



GC-MS Analysis of Phthalates: Comparison of GC Stationary Phase Performance

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Abstract

Phthalates are ubiquitous in the environment and have attracted attention due to their potential adverse impact on human health. For this reason, detection and separation of phthalates has become a necessity. Gas chromatography is an effective approach for separating phthalates, and it can be paired with several detection techniques, including electron capture (ECD), flame ionization (FID), and mass spectrometry (MS). In this study, Pro EZGC gas chromatographic modeling software was employed to determine the optimal stationary phases and conditions for phthalate GC-MS analysis. The separation of phthalates was compared on seven different stationary phases: Rtx-440, Rxi-XLB, Rxi-5ms, Rtx-50, Rxi-35Sil MS, Rtx-CLPesticides, and Rtx-CLPesticides2 phases. In all cases, 18 EPA- and EU-listed phthalates were analyzed in less than 6 minutes. In addition, an extended list of 37 phthalates was analyzed in less than 40 minutes using an optimized method. Both Rtx-440, which is unique to Restek, and Rxi-XLB columns showed the best resolution of the complex phthalate mixture.

Introduction

Phthalates are widely used as plasticizers in a variety of industry products. However, some phthalates are considered to be endocrine disruptors [1] and are associated with a number of problems, including birth defects [2]; high blood pressure in children [3]; pregnancy-induced hypertensive heart diseases [4]; respiratory problems [5]; and obesity [6]. The European Union (EU) and United States Environmental Protection Agency (U.S. EPA) have restricted the use of the most harmful phthalates (Table I).

GC-MS is a commonly used technique for phthalate analysis because it is simple, fast, and inexpensive. GC-MS also provides mass spectral information, making it a powerful instrumental platform for phthalate determination. Using a GC column that provides good chromatographic separations is important because structural similarities among phthalates can make MS identification and quantification challenging. For instance, many phthalates share a common base peak ion (m/z 149), which makes identification and quantification of coeluting phthalates difficult. Technical grade mixtures and isomers further complicate the problem.

A recently published review summarized the most used GC and LC columns for phthalate analysis [7]. According to the literature, GC-MS has better chromatographic resolution compared to LC-MS for phthalate determination. The most commonly employed GC columns in descending order of popularity are 5-type, XLB-type, 35-type, 17-type, 50-type, and 1-type. The separation achieved on any stationary phase can be refined by adjusting instrument conditions, but this can be a time-consuming process when done in the laboratory. Pro EZGC modeling software can quickly optimize GC parameters (e.g., carrier gas type, flow rate, temperature program, column dimensions, and guard column) to produce the shortest analysis time on a given stationary phase. In this study, libraries of 37 phthalates (Table II) were built into the Pro EZGC program for seven stationary phases: Rtx-440, Rxi-XLB, Rxi-5ms, Rtx-50, Rxi-35Sil MS, Rtx-CLPesticides, and Rtx-CLPesticides2 phases. These stationary phases were evaluated for the analysis of both regulated and unregulated phthalates.

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Experimental Chemicals

Restek's EPA Method 8061A phthalates esters mixture (cat.# 33227), which contains 15 of the target analytes, each at a concentration of 1,000 µg/mL, was used as a primary reference standard. Benzyl benzoate (cat.# 31847) was used as the internal standard. All other phthalate standards were purchased from Chem Service.

Instrument

GC-MS analysis was performed on a Shimadzu QP2010 Plus GC-MS. The instrument was equipped with one of seven Restek columns using 30 m × 0.25 mm × 0.25 µm dimensions (0.20 µm for the Rtx-CLPesticides2 column). Windows-based Pro EZGC software was used to determine optimized conditions for each column and then all columns were directly compared by analyzing samples under the single set of conditions that had produced the best overall separation. This direct comparison provides a better understanding of the differences in selectivity among the columns. Detailed column descriptions and GC-MS experimental parameters are given in Table I and Table III, respectively.

Sample Preparation

Standards were dissolved and diluted in methylene chloride. Standard solutions were prepared at 50 µg/mL (80 µg/mL for the internal standard benzyl benzoate). During sample preparation, plastics were strictly avoided; all preparation work was performed using glassware (volumetric flasks, syringes, vials, etc.).

Results and Discussion

A direct comparison of column performance for the separation of EPA- and EU-regulated phthalates was performed. Phthalate retention times on seven different phases were predicted by the Pro EZGC program under the same GC conditions. These conditions, shown in Table I, were the conditions that produced the best overall chromatographic results among the separate sets of conditions that had been optimized for each column. Coelutions were defined as compound pairs with a resolution value of less than 1.5. The total analysis time was less than 6 minutes. In order to confirm the retention time predictions made by the Pro EZGC software, chromatograms were collected on each stationary phase under the same conditions used by the software (Figure 1). Because the column lengths were not exactly 30 meters long, as they were in the simulation, the absolute retention times were slightly different from the predicted values; however, the elution orders and coeluting pairs were exactly the same as predicted. Rtx-440, Rxi-XLB, Rtx-CLPesticides, and Rxi-35Sil MS columns provided baseline separation for all EPA- and EU-listed phthalates. The two isomers of bis[4-methyl-2-pentyl] phthalate were not resolved on any of the seven phases. The elution order was comparable on the Rtx-440, Rxi-XLB, Rtx-CLPesticides, and Rxi-5ms columns.

Differences in the elution orders were observed on Rxi-35Sil MS and Rtx-50 phases. Notably, the elution orders of four pairs of phthalates changed on the Rxi-35Sil MS phase, including bis(2-methoxyethyl) phthalate/bis(4-methyl-2-pentyl) phthalate isomers (peaks 6 and 7/8), bis(2-ethoxyethyl) phthalate/di-n-pentyl phthalate (peaks 9 and 10), butyl benzyl phthalate/hexyl-2-ethylhexyl phthalate (peaks 12 and 13), and bis(2-butoxyethyl) phthalate/bis(2-ethylhexyl) phthalate (peaks 14 and 15).

