	Split/splitless inlet
Inlet Seal	Agilent Ultra Inert gold seal (p/n 5190-6144)
Mode	Pulsed splitless
Injection Pulse Pressure	25 psi until 0.75 min
Purge Flow to Split Vent	50 mL/min at 0.70 min
Injection Volume	1.0 µL
Syringe	Agilent Blue Line autosampler syringe, 10 µL, fixed needle, 23-26/42/cone, with PTFE-tip plunger (p/n G4513-80203)
Septum	Advanced Green septum, 11 mm (p/n 8010-0208)
Inlet Temperature	280 °C
Inlet Liner	Agilent Ultra Inert inlet liner, low pressure drop, glass wool (p/n 5190-2295)
Column	Agilent J&W BD-EUPAH column, 20 m × 0.18 mm, 0.14 µm. Cut into two 10 m sections. (p/n 121-9627)
Carrier Gas and Flow Rates	Hydrogen, column 1 = 0.9 mL/min constant flow, column 2 = 1.1 mL/min constant flow
12.25 min Temperature Program (°C)	50 °C (0.5 min hold), 50 °C/min to 220 °C (no hold), 12.5 °C/min to 300 °C (1.2 min hold), 0.75 min postrun
5.25 min Temperature Program (°C)	50 °C (0.5 min hold), 50 °C/min to 200 °C (no hold), 80 °C/min to 300 °C (1.75 min hold), 0.75 min postrun
Postrun Backflush Temperature	300 °C
Postrun Backflush Time	0.75 min
Postrun Backflush Flows	7.23 mL/min for column 1, 7.48 mL/min for column 2.
MSD	
MSD	Agilent 5977C GC/MSD with Inert Plus source and turbomolecular pump
Source	Inert Plus source with optional 9 mm extractor lens (p/n G3870-20449)
Transfer Line Temperature	300 °C
Ion Source Temperature	300 °C
Quadrupole Temperature	150 °C
EM voltage Gain mode	Gain, 1.0 for 4.5 min method. 2.0 for 11.5 min method.
Mode	SIM
Tune	ETUNE.U
Solvent Delay	1.7 min for 4.5 min method, 3.0 min for 11.5 min method.

Table 2. Names, abbreviations, CAS numbers, retention times, and target and qualifier ions of phthalates studied.

Name	Abbreviation	CAS No.	Target Ions (m/z)	Qualifier Ions 1 (m/z)	Qualifier Ions 2 (m/z)	Qualifier Ions 3 (m/z)
Dimethyl phthalate	DMP	131-11-3	163	77	194	133
Diethyl phthalate	DEP	84-66-2	149	177	105	222
Diallyl phthalate	DAP	131-17-9	149	41	132	189
1,2-Benzenedicarboxylic acid, bis(2-methylpropyl) ester	DIBP	84-69-5	149	223	167	104
Dibutyl phthalate	DBP	84-74-2	149	223	205	104
Bis(2-methoxyethyl) phthalate	DMEP	117-82-8	149	176	104	59
1,2-Benzenedicarboxylic acid, bis(4-methylpentyl) ester	BMPP	146-50-9	149	251	167	85
1,2-Benzenedicarboxylic acid, bis(2-ethoxyethyl) ester	DEEP	605-54-9	149	73	104	193
Diamyl phthalate	DPP	131-18-0	149	237	219	104
1,2-Benzenedicarboxylic acid, dihexyl ester	DHXP	84-75-3	149	104	233	251
Benzyl butyl phthalate	BBP	85-68-7	149	91	104	206
Bis(2-butoxyethyl) phthalate	DBEP	117-83-9	149	193	101	85
Dicyclohexyl phthalate	DCHP	84-61-7	149	167	104	249
Bis(2-ethylhexyl) phthalate	DEHP	117-81-7	149	167	113	104
Di-n-octyl phthalate	DNOP	117-84-0	149	279	104	261
Bis(2-propylheptyl) phthalate	DPHP	53306-54-0	149	55	167	307
1,2-Benzenedicarboxylic acid, dinonyl ester	DNP	84-76-4	149	293	275	150
Diisononyl phthalate	DINP	28553-12-0	293	149	167	
Diisodecyl phthalate	DIDP	26761-40-0	307	149	167	

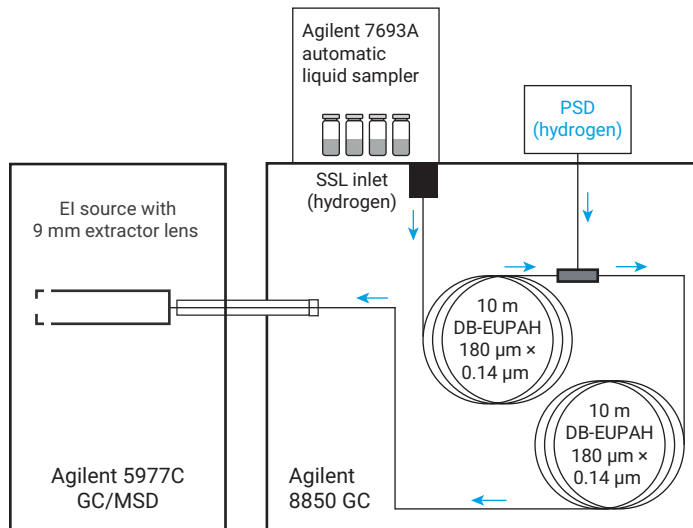


Figure 1. System configuration of an Agilent 8850 GC coupled with an Agilent 5977C GC/MSD.

Pulsed splitless injections are used to maximize the transfer of phthalates into the column and minimize unwanted interactions in the hot inlet. The Ultra Inert inlet liner, low pressure drop with wool (p/n 5190-2295), worked well for this application.

