

Evaluation of the Equality of Non-Polar Capillary Columns in GC/MS Analysis of Food Contact Plastics

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Received June 25, 2013; revised July 25, 2013; accepted August 27, 2013

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ABSTRACT

Non-polar capillary columns for GC/MS are widely utilized in the analysis of additives for food contact materials. Though various kinds of non-polar capillary columns are commercially available, the equality of their performance has not been verified. Herein, ninety-six additives for food contact plastics were analyzed using fifteen kinds of columns, and the peak separation, retention times, and peak areas of each additive were compared. The additives, with various chemical properties, comprised forty four plasticizers, twenty lubricants, twenty antioxidants, nine ultraviolet absorbers, and three other compounds. $10 \mu\text{g}\cdot\text{mL}^{-1}$ test solutions were prepared in acetone, and injected to the GC/MS. The fifteen columns were classified into five categories based on the chromatogram pattern and peak separation. To facilitate comparison of the retention time and detection sensitivity of the columns for the additives, the relative retention time (RRT) and relative peak area (RPA) were calculated by using dibutylphthalate or 4-*tert*-butylphenylsalicylate as an internal standard. The RRTs of the additives on each column were essentially similar. However, the RRT of the additives which were detected in the later stages differed slightly. Although the RPA of the plasticizers and lubricants were roughly similar, column-to-column differences were observed for certain additives, such as antioxidants and ultraviolet absorbers. Furthermore, certain fatty acids, antioxidants, two plasticizers, and two benzophenone type ultraviolet absorbers were not detected in the chromatograms of two columns.

Keywords: Non-Polar Capillary Column; GC/MS Analysis; Additives for Food Contact Plastics; Relative Retention Time; Relative Peak Area

1. Introduction

Gas chromatography/mass spectroscopy (GC/MS) is used world-wide as an analytical method for screening, identification, and quantification of various chemicals. GC/MS analysis is particularly suitable for identification and quantification of chemicals used for food contact materials. Quantification of the residual or migration levels of such chemicals is important for safety assurance and specification testing for national regulatory purposes. Recently, the quantification of plasticizers, antioxidants, alkyl phenols, and benzophenones has been reported using certain simultaneous analytical methods employing a non-polar capillary column [1-5]. In these reports, various non-polar columns such as HP-5MS, DB5-MS, and TRB-5MS were used based on the author's selection. The most commonly used non-polar capillary column is the 5% diphenyl polysiloxane/dimethyl polysiloxane phase type. However, various non-polar columns made

from several materials, such as the phenyl-arylene/ime-thyl polysiloxane phase and siloxane-carborane phase type are commercially available and used in GC/MS analyses. Furthermore, certain columns have also been developed for high temperature analysis. The characteristics of the columns vary widely depending on the technique of the manufacturers. Column catalogues provide comparisons of the performance of competitors. However, to date, these performance comparisons have not been verified.

Herein, ninety six additives used for food contact plastics were selected from the analytes studied in our previous reports [6-10] or from the positive list of EU [11], and a mixture of these chemicals was analyzed via GC/MS. The peak separation, the retention time, and the peak area of each additive were analyzed. Based on comparison of the performance parameters of fifteen columns, the equality of commercially available non-polar columns is verified herein. The properties of these columns are compared based on analysis of additives used for

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food contact plastics.

2. Materials and Methods

2.1. Column

Fifteen columns (15 m × 0.25 mm, thickness 0.1 μm) were used in this study. HP-5MS, DB-5, DB-5HT, DB-5MS, CP-Sil 8CB-MS (CP-Sil), and VF-5ms were purchased from Agilent Technologies (CA, USA). ZB-5 and ZB-5MS were purchased from Phenomenex (CA, USA). Rxi-5HT and Rxi-5Sil MS (Rxi-5) were purchased from Restek (PA, USA). Three lots (SL100102, SL110302, and SL110601) of Inert Cap 5MS/Sil (Inert Cap) were purchased from GL Sciences (Tokyo, Japan). TR-5MS was purchased from Thermo Fisher Scientific (PA, USA). BPX5 and HT5 were purchased from SGE Analytical Science (Victoria, Australia). 007-5MS was purchased from Quadrex (CT, USA). The detailed properties of these columns, such as the phase type, serial number, and accurate length are listed in **Table 1**.

2.2. Standard

Ninety six standards were purchased from Tokyo Chemical Industry Co. Ltd., (Tokyo, Japan), Wako Pure Chemical Industries, Ltd., (Osaka, Japan), Kanto Chemical

Co. Inc. Co. (Tokyo, Japan), and Sigma Aldrich, Japan (Tokyo, Japan), comprising forty-four plasticizers, twenty lubricants, twenty antioxidants, nine ultraviolet absorbers, and three other compounds. These standards were chosen to be representative of typical additives for food contact plastics. Detailed specifications of the additives are listed in **Tables 2** and **3**.