Rtx-440 and Rxi-XLB columns had the best overall separations under these conditions. Peaks that coeluted on other phases were well resolved on Rtx-440 and Rxi-XLB columns. Pairs that were not resolved on other phases include bis(2-ethylhexyl) phthalate and dicyclohexyl phthalate (peaks 15 and 16) on the Rxi-5ms column; bis(2-ethylhexyl) phthalate and butyl benzyl phthalate (peaks 15 and 12) on the Rtx-50 column; and bis(2-methoxyethyl) and bis[4-methyl-2-pentyl] phthalate (peaks 6 and 7,8) as well as bis(2-ethoxyethyl) phthalate and di-n-pentyl phthalate (peaks 9 and 10) on the Rtx-CLPesticides2 column. In technical grade isomer mixtures, it is possible to identify isomer groups, such as diisononyl phthalate and diisodecyl phthalate (peaks 18 and 19), but it is impossible to completely resolve each individual isomer within a group. Fortunately, unique extracted ions are available for identification and quantification, e.g., m/z 293 for diisononyl phthalate, and m/z 307 for diisodecyl phthalate (Figure 1).

A more comprehensive comparison of the seven stationary phases was also conducted for the separation of 37 phthalates (a total number of 40 peaks, including three isomers) using the retention times predicted by the Pro EZGC program (Table II). The GC parameters specified in Table III provided separation of 34 out of 40 peaks on both Rtx-440 and Rxi-XLB columns in less than 40 minutes and the two phases produced different coelutions. The chromatogram on the Rtx-440 column was collected and shown in Figure 2. For some pairs that were not baseline-resolved, the resolution is still adequate for qualitative analysis. There is no single set of conditions that is optimal for all phases. The program with the best overall results in terms of speed and the most peaks resolved was selected for this direct column comparison. Analysts can optimize conditions for their specific target lists using the Pro EZGC program. Based on the overall analysis speed and high degree of target analyte separation, the Rtx-440 and Rxi-XLB columns are recommended for phthalate GC-MS analysis.

Table I: Predicted Elution Times for Regulated Phthalates on Various Restek GC Columns

Column: 30 m x 0.25 mm x 0.25 µm (0.20 µm for Rtx-CLPesticides2 column)
 Constant linear velocity: 66.7 cm/sec
 Oven: 200 °C (hold 0.5 min) to 330 °C (320 °C for Rtx-50 column) at 30 °C/min (hold 1 min)

Peak #	Name	Listed	Retention Time (min)							CAS #	Purity
			Rtx-440 (cat. 12923)	Rxi-XLB (cat. 13723)	Rtx-CLPesticides (cat. 11123)	Rxi-35Sil MS (cat. 13823)	Rtx-50 (cat. 10523)	Rxi-5ms (cat. 13423)	Rtx-CLPesticides2 (cat. 11323)		
1	Dimethyl phthalate*	EPA 8061A, EPA Priority	1.28	1.16	1.14	1.29	1.46	1.10	1.23	131-11-3	Neat
2	Diethyl phthalate*	EPA 8061A, EPA Priority, EU	1.54	1.39	1.33	1.55	1.73	1.30	1.47	84-66-2	Neat
3	Benzyl benzoate	Internal Standard	2.11	1.87	1.56	2.17	2.31	1.70	1.88	120-51-4	Neat
4	Diisobutyl phthalate*	EPA 8061A	2.25	2.04	1.88	2.21	2.34	1.91	2.10	84-69-5	Neat
5	Di- <i>n</i> -butyl phthalate*	EPA 8061A, EPA Priority	2.58	2.33	2.10	2.53	2.69	2.17	2.38	84-74-2	Neat
6	Bis(2-methoxyethyl) phthalate*	EPA 8061A	2.74	2.48	2.26	2.86	3.10	2.27	2.63	117-82-8	Neat
7	Bis(4-methyl-2-pentyl) phthalate isomer 1*	EPA 8061A	2.85	2.62	2.37	2.71	2.83	2.50	2.64	84-63-9	Neat
8	Bis(4-methyl-2-pentyl) phthalate isomer 2*	EPA 8061A	2.86	2.63	2.37	2.72	2.84	2.51	2.65	84-63-9	Neat
9	Bis(2-ethoxyethyl) phthalate*	EPA 8061A	3.08	2.80	2.51	3.13	3.33	2.59	2.90	605-54-9	Neat
10	Di- <i>n</i> -pentyl phthalate*	EPA 8061A	3.16	2.91	2.58	3.08	3.21	2.71	2.89	131-18-0	Neat
11	Di- <i>n</i> -hexyl phthalate*	EPA 8061A	3.73	3.46	3.07	3.61	3.69	3.25	3.42	84-75-3	Neat
12	Butyl benzyl phthalate*	EPA 8061A, EPA Priority, EU	3.85	3.56	3.12	3.93	4.13	3.30	3.63	85-68-7	Neat
13	Hexyl 2-ethylhexyl phthalate	EPA 8061A	3.98	3.72	3.29	3.83	3.92	3.52	3.66	75673-16-4	Technical
14	Bis(2-butoxyethyl) phthalate*	EPA 8061A	4.12	3.82	3.39	4.08	4.21	3.60	3.85	117-83-9	Neat
15	Bis(2-ethylhexyl) phthalate*	EPA 8061A	4.21	3.95	3.52	4.05	4.12	3.82	3.91	117-81-7	Neat
16	Dicyclohexyl phthalate*	EPA 8061A, EPA Priority, EU	4.33	4.04	3.55	4.42	4.58	3.78	4.08	84-61-7	Neat
17	Di- <i>n</i> -octyl phthalate*	EPA 8061A, EPA Priority, EU	4.76	4.50	3.97	4.59	4.62	4.24	4.39	117-84-0	Neat
18	Diisononyl phthalate	EU	5.10	4.84	4.23	4.84	4.84	4.50	4.64	68515-48-0	Isomer mix
19	Diisodecyl phthalate	EU	5.20	4.95**	4.42	5.01	5.18	4.71	4.90	26761-40-0	Isomer mix
20	Dinonyl phthalate*	EPA 8061A	5.24	4.95**	4.39	5.04	5.10	4.72	4.83	84-76-4	Neat

Note: Shading indicates coeluting peaks ($R_s < 1.5$). For each column, different colors of shading indicate different coelution pairs.

*These compounds are contained in Restek's EPA Method 8061A phthalates esters mixture (cat.# 33227).