Phthalate calibration standards were prepared in two separate calibration sets. The first set contained 17 of the 19 phthalates and the second set contained DINP and DIDP phthalate isomers.

A 15-component custom phthalates mix was purchased from Ultra Scientific (now Agilent). The concentration of each component was 1,000 μg/mL in isooctane. Diallyl phthalate (DAP), DINP, and DIDP were purchased in pure form from Agilent. Pure bis(2-propylheptyl) phthalate (DPHP) was purchased from Millipore Sigma.

Calibration standards were prepared for 17 phthalates at 11 concentration levels: 1, 2.5, 5, 10, 20, 50, 100, 250, 500, 800, and 1,000 ng/mL in isooctane. Calibration standards for the DIDP and DINP isomers were dissolved in isooctane at 11 concentration levels: 50, 100, 250, 500, 750, 1,000, 2,500, 5,000, 7,500, 10,000, and 20,000 ng/mL. See Table 2 and Figure 2 for compound identifications. All quantitative measurements were performed with Agilent MassHunter quantitative analysis software, version 11.1.

Results and discussion

Column selection

Figure 2 shows the separation of 17 phthalates using a typical GC/MS method with helium carrier gas and a 0.25 mm column. Using hydrogen carrier gas and a 0.18 mm column with the appropriate oven ramp rate provides the

same resolution in less than half the time. Therefore, the Agilent J&W HP-5ms Ultra Inert 20 m × 0.18 mm column was evaluated for use with the desired fast oven ramp method.

As shown in Figure 3, increasing the oven ramp rates to make a faster method results in the complete coelution of DEHP and DCHP, which are two important targets for the method.

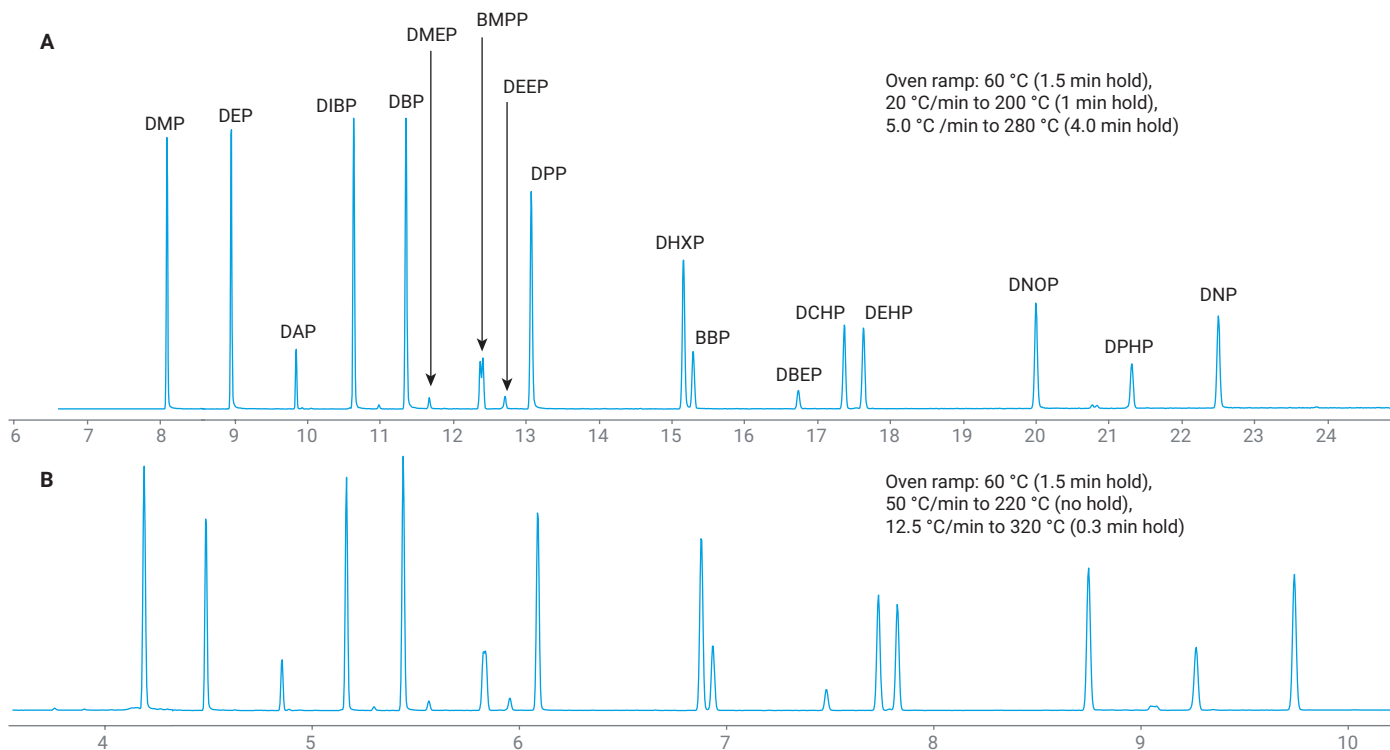


Figure 2. 1,000 ng/mL phthalate standard. (A) Agilent J&W HP-5ms Ultra Inert column, 30 m × 0.25 mm, 0.25 μm, with helium carrier gas. (B) Agilent J&W HP-5ms Ultra Inert column, 20 m × 0.18 mm, 0.18 μm, with hydrogen carrier gas. SIM m/z 163 quantifier for DMP and SIM m/z 149 quantifier for all others.