2.3. Preparation of Test Solution

Each stock solution was prepared by dissolving 20.0 mg of standard in 20 mL of acetone or dichloromethane (1 mg·mL⁻¹). Test solution A was prepared by mixing 1 mL of each stock solution of the plasticizers and three others, followed by dilution to 100 mL with acetone (10 μg·mL⁻¹). Test Solution B was prepared by mixing 1 mL of each stock solution of lubricants and antioxidants and ultraviolet absorbers, followed by dilution to 100 mL with acetone (10 μg·mL⁻¹).

2.4. GC/MS Conditions

GC/MS analysis was carried out on an Agilent Technologies 7890 GC coupled to a 5975 C mass selective detector (MSD). Injections (1 μL) were made in splitless mode via a 7693 autosampler. The column temperature was programmed to increase in increments of 20°C min⁻¹ from 50°C to 320°C, at which point the temperature was held for 20 min. The GC injection temperature was 250°C and the transfer line temperature was 280°C. Helium was used as the carrier gas at a flow rate of 1.0 mL min⁻¹. The MSD was operated in scan mode, scanning from *m/z* 40 to 800 using electron impact ionization at 70 eV. Tuning was performed in DFTPP tune mode.

2.5. Measurement and Data Analyses

Test solutions were injected three times each per column. The peak separation and column classification were based on comparison of the total ion chromatogram patterns. Retention times and peak areas of quantitative ions were obtained from each chromatogram. When an additive was detected as multi-peaks, the retention time of the highest peak was used for analysis. The relative retention time (RRT) and the relative peak area (RPA) were calculated by using dibutylphthalate (A17) in test solution A or 4-*tert*-butylphenylsalicylate (B47) in test Solution B as an internal standard.

3. Results and Discussion

3.1. Peak Isolation and Column Classification

Typical total ion chromatograms (TIC) of the test solution, obtained using the columns, are shown in **Figures 1** and **2**. Though generally similar, the chromatogram patterns exhibited subtle differences. Thus, based on

Table 1 Properties of columns used in this study.

Column	Phase type*	Serial	Accurate length** (m)
HP-5MS	(a)	US9609347H	15.1
DB-5	(a)	USB118311A	16.5
ZB-5	(a)	191475	15.2
DB-5HT	(a)	USB330333A	15.9
ZB-5HT	(a)	220081	15.8
Rxi-5HT	(a)	1041920	15.7
DB-5MS	(b)	US8649613A	16.5
Rxi-5	(b)	1031442	15.0
Inert Cap	(b)	SL100102	15.7
		SL110302	15.7
		SL110601	15.7
CP-Sil	(b)	2217061	15.0
VF-5ms	(b)	2203501	15.2
TR-5MS	(b)	12806A33	15.7
BPX5	(b)	12843A69	15.5
007-5MS	(b)	110506F	16.1
HT5	(c)	12666B15	15.1

*: (a) 5% diphenyl polysiloxane/dimethyl polysiloxane, (b) phenyl-arylene/dimethyl polysiloxane, (c) siloxane-carborane. **: Accurate length = (diameter × 3.14 × number of rolls) + margin length.

Table 2. Properties of additives for test Solution A.