**Peaks 19 and 20 are non-isobaric and can be separated using selected ion monitoring.

Although GC-MS is generally preferred because it provides more definitive information, phthalate analysis can also be successfully accomplished by GC-ECD. EPA 8061A is a method used to identify and quantify phthalates in aqueous and solid matrices using a parallel column configuration and dual electron capture detectors [8]. Rtx-440 and Rxi-35Sil MS columns are ideal as a parallel dual column set. The Pro EZGC software provided rapid analysis conditions using the Rtx-440 column, and the Rxi-35Sil MS column served as an excellent confirmation column, due to the elution order changes that were observed. GC-ECD conditions can be easily translated from the GC-MS methods shown in Table III using Restek's free online EZGC method translator; alternatively, optimized GC-ECD instrument parameters and example chromatograms are available at <http://blog.restek.com/?p=17388> [9].

Table II: Predicted Elution Times for Phthalates (Extended List) on Various Restek GC Columns

Column: 30 m x 0.25 mm x 0.25 µm (0.20 µm for Rtx-CLPesticides2 column)

Constant linear velocity: 48 cm/sec

Oven: 150 °C (hold 0.8 min) to 200 °C at 5 °C/min to 275 °C at 3 °C/min (hold 2 min)

Peak #	Name	Listed	Rtx-440 (cat. 12923)	Rxi-XLB (cat. 13723)	Rxi-5ms (cat. 13423)	Rtx-50 (cat. 10523)	Rxi-35Sil MS (cat. 13823)	Rtx-CLPesticides (cat. 11123)	Rtx-CLPesticides2 (cat. 11323)	CAS #	Purity
1	Dimethyl phthalate*	EPA 8061A, EPA Priority	4.606	3.924	3.294	5.912	4.902	3.75	4.334	131-11-3	Neat
2	Dimethyl isophthalate	—	5.491	4.690	3.85	6.35	5.498	4.174	4.793	1459-93-4	Neat
3	Diethyl phthalate*	EPA 8061A, EPA Priority, EU	6.537	5.642	4.762	7.809	6.785	5.24	6.106	84-66-2	Neat
4	Benzyl benzoate	Internal Standard	9.931	8.667	N/A	11.099	N/A	6.725	8.583	120-51-4	Neat
5	Diisobutyl phthalate*	EPA 8061A	11.185	10.029	8.817	11.817	11.008	9.101	10.333	84-69-5	Neat
6	Di- <i>n</i> -butyl phthalate*	EPA 8061A, EPA Priority	13.152	11.850	10.405	14.031	13.094	10.481	12.029	84-74-2	Neat
7	Bis(2-methoxyethyl) phthalate*	EPA 8061A	14.343	12.784	11.045	17.095	15.424	11.54	13.725	117-82-8	Neat
8	Bis(4-methyl-2-pentyl) phthalate isomer 1*	EPA 8061A	15.192	13.754	12.47	15.184	14.454	12.166	13.825	84-63-9	Neat
9	Bis(4-methyl-2-pentyl) phthalate isomer-2*	EPA 8061A	15.350	13.828	12.55	15.277	14.542	12.233	13.906	84-63-9	Neat
10	Bis(2-ethoxyethyl) phthalate*	EPA 8061A	16.910	15.132	13.199	19.063	17.59	13.186	15.875	605-54-9	Neat
11	Di- <i>n</i> -pentyl phthalate*	EPA 8061A	17.454	15.880	13.856	17.974	17.128	13.588	15.768	131-18-0	Neat
12	Butyl cyclohexyl phthalate	—	19.452	17.689	15.478	21.19	19.843	14.979	17.96	84-64-0	Technical
13	Butyl 2-ethylhexyl phthalate	—	19.823	18.172	16.174	20.062	19.238	15.566	17.958	85-69-8	Technical
14	Di- <i>n</i> -hexyl phthalate*	EPA 8061A	22.138	20.279	17.984	22.152	21.469	17.215	19.829	84-75-3	Neat
15	Butyl octyl phthalate	—	22.338	20.557	18.136	22.37	21.668	17.344	20.009	84-78-6	Technical
16	Butyl benzyl phthalate*	EPA 8061A, EPA Priority, EU	22.799	20.783	18.029	25.365	23.782	17.384	21.128	85-68-7	Neat
17	Hexyl 2-ethylhexyl phthalate	EPA 8061A	24.404	22.668	20.266	24.110	23.500	19.126	22.049	75673-16-4	Technical
18	Butyl isodecyl phthalate	—	24.632	22.793	20.392	24.220	23.685	19.424	22.22	42343-36-2	Technical
19	Bis(2-ethylhexyl) hexahydro phthalate	—	25.066	23.389	21.254	23.089	23.063	19.142	21.961	84-71-9	Neat
20	Bis(2- <i>n</i> -butoxyethyl) phthalate*	EPA 8061A	25.601	23.563	20.930	26.746	25.647	19.849	23.533	117-83-9	Neat
21	Dicyclohexyl phthalate*	EPA 8061A, EPA Priority, EU	26.651	24.495	21.771	28.989	27.671	20.530	24.792	84-61-7	Neat
22	Bis(2-ethylhexyl) phthalate*	EPA 8061A	26.692	24.845	22.585	25.903	25.458	21.135	24.048	117-81-7	Neat
23	Butyl- <i>n</i> -decyl phthalate	—	27.362	25.268	22.657	26.888	26.410	21.404	24.471	89-19-0	Technical
24	Diphenyl phthalate	—	27.987	25.712	22.372	32.277	30.170	21.614	26.473	84-62-8	Neat
25	Bis(4-methylcyclohexyl) phthalate isomer 1	—	28.003	25.922	23.016	29.547	28.476	21.677	25.923	59-43-8	Isomer mix
26	Bis(4-methylcyclohexyl) phthalate isomer 2	—	29.002	26.993	23.816	30.345	29.400	22.604	26.739	59-43-8	Isomer mix
27	Hexyl isodecyl phthalate	—	29.176	27.271	24.523	28.189	27.965	23.224	26.336	61702-81-6	Technical
28	Benzyl 2-ethylhexyl phthalate	—	29.791	27.781	24.747	31.498	30.216	23.219	27.594	27215-22-1	Technical

(Continued from page 4.)