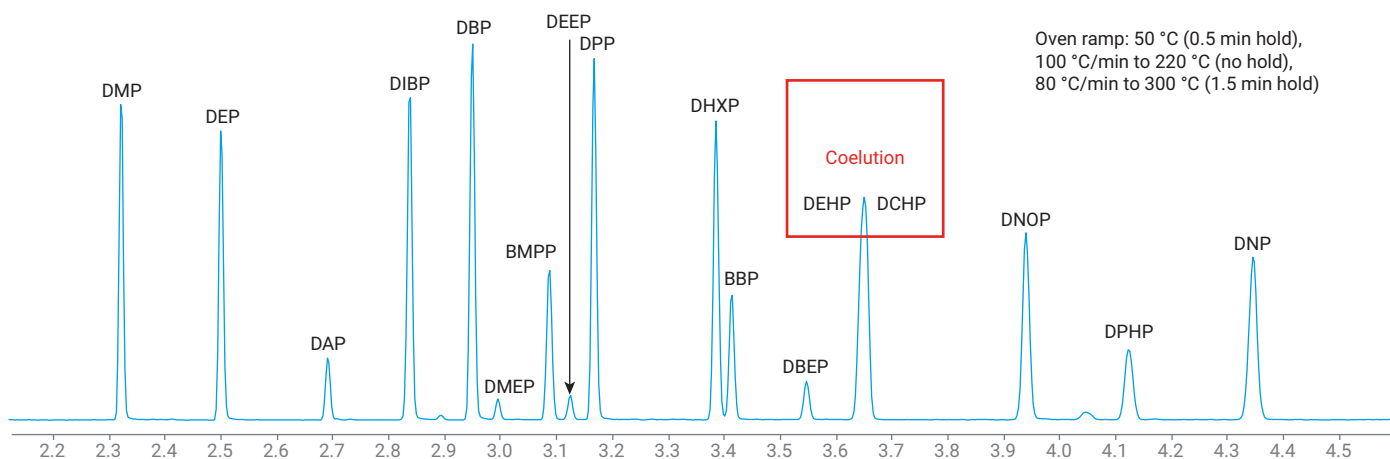


Figure 3. 1,000 ng/mL phthalate standard. Agilent J&W HP-5ms Ultra Inert column, 20 m × 0.18 mm, 0.18 μm, with hydrogen carrier gas and faster oven ramp. SIM m/z 163 quantifier for DMP and SIM m/z 149 quantifier for all others.

To address this, a column with a high arylene content, Agilent J&W DB-EUPAH, was tested. The DB-EUPAH column has the same length and diameter, but somewhat smaller film thickness (0.14 μm) compared to the HP-5ms Ultra Inert column.

Figure 4 shows the separation of 17 phthalates using a conventional oven ramp rate for higher resolution and using a very fast ramp rate.

The DB-EUPAH column with the conventional ramp rate provided similar resolution to that with the HP-5ms Ultra Inert column, but with a different elution order for some compounds. With the fast ramp method, all 17 phthalates were adequately resolved. Note that the elution order of DEHP and BBP reversed in the fast method. At intermediate ramp rates they coeluted, making the fast oven ramp a requirement for their resolution. The DB-EUPAH column was thus selected for further development of the method.

Midcolumn postrun backflushing

With the fast method, the last analyte of the 17 phthalates (DNP) eluted in less than 3.7 minutes. The oven run time was extended to 4.5 minutes to accommodate the complete elution of all the isomers of DIDP. Given the types of samples

from which the phthalates were extracted, higher boiling matrix components must be removed from the column to avoid contamination and ghost peaks in subsequent runs. An extended postrun bakeout time is often used to remove them. To avoid significantly increasing the method run time, midcolumn backflushing was used here instead. During analysis, the first 10 m section of the column had a flow rate of 0.9 mL/min of hydrogen, and the second section had a flow rate of 1.2 mL/min. The added 0.3 mL/min was supplied and controlled by the PSD. At the end of the analysis run, the oven postrun temperature was maintained at 300 $^{\circ}\text{C}$, the inlet pressure was reduced, and the purge gas flow was raised at the midcolumn connection by the PSD. The carrier gas flowed backwards through the first half of the column at 7.23 mL/min and forwards through the second half at 7.48 mL/min. These flows were maintained for 0.75 minutes, which was sufficient to remove any heavy matrix components for this setup. Note that a turbo pump was required for use with these flows.

Figures 5 and 6 show the chromatograms obtained with the fast and conventional methods using the DB-EUPAH column and midcolumn backflushing configuration.