Application	Type	No.	Additives	CAS No.	Molecular weight	Quantitative ion (<i>m/z</i>)
Plasticizer	Adipate	A1	Diisopropyladipate	6938-94-9	230	129
		A2	Dipropyladipate	106-19-4	230	171
		A3	Diisobutyladipate	141-04-8	258	129
		A4	Dibutyladipate	105-99-7	258	185
		A5	Dialkyladipate*	—	—	129
		A6	Di(2-ethylhexyl)adipate	103-23-1	370	129
		A7	Heptylnonyladipate*	68515-75-3	370	129
		A8	Diocetyladipate	123-79-5	370	241
		A9	Dibenzyladipate	2451-84-5	326	91
		A10	Diisononyladipate*	33703-08-1	398	129
		A11	Diisodecyladipate*	27178-16-1	426	129
	Phthalate	A12	Dimethylphthalate	131-11-3	194	163
		A13	Diethylphthalate	84-66-2	222	149
		A14	Diisopropylphthalate	605-45-8	250	149
		A15	Dipropylphthalate	131-16-8	250	149
		A16	Diisobutyl phthalate	84-69-5	278	149
		A17	Dibutylphthalate	84-74-2	278	149
		A18	Benzylbutylphthalate	85-68-7	312	149
		A19	Diisoheptylphthalate*	41451-28-9	362	149
		A20	Di(2-ethylhexyl) 1,2,3,6-tetrahydrophthalate	2915-49-3	394	124
		A21	Dicyclohexylphthalate	84-61-7	330	149
		A22	Di(2-ethylhexyl)phthalate	117-81-7	390	149
		A23	Diisooctylphthalate*	27554-26-3	390	149
		A24	Dinonylphthalate*	84-76-4	418	149
		A25	Di(2-ethylhexyl)isophthalate	137-89-3	390	112
		A26	Di- <i>n</i> -octylphthalate	117-84-0	390	149
		A27	Di(2-ethylhexyl)terephthalate	6422-86-2	390	112
		A28	Diisononyl 1,2-cyclohexanedicarboxylate	166412-73-8	424	155
		A29	Diisononylphthalate*	28553-12-0	418	149
		A30	Diisodecylphthalate*	26761-40-0	446	149
		A31	Tri(2-ethylhexyl)trimellitate	3319-31-1	546	305
Citrate	A32	Tributylcitrate	77-94-1	360	185	
	A33	Acetyltributylcitrate	77-90-7	402	185	
Sebacate	A34	Dibutylsebacate	109-43-3	314	241	
	A35	Di(2-ethylhexyl)sebacate	122-62-3	426	185	
Glycol	A36	2,2,4-Trimethyl-1,3-pentanediol diisobutyrate	6846-50-0	286	71	
	A37	Glyceroldiacetate laurate	30899-62-8	316	159	
	A38	Neopentylglycoldibenzoate	4196-89-8	312	105	
	A39	Triethyleneglycoldi(2-ethylhexanoate)	94-28-0	402	171	
Other	A40	Dibutylmaleate	105-76-0	228	99	
	A41	Di(2-ethylhexyl)azelate	103-24-2	412	171	
	A42	<i>p</i> - <i>tert</i> -Octylphenol	140-66-9	206	135	
	A43	2-Ethylhexyldiphenylphosphate	1241-94-7	362	251	
	A44	Cresyldiphenylphosphate*	26444-49-5	340	340	
Other	A45	<i>n</i> -Tetradecanol	112-72-1	214	55	
	A46	Nonylphenol	25154-52-3	220	107	
	A47	Bisphenol A	80-05-7	228	213	

*: Mixture of two or more compounds.

Table 3. Properties of additives for test Solution B.