Table II: Predicted Elution Times for Phthalates (Extended List) on Various Restek GC Columns

Column: 30 m x 0.25 mm x 0.25 µm (0.20 µm for Rtx-CLPesticides2 column)

Constant linear velocity: 48 cm/sec

Oven: 150 °C (hold 0.8 min) to 200 °C at 5 °C/min to 275 °C at 3 °C/min (hold 2 min)

Peak #	Name	Listed	Rtx-440 (cat. 12923)	Rxi-XLB (cat. 13723)	Rxi-5ms (cat. 13423)	Rtx-50 (cat. 10523)	Rxi-35Sil MS (cat. 13823)	Rtx- CLPesticides (cat. 11123)	Rtx- CLPesticides2 (cat. 11323)	CAS #	Purity
29	Bis(4-methylcyclohexyl) phthalate isomer-3	—	29.964	28.034	24.617	31.189	30.285	23.498	27.559	59-43-8	Isomer mix
30	Bis(2-ethylhexyl) isophthalate	—	30.132	28.037	25.684	28.133	28.243	23.907	26.648	137-89-3	Neat
31	Bis(2-(ethoxyethoxy) ethyl) phthalate	—	30.233	28.434	24.879	32.942	31.252	23.995	28.681	117-85-1	Technical
32	Di- <i>n</i> -octyl phthalate*	EPA 8061A, EPA Priority, EU	31.562	29.626	26.796	30.475	30.328	24.915	28.455	117-84-0	Neat
33	<i>n</i> -Hexyl decyl phthalate	—	31.680	29.748	26.878	30.788	30.450	24.994	28.566	25724-58-7	Technical
34	Diphenyl isophthalate	—	32.362	29.850	N/A	34.707	32.396	25.114	29.437	744-45-6	Neat
35	Dibenzyl phthalate	—	33.234	30.725	27.141	37.396	35.372	25.501	31.359	523-31-9	Neat
36	Diisononyl phthalate	EU	33.684	31.802	28.779	32.500	32.708	27.391	30.811	68515-48-0	Isomer mix
37	Di- <i>n</i> -octyl isophthalate	—	34.483	32.463	29.168	32.035	N/A	27.223	30.388	4654-18-6	Neat
38	Diisodecyl phthalate	EU	35.775	33.792	30.876	35.041	N/A	29.11	32.169	26761-40-0	Isomer mix
39	Dinonyl phthalate*	EPA 8061A	36.159	34.103	30.994	34.609	34.705	28.867	32.604	84-76-4	Neat
40	<i>n</i> -Octyl- <i>n</i> -decyl phthalate	—	36.182	34.170	30.961	34.664	34.7	28.861	32.628	119-07-3	Technical

Note: Shading indicates coeluting peaks ($R_s < 1.5$). For each column, different colors of shading indicate different coelution pairs.

*These compounds are contained in Restek's EPA Method 8061A phthalates esters mixture (cat.# 33227).

Table III GC-MS Parameters

Parameters	Values for EPA EU List	Values for Extended List
Inlet temperature (°C)	280	280
Injection volume (µL)	1.0	1.0
Liner	Restek Premium 3.5 mm Precision liner w/ wool (cat.# 23320.1)	Restek Premium 3.5 mm Precision liner w/ wool (cat.# 23320.1)
Oven temperature program	200 °C (hold 0.5 min) to 330 °C* at 30 °C/min (hold 1 min)	150 °C (hold 0.8 min) to 200 °C at 5 °C/min to 275 °C at 3 °C/min (hold 2 min)
Carrier gas: He control mode	Constant linear velocity: 66.7 cm/sec @ 200 °C (3 mL/min**)	Constant linear velocity: 48 cm/sec @ 150 °C (1.6 mL/min)
Split ratio	20:1	20:1
Detector	MS	MS
Mode:	Full Scan (59–400)	Full Scan (59–400)
Transfer line temp.:	300 °C	300 °C
Scan event time	0.1 sec	0.1 sec
Analyzer type:	Quadrupole	Quadrupole
Source temp.:	280 °C	280 °C
Solvent delay time:	0.9 min	2.5 min
Tune type:	PFTBA	PFTBA
Ionization mode:	EI	EI

*320 °C for Rtx-50 column

**3 mL/min may be too high for some instruments. Consult your instrument operations manual prior to programming.

Conclusion

The seven GC columns most commonly used for phthalate analysis were directly compared using the Pro *EZGC* program, which provided flexibility and ease in GC optimization. The superior selectivity and high efficiency of the Rtx-440 and Rxi-XLB columns resulted in fast analysis times for both the regulated phthalates and the extended list. With good resolution, high maximum operating temperatures (340 °C for Rtx-440 and 360 °C for Rxi-XLB [Table IV]), and minimum phase bleed, the Rtx-440 and Rxi-XLB columns are the preferred choices for phthalate GC-MS analysis. A dual column set of Rtx-440 and Rxi-35Sil MS columns is recommended if GC-ECD is used instead of GC-MS.

Table IV: Maximum Temperatures

	Rtx-440 (cat. 12923)	Rxi-XLB (cat. 13723)	Rxi-5ms (cat. 13423)	Rtx-50 (cat. 10523)	Rxi-35Sil MS (cat. 13823)	Rtx-CLPesticides (cat. 11123)	Rtx-CLPesticides2 (cat. 11323)
Maximum Temperature (°C)	340	360	350	320	360	340	340

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Figure 1: EPA- and EU-listed phthalates and internal standard (benzyl benzoate) are shown under full scan mode and selected ion scan mode (m/z 293 and m/z 307) on seven different GC stationary phases (see Table III for conditions).