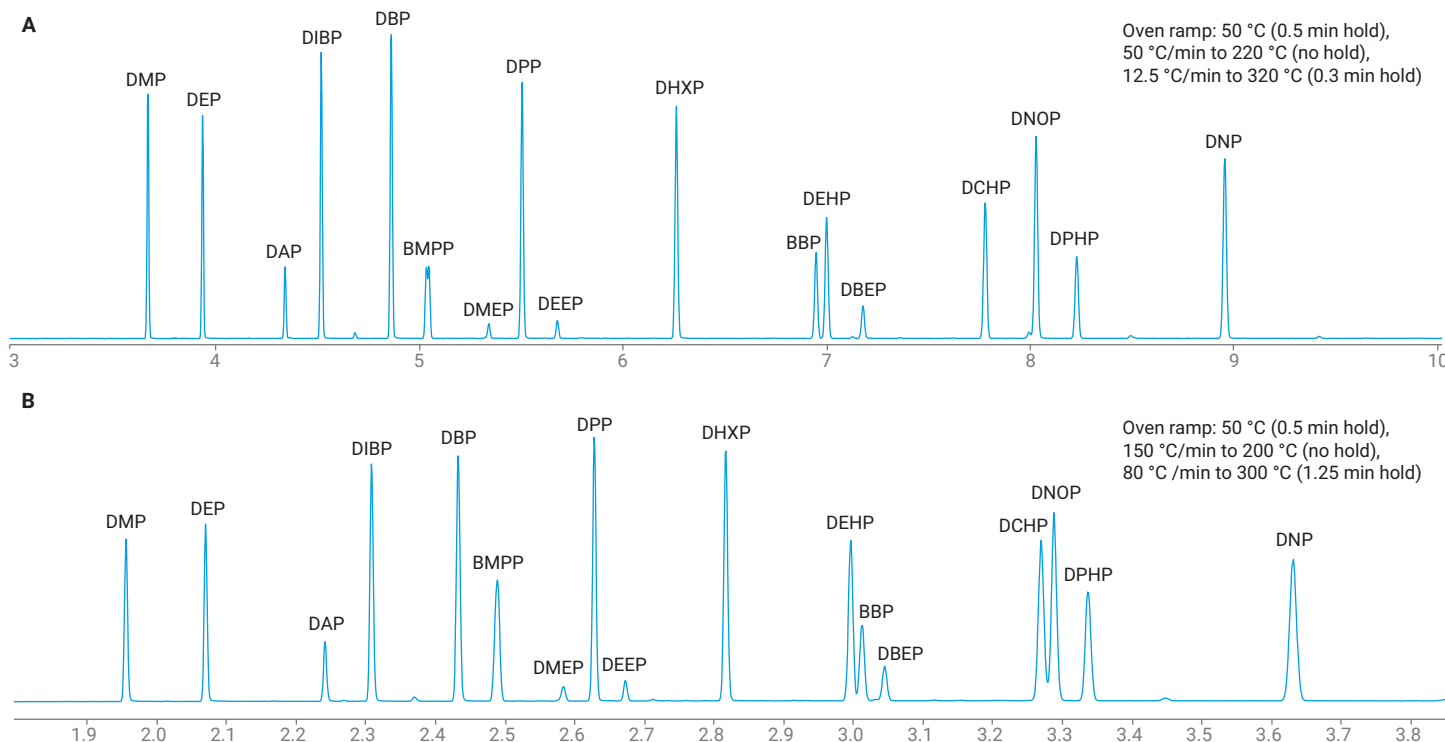


Figure 4. 1,000 ng/mL phthalate standard. (A) An Agilent J&W DB-EUPAH column, 20 m $\times$ 0.18 mm, 0.14 μm , with hydrogen carrier gas and nominal oven ramp. (B) Same column and hydrogen carrier gas flow with fast oven ramp.

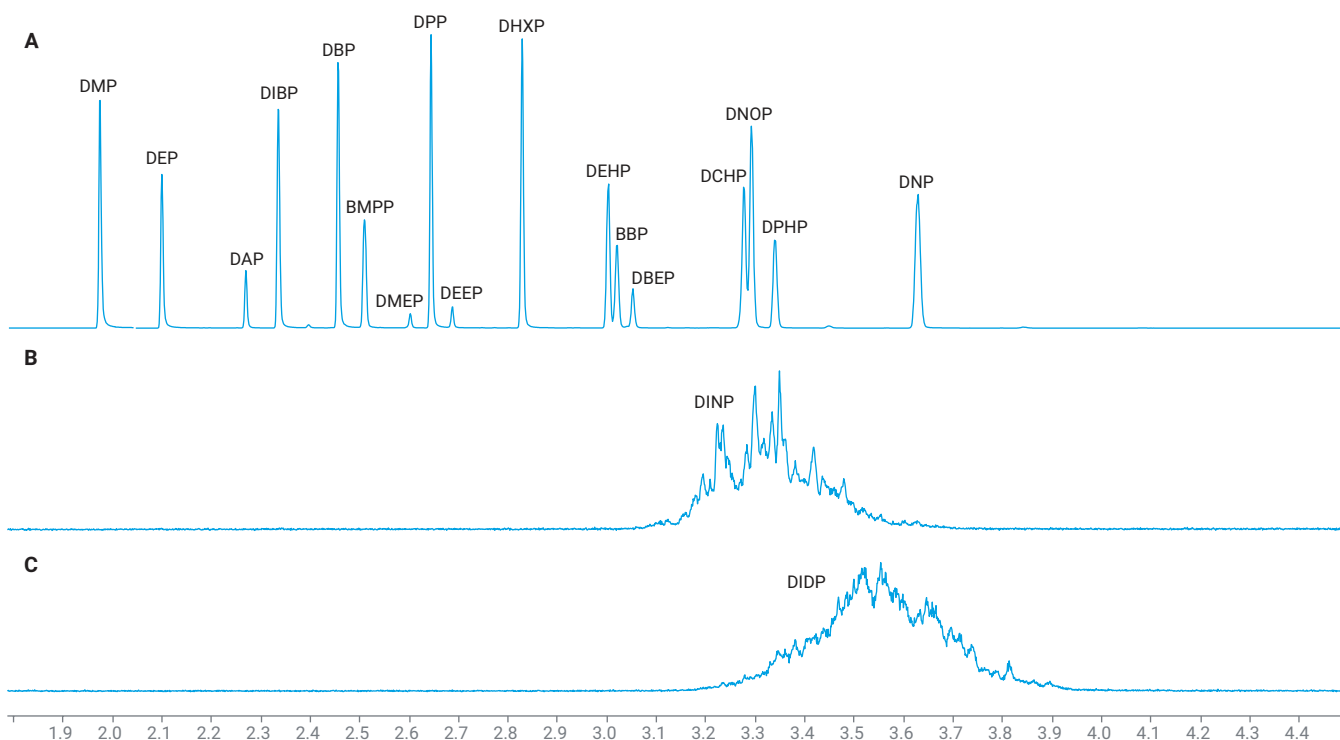


Figure 5. (A) Chromatogram of the 1,000 ng/mL 17 phthalate standard with the fast oven ramp method. SIM m/z 163 quantifier for DMP and SIM m/z 149 quantifier for all others. (B and C) 1,000 ng/mL DINP and DIDP phthalate standard. SIM m/z 293 quantifier for DINP and SIM m/z 307 quantifier for DIDP.