Application	Type	No.	Additives	CAS No.	Molecular weight	Quantitative ion (<i>m/z</i>)
Lubricant	Amide	B1	Hexadecanamide	629-54-9	255	59
		B2	9-Octadecenamide	301-02-0	282	59
		B3	Octadecanamide	124-26-5	284	59
		B4	<i>cis</i> -13-Docosenamide	112-84-5	337	59
		B5	Docosanamide	3061-75-4	339	59
	Fatty ester	B6	Ethyl- <i>n</i> -hexadecanoate	628-97-7	284	88
		B7	Ethyl (<i>Z,Z</i>)-9,12-Octadecadienoate	544-35-4	308	67
		B8	Ethyl- <i>cis</i> -9-octadecenoate	111-62-6	310	55
		B9	Ethyl octadecanoate	111-61-5	312	88
	Fatty acid	B10	Dodecanoic acid	143-07-7	200	60
		B11	Tetradecanoic acid	544-63-8	228	60
		B12	Hexadecanoic acid	57-10-3	256	60
		B13	Octadecanoic acid	57-11-4	284	60
	Hydrocarbon	B14	<i>n</i> -Tetradecane	629-59-4	198	71
		B15	<i>n</i> -Hexadecane	629-73-2	226	71
		B16	<i>n</i> -Octadecane	593-45-3	254	71
		B17	<i>n</i> -Eicosane	112-95-8	282	71
		B18	<i>n</i> -Docosane	629-97-0	310	71
		B19	<i>n</i> -Tetracosane	646-31-1	338	71
		B20	<i>n</i> -Hexacosane	630-01-3	366	71
Antioxidant	Phenol	B21	3- <i>tert</i> -Butyl-4-hydroxyanisole	121-00-6	180	165
		B22	2,6-Di- <i>tert</i> -butyl-4-methylphenol	128-37-0	220	205
		B23	2,6-Di- <i>tert</i> -butyl-4-ethylphenol	4130-42-1	234	219
		B24	2,6-Di- <i>tert</i> -butyl-4-hydroxymethylphenol	88-26-6	236	221
		B25	2,2'-Methylenebis(6- <i>tert</i> -butyl-4-methylphenol)	119-47-1	341	177
		B26	2,2'-Ethylidenebis(4,6-di- <i>tert</i> -butylphenol)	35958-30-6	439	233
		B27	2,2'-Methylenebis(4-ethyl-6- <i>tert</i> -butylphenol)	88-24-4	369	191
		B28	4,4'-Methylenebis(2,6-di- <i>tert</i> -butylphenol)	118-82-1	425	409
		B29	2,2'-Methylenebis[6-(1-methylcyclohexyl)- <i>p</i> -cresol]	77-62-3	420	420
		B30	Octadecyl-3,5-di- <i>tert</i> -butyl-4-hydroxyhydro cinnamate	2082-79-3	531	530
		B31	1,1,3-Tris(2-methyl-4-hydroxy-5- <i>tert</i> -butylphenyl)butane	1843-03-4	544	339
		B32	1,3,5-Tris(3,5-di- <i>tert</i> -butyl-4-hydroxybenzyl)- <i>s</i> -triazine-2,4,6-(1 <i>H</i> ,3 <i>H</i> ,5 <i>H</i>)-trione	27676-62-6	784	219
		B33	1,3,5-Trimethyl-2,4,6-tris(3,5-di- <i>tert</i> -butyl-4-hydroxybenzyl)benzene	1709-70-2	775	775
	B34	2,2'-Oxamidobis [ethyl-3-(3,5-di- <i>tert</i> -butyl-4-hydroxyphenyl)propionate]	70331-94-1	697	219	
	B35	1,3,5-Tris(4- <i>tert</i> -butyl-3-hydroxy-2,6-dimethylbenzyl)-1,3,5-triazine-2,4,6-(1 <i>H</i> ,3 <i>H</i> ,5 <i>H</i>)-trione	40601-76-1	900	190	
	Sulfurate	B36	4,4'-Thiobis(3-methyl-6- <i>tert</i> -butylphenol)	96-69-5	359	358
		B37	Didodecyl 3,3'-thiodipropionate	123-28-4	515	55
		B38	Di- <i>n</i> -tetradecyl 3,3'-thiodipropionate	16545-54-3	571	57
		B39	Di-octadecyl 3,3'-thiodipropionate	693-36-7	683	55
	Phosphite	B40	Tris(2,4-di- <i>tert</i> -butylphenyl)phosphite	31570-04-4	647	441
Ultraviolet absorber	Benzophenone	B41	2-Hydroxy-4-methoxybenzophenone	131-57-7	228	227
		B42	2,2'-Dihydroxy-4-methoxybenzophenone	131-53-3	244	244
		B43	2-Hydroxy-4- <i>n</i> -octabenzophenone	1843-05-6	326	213
		B44	2-(2'-Hydroxy-5'-methylphenyl)benzotriazole	2440-22-4	225	225
	Benzotriazole	B45	2-(2'-Hydroxy-3'- <i>tert</i> -butyl-5'-methylphenyl)-5-chlorobenzotriazole	3896-11-5	316	300
	B46	2-(2'-Hydroxy-3',5'-di- <i>tert</i> -amylphenyl) benzotriazole	25973-55-1	351	322	
Other	B47	4- <i>tert</i> -Butylphenylsalicylate	87-18-3	270	121	
	B48	2,4-Di- <i>tert</i> -butylphenyl-3,5-di- <i>tert</i> -butyl-4-hydroxybenzoate	4221-80-1	439	233	
	B49	2,5-Di(5- <i>tert</i> -butylbenzoxazol-2-yl)thiophene	7128-64-5	430	415	