Rtx-440

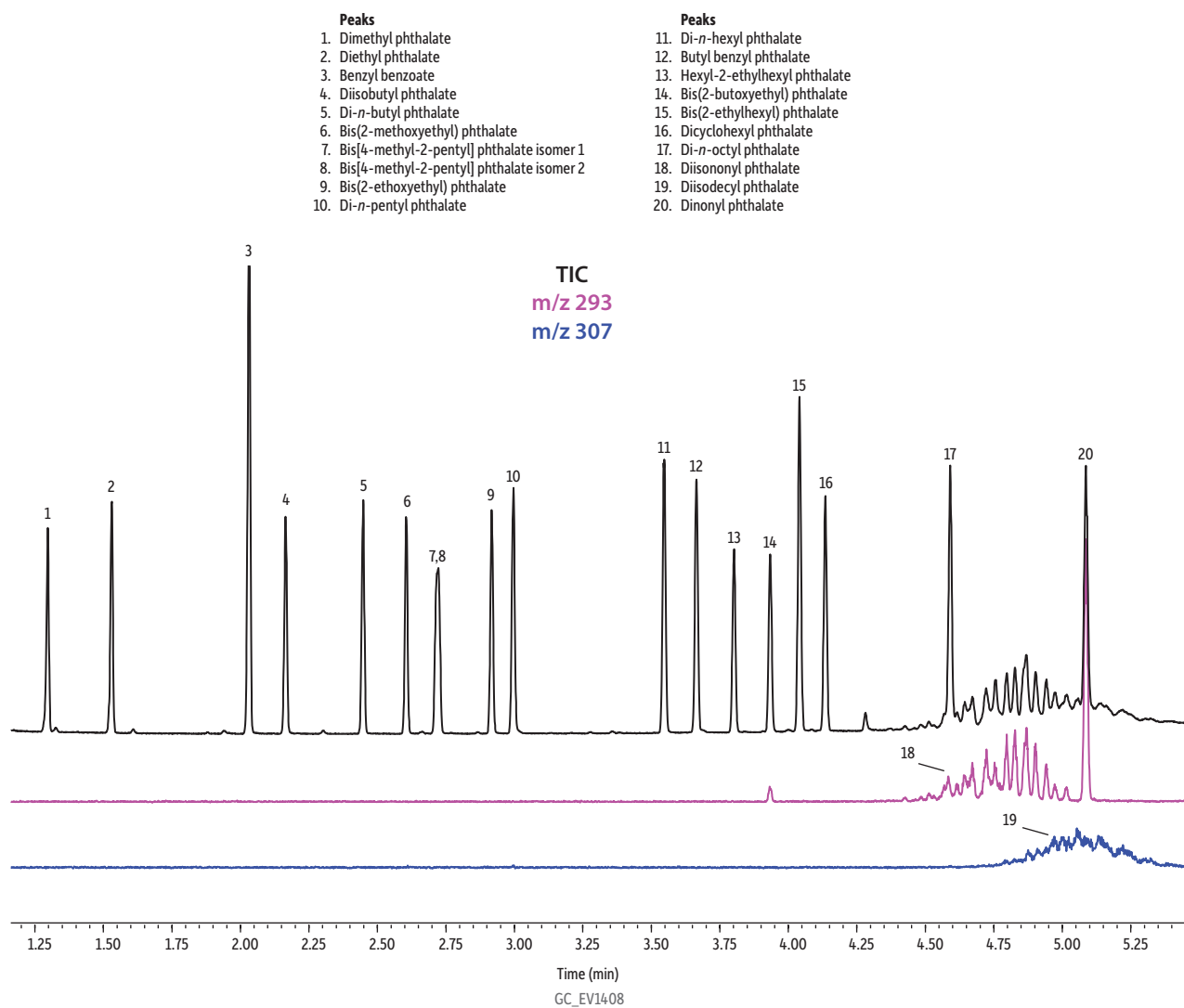
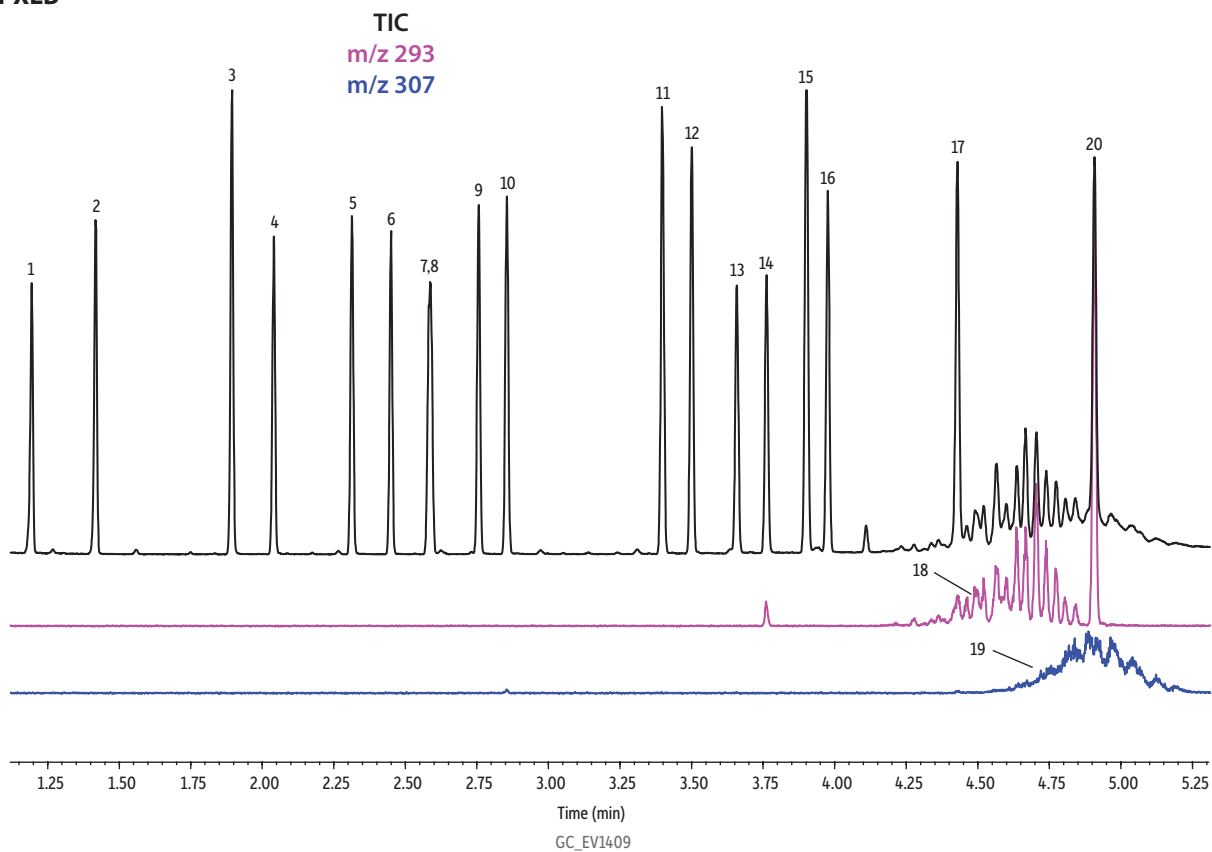