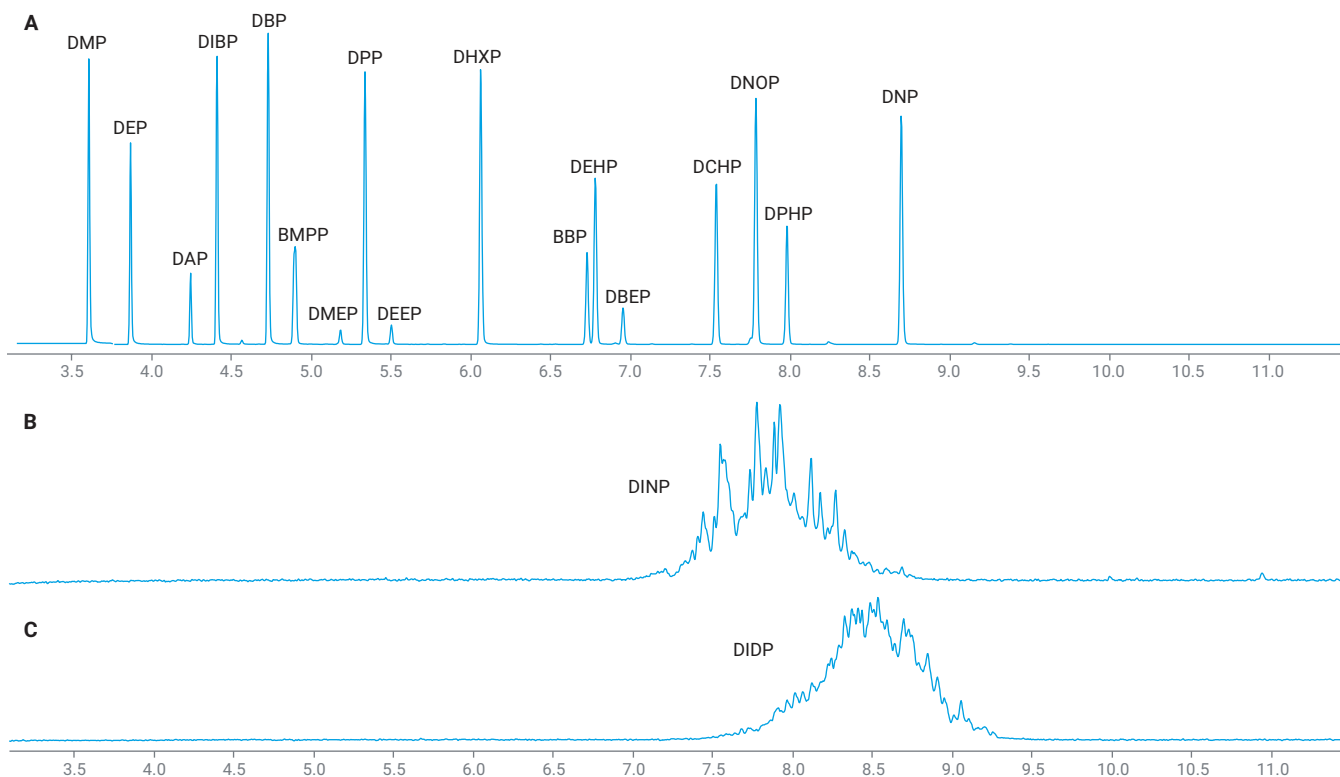


Figure 6. (A) Chromatogram of the 1,000 ng/mL 17 phthalate standard with the conventional oven ramp method. SIM m/z 163 quantifier for DMP and SIM m/z 149 quantifier for all others. (B and C) 1,000 ng/mL DINP and DIDP phthalate standard. SIM m/z 293 quantifier for DINP and SIM m/z 307 quantifier for DIDP.

Initial calibration and instrument detection limit

Calibration was performed for 17 of the 19 phthalates from 1 to 1,000 pg. With the fast method, five were linear and 12 required quadratic fits, with 13 compounds having 1 pg as the lowest calibration level. DINP and DIDP, both mixtures of isomers, were calibrated from 100 to 20,000 pg. DINP required a quadratic fit.

The relative standard error (RSE) value was used to guide the selection of a quadratic fit and/or removal of the lowest calibration points to achieve an RSE value of < 20%. The calibration results for the fast method are listed in Table 3.

An instrument detection limit (IDL) study was performed after completion of the initial calibration. Eight trials were performed with the 1 pg calibration standard for the 17 individual phthalates. The calculated IDLs were obtained by applying Equation 1. For compounds with lower signal-to-noise ratios, eight trials were performed with the 2.5 pg calibration standard for DAP, DEEP, and DBEP, and the 5 pg calibration standard for DMEP. Eight trials were performed with the 50 pg calibration standard for DINP and DIDP. Table 3 lists the calculated IDLs.

Equation 1. Formula for IDL calculations.

$$IDL = s \times t(n-1, 1-\alpha = 99) = s \times 2.998$$

Where:

$t(n-1, 1-\alpha) = t$ value for the 99% confidence level with $n-1$ degrees of freedom

n = number of trials (eight)

s = standard deviation of the eight trials

Calibration for the conventional method was also performed with the 17 phthalate standards from 1 to 1,000 pg. With this method, 15 compounds were linear and two required quadratic fits, with 13 compounds having 1 pg as the lowest calibration level. DINP and DIDP were calibrated from 50 to 20,000 pg and required a quadratic fit.

The RSE value was used to guide the selection of a quadratic fit and/or removal of the lowest calibration points to achieve an RSE value of < 20%. The calibration and IDL results for the conventional method are listed in Table 4.

Figures 7 and 8 show the calibration plots for the fast and conventional methods, respectively.

Table 3. Fast method results for 11 level SIM mode calibration over a range of 1 to 1,000 pg.