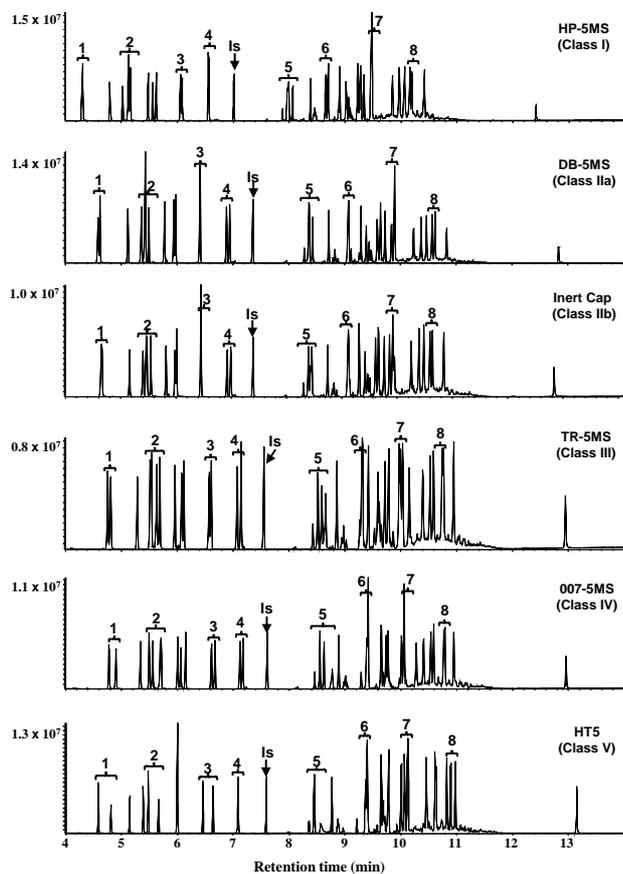


Figure 1. Typical GC/MS total ion chromatograms for test Solution A.

the chromatogram pattern and separation of fifteen peak groups (peak Groups 1-8 in test Solution A, and peak Groups 9-15 in test Solution B) in the analyses of the additives, the fifteen columns were classified into various categories (Classes I-V, **Table 4**).

Class I contains six columns which are 5% diphenyl polysiloxane/dimethyl polysiloxane phase type columns. Complete separation of the peaks in Groups 3, 6, 8, 10, and 11 was achieved in the total ion chromatograms of Class I, whereas the peaks in Groups 1, 7, and 15 were not separated. However, there were certain differences in the separation of peak Groups 2, 5, 12, and 14. In particular, among the Class I columns, the chromatogram obtained using the Rxi-5HT column was slightly different from those using other columns in terms of the separation pattern of peak Groups 4 and 9.

The chromatogram patterns obtained using the phenyl-arylene/dimethyl polysiloxane phase type columns were different from those of the other columns, thus, the columns were divided into Classes II to IV. Class II contains five columns. The chromatograms obtained with these columns showed clear separation of the peaks in Groups 4, 8, 9, 10, 11, 12, and 14, whereas the peaks in Groups 3 and 6 were not separated. The five

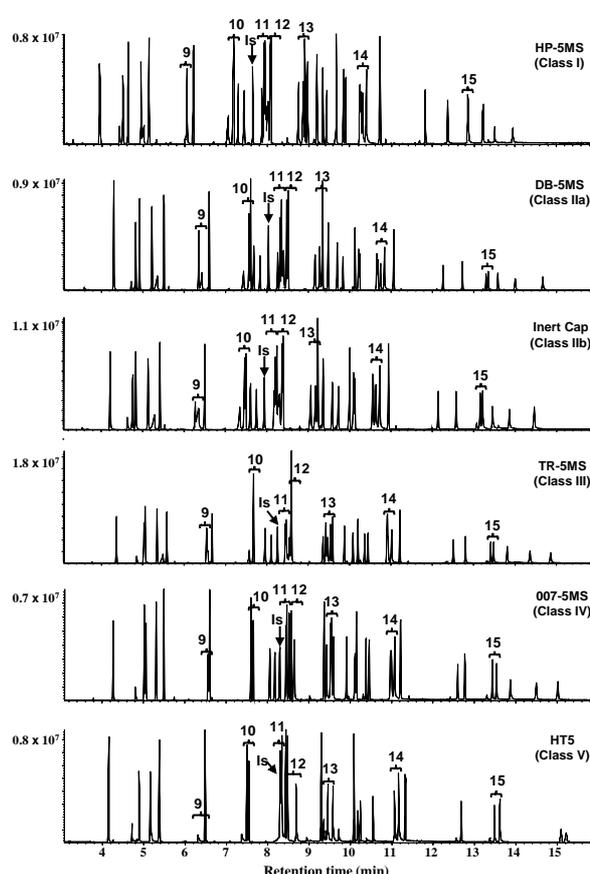


Figure 2. Typical GC/MS total ion chromatograms for test Solution B.

columns in Class II were further classified into Class IIa and Class IIb based on the separation pattern of peak Groups 1, 5, 7, and 14. Class III contained two columns. The TIC showed clear separation of the peaks in Groups 1-5, 7, 8, 11, 13, and 15. However, only the chromatogram pattern obtained using the 007-5MS column which contains a phenyl-arylene/dimethyl polysiloxane phase, was different from that of Classes II and III. Most of the peaks in the chromatogram of from this column appeared to be sharper than those obtained with the other columns. Furthermore, five additives such as fatty acids (B10-13) and a phenol type antioxidant (B32) could not be detected using the 007-5MS column. Therefore, the 007-5MS column was classified into Class IV.