Figure 1 Continued

Rxi-XLB



Rtx-CLPesticides

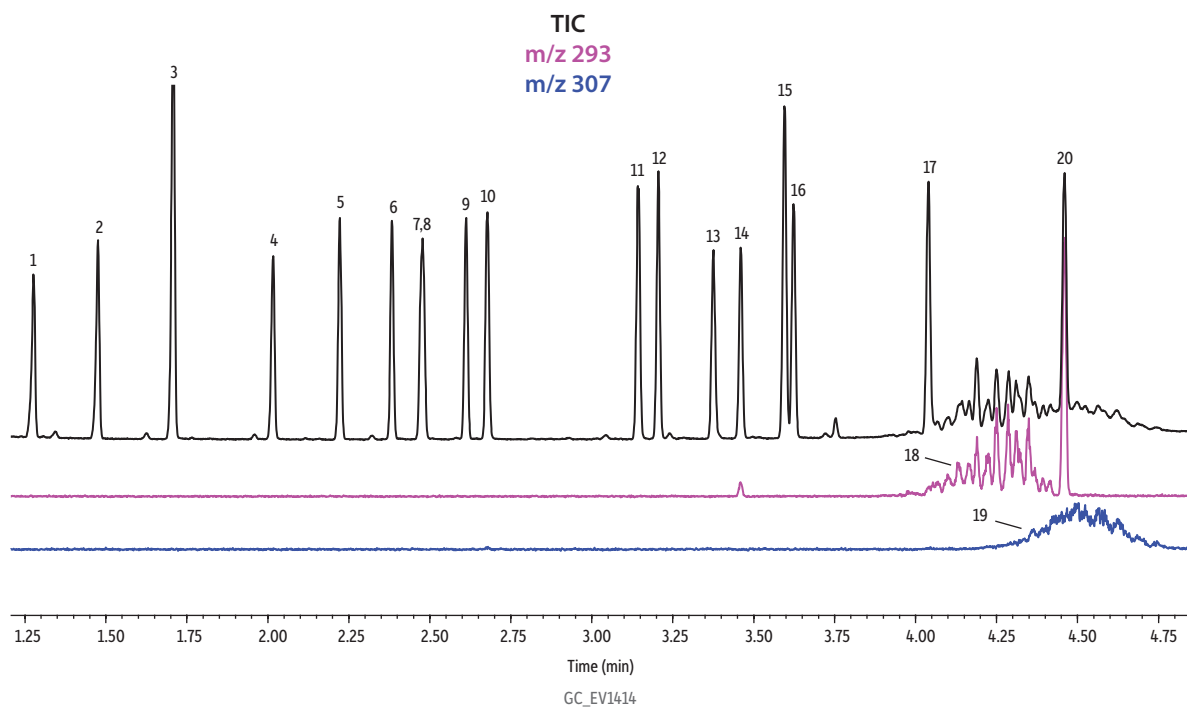
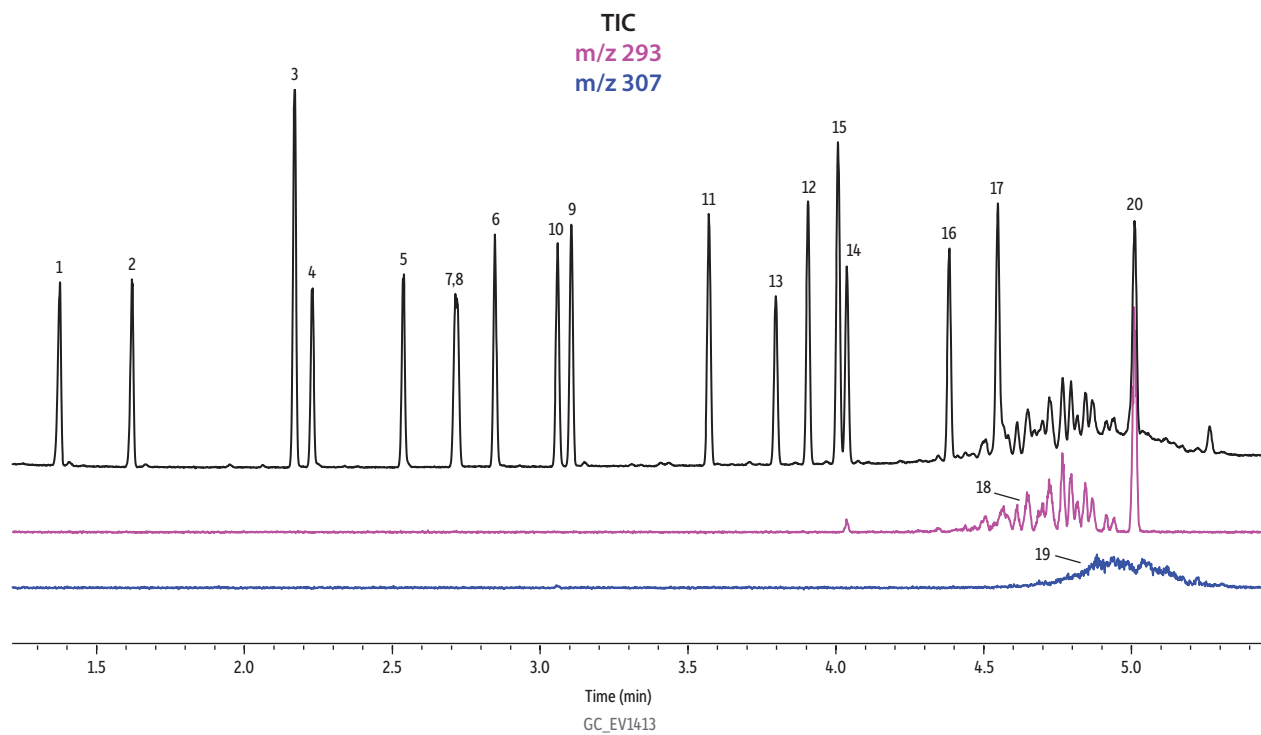