RT	Name	CF	CF Limit Low	CF Limit High	CF Weight	RSE	CF R2	Conc IDL	IDL (pg)
1.982	DMP	Quadratic	1	1000	1/x	10.3	0.999	1	0.23
2.097	DEP	Linear	1	1000	1/x	12.5	0.999	1	0.35
2.267	DAP	Quadratic	2.5	1000	1/x	7.8	0.999	2.5	0.41
2.333	DIBP	Linear	1	1000	1/x	11.6	0.998	1	0.17
2.453	DBP	Linear	1	1000	1/x	5.9	0.999	1	0.36
2.507	BMPP	Quadratic	1	1000	1/x	4.0	0.999	1	0.17
2.600	DMEP	Quadratic	5	1000	1/x	8.4	0.999	5	2.45
2.643	DPP	Linear	1	1000	1/x	8.6	0.998	1	0.15
2.685	DEEP	Quadratic	2.5	1000	1/x	8.4	0.999	2.5	1.32
2.827	DHXP	Linear	1	1000	1/x	10.9	0.998	1	0.19
3.001	DEHP	Quadratic	2.5	1000	1/x	6.0	0.999	1	0.64
3.019	BBP	Quadratic	1	1000	1/x	4.3	0.999	1	0.20
3.050	DBEP	Quadratic	2.5	1000	1/x	6.4	1.000	2.5	0.59
3.274	DCHP	Quadratic	1	1000	1/x	5.2	0.999	1	0.24
3.292	DNOP	Quadratic	1	1000	1/x	3.7	1.000	1	0.26
3.340	DPHP	Quadratic	1	1000	1/x	8.5	0.999	1	0.27
3.628	DNP	Quadratic	1	1000	1/x	5.9	1.000	1	0.28
3.400	DINP	Quadratic	100	20000	1/x	10.2	0.999	50	5.76
3.550	DIDP	Linear	100	20000	1/x	11.9	0.997	50	3.54

Table 4. Conventional method results for 11 level SIM mode calibration over a range of 1 to 1,000 pg.

RT	Name	CF	CF Limit Low	CF Limit High	CF Weight	RSE	CF R2	Conc IDL	IDL (pg)
3.553	DMP	Quadratic	1	1000	1/x	8.0	0.999	1	0.13
3.829	DEP	Linear	1	1000	1/x	5.4	0.999	1	0.51
4.207	DAP	Quadratic	1	1000	1/x	6.0	1.000	1	0.13
4.374	DIBP	Linear	1	1000	1/x	4.9	1.000	1	0.08
4.699	DBP	Linear	1	1000	1/x	11.9	0.999	1	0.25
4.862	BMPP	Linear	1	1000	1/x	5.4	0.999	1	0.17
5.152	DMEP	Linear	2.5	1000	1/x	8.9	0.999	2.5	1.35
5.308	DPP	Linear	1	1000	1/x	7.0	0.999	1	0.07
5.475	DEEP	Linear	2.5	1000	1/x	8.6	0.998	2.5	2.19
6.040	DHXP	Linear	1	1000	1/x	6.2	0.999	1	0.09
6.710	BBP	Linear	1	1000	1/x	5.6	0.999	1	0.15
6.759	DEHP	Linear	2.5	1000	1/x	7.1	0.999	1	0.31
6.937	DBEP	Linear	2.5	1000	1/x	7.5	0.999	2.5	0.85
7.524	DCHP	Linear	1	1000	1/x	5.2	1.000	1	0.15
7.771	DNOP	Linear	1	1000	1/x	3.5	1.000	1	0.21
7.970	DPHP	Linear	1	1000	1/x	4.3	1.000	1	0.15
8.690	DNP	Linear	1	1000	1/x	3.5	1.000	1	0.25
7.900	DINP	Quadratic	50	20000	1/x	3.4	1.000	50	9.3
8.500	DIDP	Quadratic	50	20000	1/x	9.6	0.999	50	5.7