The HT5 column is a siloxane-carborane phase type column. This column was classified into Class V. The chromatogram pattern obtained with this column was different from those obtained using the other columns. Thirteen kinds of additives, comprising two plasticizers (A11 and A42), six antioxidants (B24, B31, B32, and B34-36), two benzophenone type ultraviolet absorbers (B41 and B43), and three others (A45-A47) could not be detected in the chromatogram.

Table 4. Column classification and peak separations in respective chromatograms.

Class	Column	Phase type	Peak group in test Solution A								Peak group in test Solution B						
			1	2	3	4	5	6	7	8	9	10	11	12	13	14	15
			A1	A2	A4	A16	A32	A19	A8	A11	B11	B6	B7	B1	B3	B4	B30
			A12	A13	A15	A46	A33	A43	A9	A20	B24	B17	B8	B9	B19	B5	B37
				A36			A34		A21	A26			B13	B18	B25	B36	
				A42			A47		A22	A28			B42			B43	
Class I	HP-5MS	(a)	n	y/n	y	n	y/n	y	n	y	n	y	y	y/n	y/n	y	n
	DB-5	(a)	n	y	y	n	y	y	n	y	n	y	y	y	y/n	y	n
	ZB-5	(a)	n	y/n	y	n	y/n	y	n	y	n	y	y	y	y/n	y	n
	DB-5HT	(a)	n	y	y	n	y/n	y	n	y	n	y	y	y	y/n	y	n
	ZB-5HT	(a)	n	y/n	y	n	y	y	n	y	n	y	y	y	y/n	y/n	n
	Rxi-5HT	(a)	n	y	y	y	y	y	n	y	y	y	y	y	y/n	y/n	n
Class IIa	DB-5MS	(b)	y	y/n	n	y	n	n	n	y	y	y	y	y	n	y	y
	Rxi-5	(b)	y	y/n	n	y	n	n	n	y	y	y	y	y	n	y	y
Class IIb	Inert Cap	(b)	n	y/n	n	y	y	n	y	y	y	y	y	y	n	y/n	y
	CP-Sil	(b)	n	y	n	y	y	n	y	y	y	y	y	y	y	y/n	y
	VF-5ms	(b)	y/n	y	n	y	y	n	y	y	y	y	y	y	y	y/n	y
Class III	TR-5MS	(b)	y	y	y	y	y	n	y	y	n	n	y	n	y	n	y
	BPX5	(b)	y	y	y	y	y	n	y	y	y/n	n	y	n	y	n	y
Class IV	007-5MS	(b)	y	y/n	y	y	y	y/n	n	n	*	y	y	*	y	n	y
Class V	HT5	(c)	y	*	y	*	*	y	y/n	y	*	y	n	n	n	*	y

(a): 5% diphenyl polysiloxane/dimethyl polysiloxane, (b): phenyl-arylene/dimethyl polysiloxane, (c): siloxane-carborane. y: Peaks were separated completely. y/n: Peaks were incompletely separated. n: Peaks overlapped completely. *: A portion of peak was not detected.

3.2. Comparison of Retention Time

The accurate lengths of all of the columns were determined to be between 15 m and 16.5 m (Table 1). The inner diameters and film thickness were not measured; these may vary depending on the column products as well as length. These differences should affect the retention times of the additives. Therefore, the relative retention times (RRTs) of the additives during the analyses using the columns were compared. Dibutylphthalate (A17) and 4-*tert*-butylphenylsalicylate (B47) are chosen for use as internal standards in test Solutions A and B, respectively, given that these substances were detected in the approximate middle of the additives detected in all of the chromatograms, and did not overlap with the peaks of other additives in the chromatograms. Analysis of the additives using the Inert Cap column were performed using three lots of the column having different serial numbers. The obtained analytical data were used as an index of reproducibility for instrumental conditions, internal standard correction, and lot-to-lot variation.

The RRT of the typical columns and coefficient of variation (CV) using three lots of Inert Cap, Class I, Class II, and all fifteen columns are shown in Tables 5 and 6.

The RRT of the additives were not significantly different. However, in the case of certain additives with similar retention times, the order of detection changed from column to column. The CV of the RRTs of the additives using the three lots of Inert Cap products were 0.1% - 0.9%, and were mostly below 0.5%. The results suggest that there were no differences in the instrumental conditions of this study. The additives detected in the early stage (RRT < 0.75) and middle stages (RRT = 0.75 - 2, approximate retention time of 5.6 - 15 minutes) had largely similar RRTs, irrespective of the columns, and their CVs were low (0.2% - 1.9%). However, the RRTs of the additives detected at the later stage (RRT > 2) varied widely. Thus, CVs appear to be high (3.2% - 6.3%). This later stage corresponds largely to the maximum temperature (320°C) holding period in the column tem-

Table 5. Relative retention time of additives in test Solution A.