Figure 1 Continued

Rxi-35Sil MS



Rtx-50

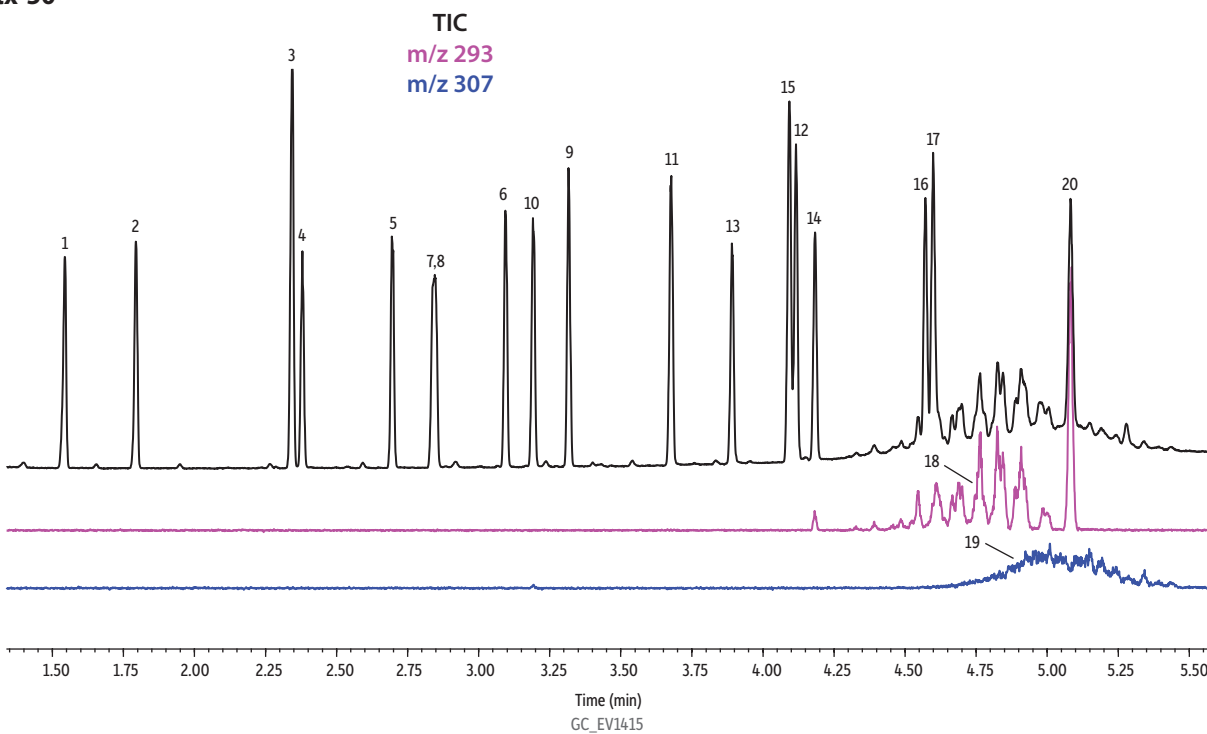
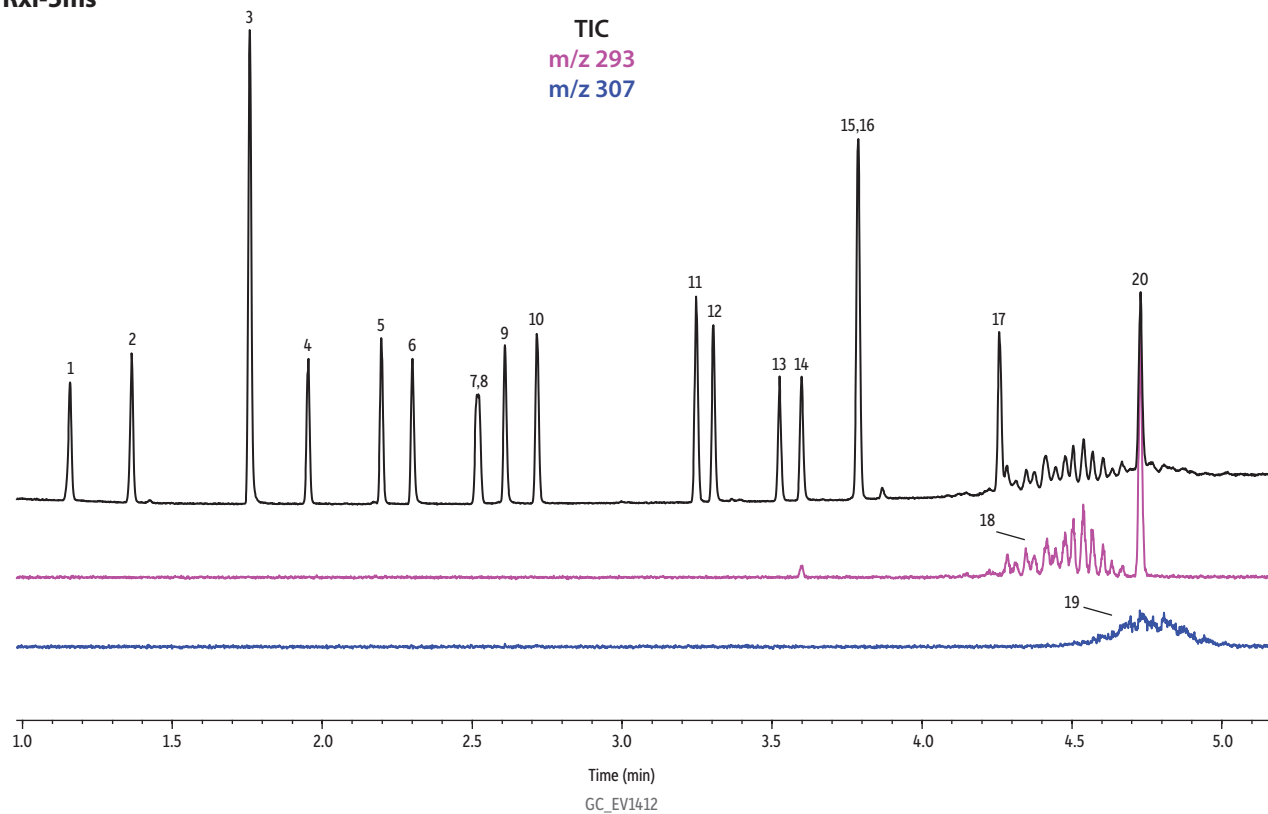