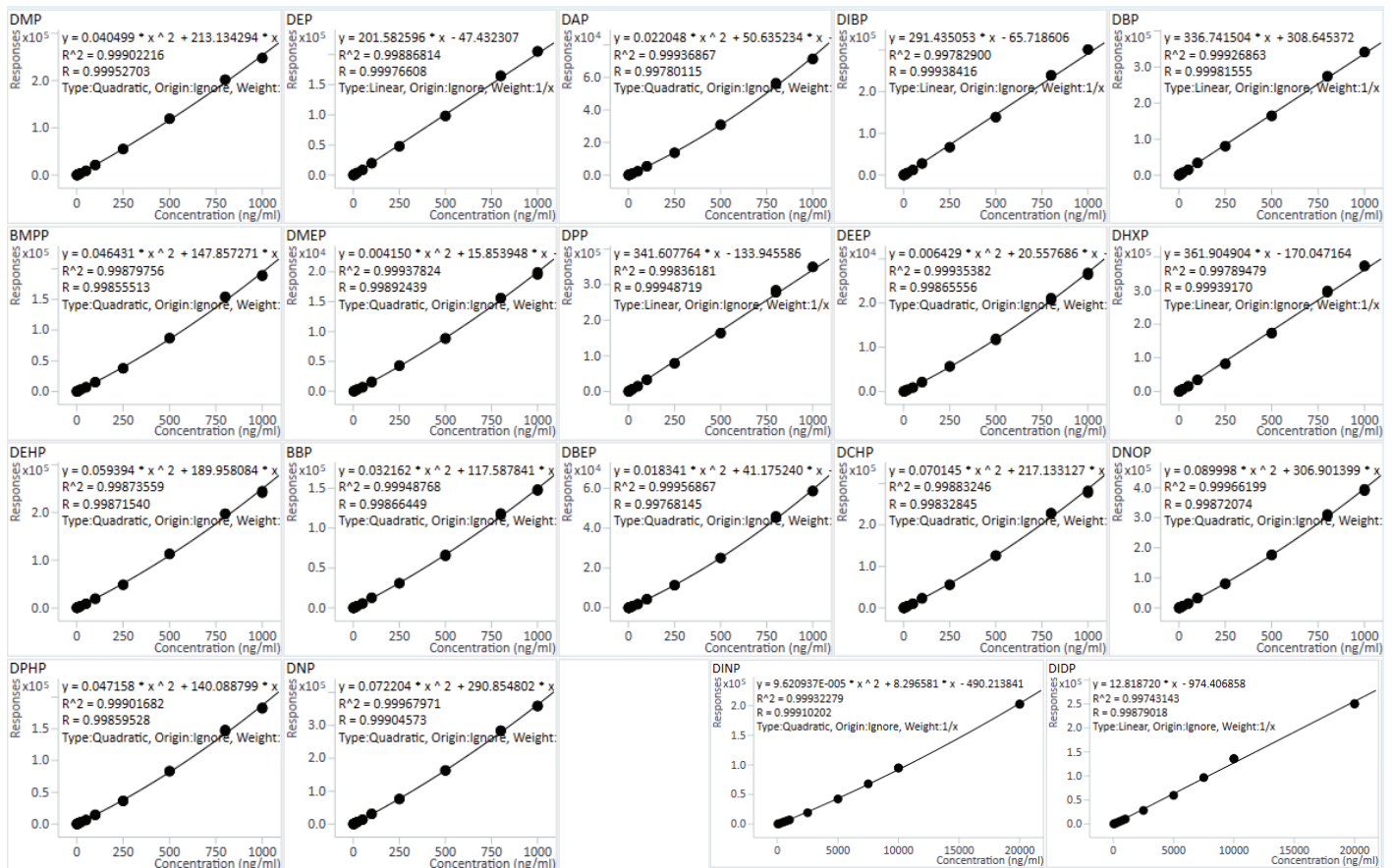


Figure 7. Calibration plots for the fast method.

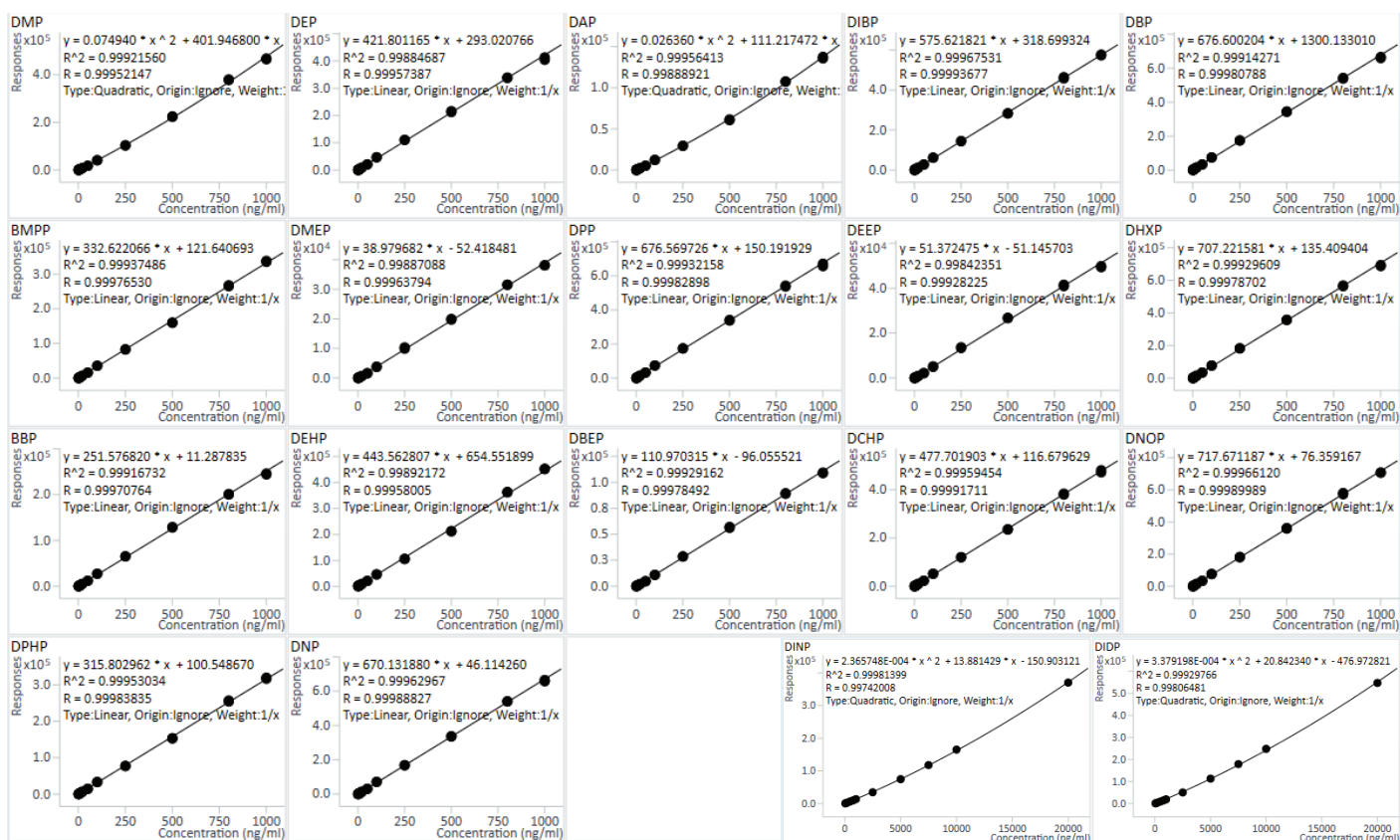


Figure 8. Calibration plots for the conventional method.