No.	Class I	Class IIa	Class IIb	Class III	Class IV	Class V	CV (%)			
	HP-5MS	DB-5MS	Inert Cap	TR-5MS	007-5MS	HT5	Inert Cap 3 lots	Class I	Class II	All column
A17	7.01	7.37	7.22	7.56	7.61	7.60	-	-	-	-
A12	0.61	0.62	0.62	0.64*	0.64*	0.63*	0.2	0.9	0.6	1.5
A1	0.61	0.63	0.62	0.63*	0.63*	0.60*	0.2	0.9	0.7	1.2
A40	0.68	0.69	0.69	0.70	0.70	0.68	0.2	0.6	0.5	0.9
A2	0.72	0.73	0.72	0.73	0.73*	0.71	0.2	0.6	0.4	0.8
A36	0.73	0.74	0.73	0.73	0.72*	0.72	0.2	0.6	0.6	1.0
A13	0.73	0.74	0.73	0.75	0.75*	0.75	0.2	0.6	0.4	0.8
A42	0.74	0.74	0.74	0.75	0.75*	ND	0.2	0.7	0.4	0.7
A14	0.78	0.78	0.78	0.79	0.79	0.79	0.2	0.4	0.3	0.5
A45	0.79	0.81	0.80	0.80	0.80	ND	0.2	0.5	0.3	0.4
A3	0.80	0.81	0.81	0.81	0.81	0.79	0.2	0.3	0.2	0.6
A15	0.87	0.87	0.87	0.87	0.88*	0.87*	0.2	0.2	0.1	0.3
A4	0.86	0.87	0.87	0.87	0.87*	0.85*	0.2	0.2	0.2	0.6
A16	0.93	0.93	0.93	0.94	0.93	0.93	0.2	0.1	<0.1	0.2
A46	0.94	0.94	0.94	0.95	0.94	ND	0.2	0.3	<0.1	0.3
A5 (m)	1.13	1.13	1.13	1.12	1.11	1.10	0.2	0.3	0.2	0.7
A34	1.14	1.14	1.14	1.13	1.12	1.11	0.2	0.3	<0.1	0.7
A47	1.14	1.14	1.14	1.15*	1.15*	ND	0.2	0.3	0.2	0.5
A32	1.15	1.15	1.15	1.14*	1.13*	1.13	0.2	0.3	0.1	0.6
A33	1.20	1.19	1.19	1.17	1.17	1.15	0.2	0.4	0.2	1.4
A37	1.21	1.21	1.20	1.19	1.18	1.17	0.2	0.4	0.2	0.9
A38	1.24	1.24	1.24	1.23	1.23	1.23	0.2	0.3	0.1	0.4
A18	1.24	1.24	1.24	1.23	1.24	1.24	0.3	0.3	0.2	0.3
A6	1.27	1.27	1.27	1.25	1.24	1.24	0.2	0.5	0.2	0.9
A43	1.29	1.28	1.28	1.27	1.27	1.27	0.2	0.4	0.2	0.5
A19 (m)	1.29	1.29	1.29	1.27	1.27	1.28*	0.2	0.4	0.2	0.6
A39	1.32	1.31	1.30	1.29	1.28	1.27*	0.3	0.4	0.3	1.0
A44 (m)	1.32	1.31	1.31	1.31*	1.31*	1.31*	0.3	0.4	0.2	0.4
A7 (m)	1.32	1.32	1.32*	1.30*	1.29*	1.28*	0.2	0.8	0.2	1.1
A20	1.32	1.32	1.31*	1.29*	1.28*	1.29*	0.2	0.5	0.3	1.0
A21	1.33	1.33	1.33	1.32	1.32	1.33*	0.2	0.4	0.2	0.4
A9	1.35	1.35	1.35*	1.34*	1.35*	1.33*	0.2	0.5	0.2	0.6
A8	1.35	1.35	1.35*	1.33*	1.32*	1.32*	0.2	0.5	0.3	0.9
A22	1.35	1.35	1.34*	1.32*	1.32*	1.33	0.3	0.7	0.2	0.9
A23 (m)	1.35	1.40	1.36	1.36	1.34*	1.33	0.2	1.0	1.1	1.6
A24 (m)	1.41	1.40	1.39	1.37	1.37	1.38	0.2	0.5	0.3	0.8
A10 (m)	1.42	1.40	1.40	1.39	1.38	1.38	0.2	0.8	0.2	0.9
A25	1.42	1.42	1.42	1.39	1.39	1.40	0.2	0.6	0.3	0.9
A41	1.44	1.44	1.43	1.40	1.39	1.40	0.2	0.6	0.3	1.1
A26	1.45	1.45	1.44	1.42	1.42	1.42	0.2	0.6	0.4	0.8
A27	1.46	1.46	1.45	1.42	1.42	1.43	0.2	0.6	0.4	0.9
A28	1.47	1.47	1.45	1.44	1.43	1.44	0.4	0.8	0.6	1.0
A11 (m)	1.48	1.47	1.46	1.45	1.43	ND	0.5	1.0	0.5	1.1
A35	1.49	1.49	1.48	1.45	1.44	1.44	0.2	0.6	0.4	1.1
A29 (m)	1.48	1.51	1.49	1.47	1.45	1.48	0.3	0.5	0.6	1.0
A30 (m)	1.54	1.55	1.54	1.51	1.50	1.50	0.7	1.0	0.8	1.3
A31	1.77	1.79	1.75	1.71	1.70	1.73	0.2	0.8	0.7	1.3