Figure 1 Continued

Rxi-5ms



Rtx-CLPesticides2

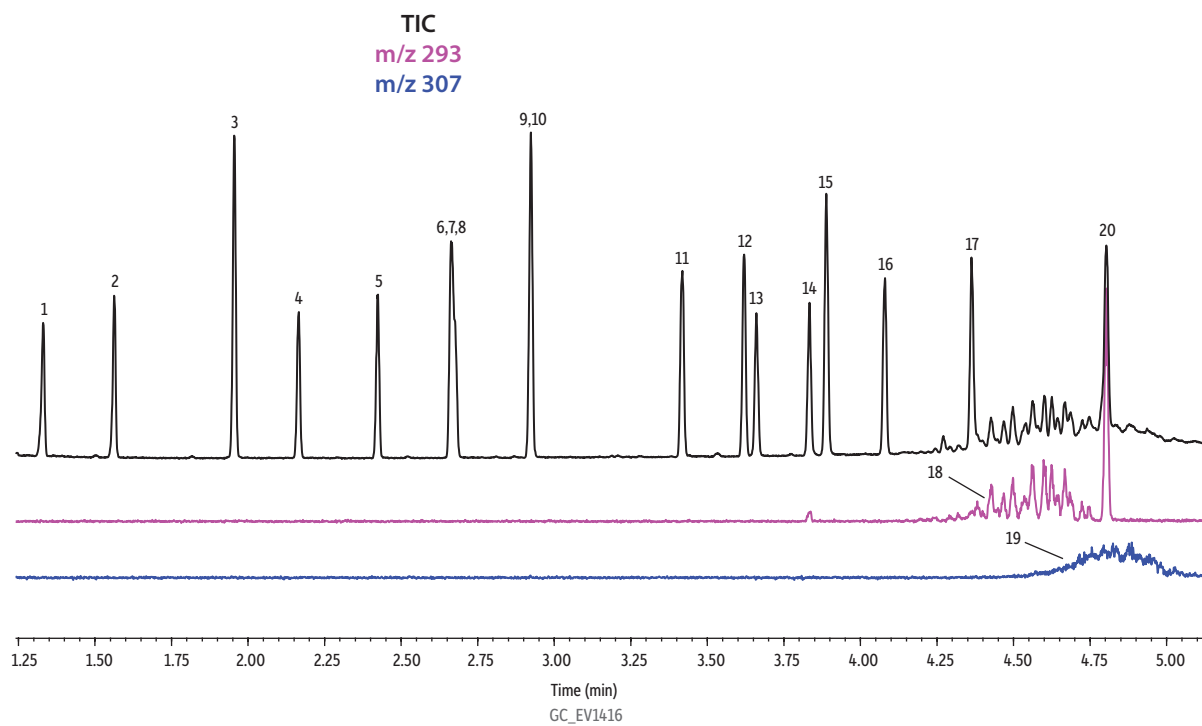
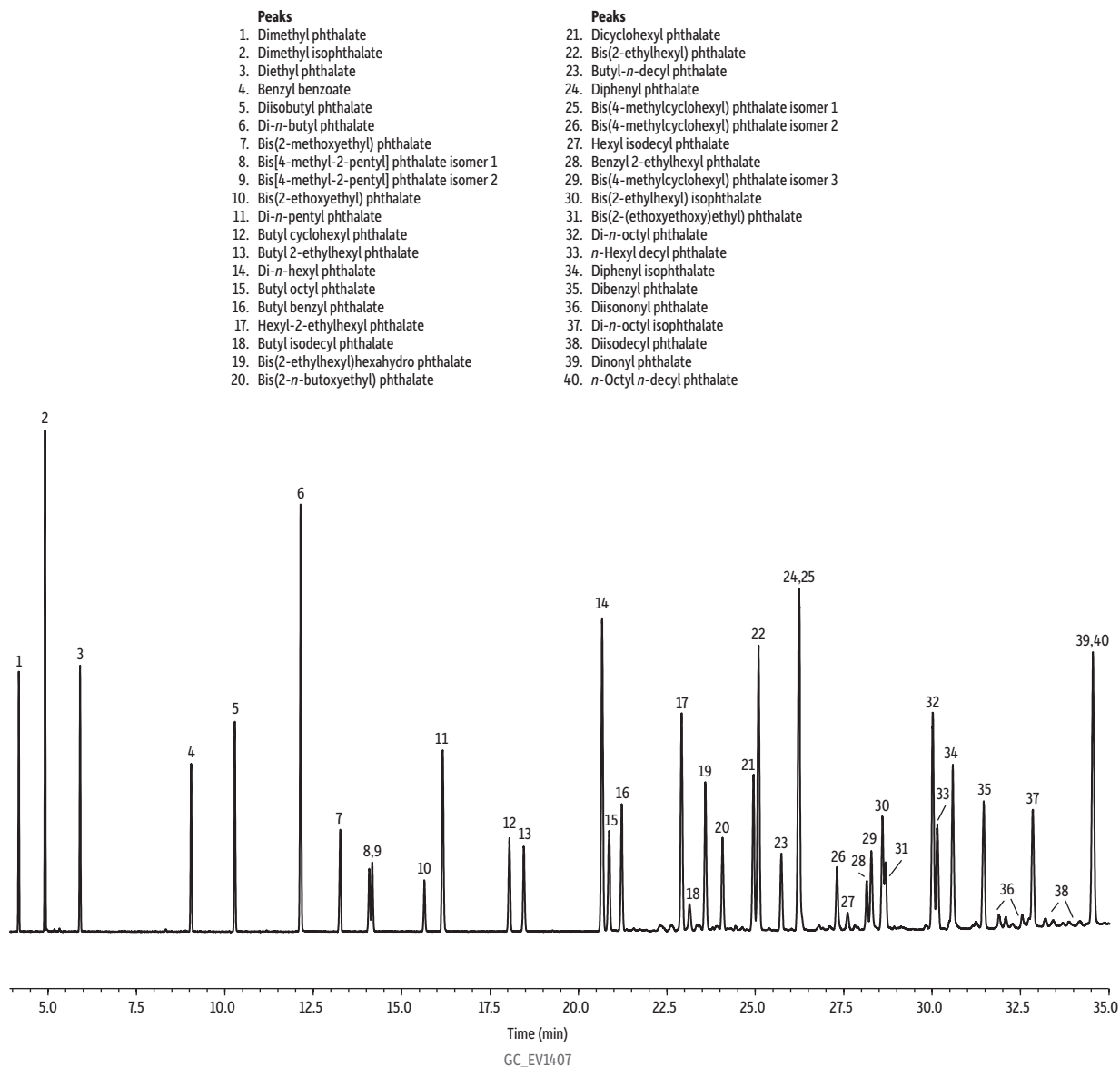


Figure 2: Target compounds on the extended phthalate list (50 µg/mL) are separated under full scan mode on an Rtx-440 column (see Table III for conditions).





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