Example: Pen cap

As an example, an old pen cap was tested to rapidly screen for the 19 targeted phthalates using an Agilent 8850 GC coupled with an Agilent 5977C GC/MSD system. The pen is shown in Figure 9. Samples were obtained by slicing pieces of the cap with a solvent-cleaned hobby knife. The slices were placed in a 2 mL vial with 1 mL of iso-octane. The vial was capped with a PTFE crimp cap and vortexed vigorously for 1 minute. Approximately 50 μ L of the extract was transferred to an autosampler vial containing a flat-bottomed insert. All the consumables (except the polyurethane caps) were baked overnight at 130 $^{\circ}$ C, as described in reference 2 before use.² The vial was capped with a polyurethane snap cap and the extract was injected. This procedure is not quantitative; it is used to rapidly determine which, if any, of the 19 targets might be present and possibly warrant further testing.



Figure 9. Pen cap tested with fast and conventional methods. Samples were sliced off from the middle of the cap as visible in the photo.

Figure 10 shows the result for the pen cap with each method. The screening results indicated the presence of DEHP, DBP, DIBP, BBP, and DEP in the pen cap. Four of the five phthalates found here were also found in pen caps of different brands in reference 3. Only DEP was not reported in reference 3, as it was not a target, although it may have been present.

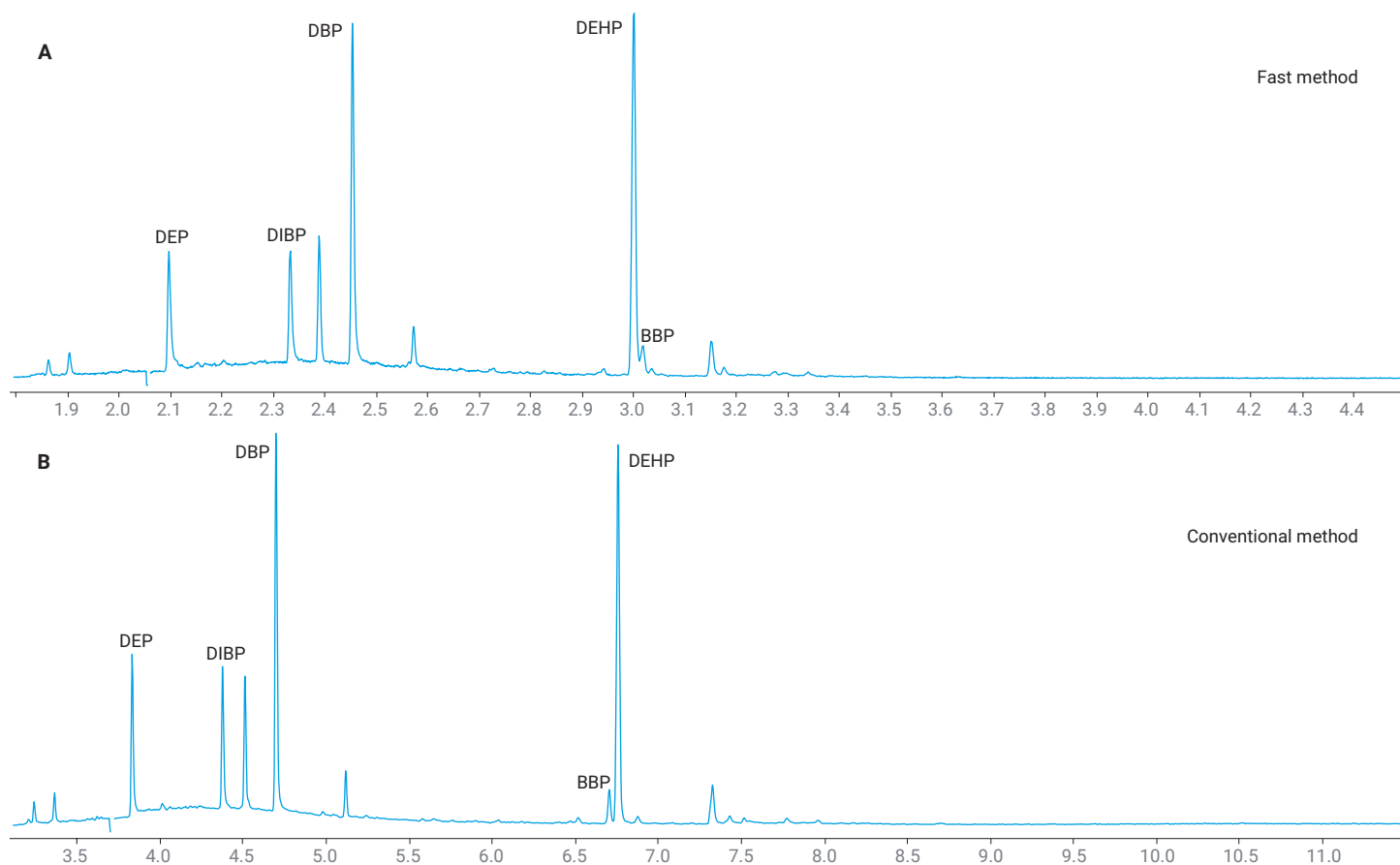


Figure 10. Pen cap analyzed with fast and conventional methods. SIM m/z 163 quantifier for DMP and SIM m/z 149 quantifier for all others.

Conclusion

The methods described in this application provide both fast and conventional GC/MS analysis with the same hardware setup. Having both methods available allows for fast screening and/or higher resolution for confirmation in the same analysis sequence. Two key factors in achieving adequate separation of the phthalates in a short time are the exceptional oven ramping capability of the Agilent 8850 GC and the choice of the Agilent J&W DB-EUPAH column. The speed of the overall analysis is further improved with the use of midcolumn backflushing to eliminate the need for long postrun bakeout times to remove heavy matrix components.

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