A17 was used as an internal standard, the values show retention times (min). Each value is the average of three analytical runs. ND: not detected. (m): Additive was detected as multi-peaks on chromatogram; the values were calculated from the maximum peak. *: The order of detection differs from the Class I columns.

Table 6. Relative retention time of additives in test Solution B.

No.	Class I	Class IIa	Class IIb	Class III	Class IV	Class V	CV (%)			
	HP-5MS	DB-5MS	Inert Cap	TR-5MS	007-5MS	HT5	Inert Cap 3 lots	Class I	Class II	All column
B47	7.38	7.68	7.62	7.96	8.06	8.34	-	-	-	-
B14	0.52	0.53	0.53	0.53	0.52	0.50	0.3	1.1	1.4	1.9
B21	0.59	0.60	0.60	0.61	0.61	0.57	0.2	0.7	0.8	1.8
B22	0.61	0.61	0.61	0.61	0.61	0.59	0.2	0.8	0.9	1.5
B23	0.65	0.65	0.65	0.65	0.64	0.62	<0.1	0.6	0.7	1.6
B10	0.66	0.67	0.66	0.66	ND	0.62	0.1	0.5	0.5	1.7
B15	0.67	0.69	0.68	0.68	0.66	0.65	0.1	0.8	0.7	1.6
B11	0.79	0.80*	0.80*	0.80*	ND	0.76	<0.1	0.4	0.3	1.4
B24	0.79	0.79*	0.79*	0.79*	0.79	ND	<0.1	0.2	0.3	0.5
B16	0.81	0.82	0.82	0.81	0.80	0.78	<0.1	0.2	0.4	1.4
B12	0.92	0.93	0.92	0.92	ND	0.88	0.2	0.2	0.1	1.1
B6	0.94	0.94	0.94	0.93	0.92	0.91*	<0.1	<0.1	0.2	1.0
B17	0.94	0.95	0.94	0.93	0.92	0.90*	<0.1	<0.1	0.3	1.3
B41	0.95	0.96	0.96	0.96	0.97	ND	<0.1	0.1	0.2	0.6
B44	0.97	0.98	0.98	0.98	0.99	1.00	0.1	<0.1	0.1	0.7
B42	1.03	1.03	1.03	1.04*	1.04*	1.08*	0.1	<0.1	0.2	1.4
B7	1.03	1.03	1.03	1.02*	1.02*	1.00*	0.1	0.2	0.1	1.0
B8	1.04	1.04	1.04	1.03*	1.02*	1.00*	<0.1	0.1	0.2	1.0
B13	1.04	1.04	1.04	1.03*	ND	1.00*	0.1	0.1	0.2	1.0
B1	1.05	1.05	1.05	1.04	1.04*	1.04*	0.2	0.2	0.1	0.3
B9	1.05	1.05	1.05	1.04	1.03*	1.02*	0.1	0.2	0.2	1.0
B18	1.06	1.06	1.06	1.04	1.03*	1.01*	0.1	0.2	0.3	1.3
B2	1.14	1.14	1.14	1.13	1.14*	1.13	0.2	0.3	0.2	0.3
B3	1.16	1.15	1.16	1.15*	1.15*	1.15*	0.1	0.4	0.3	0.4
B19	1.16	1.16	1.16	1.14*	1.13*	1.13*	<0.1	0.4	0.3	1.1
B25	1.17	1.16	1.16	1.15	1.16*	1.12*	<0.1	0.5	0.2	1.1
B26	1.20	1.18	1.18	1.16	1.15*	1.12*	0.2	0.3	0.2	1.8
B27	1.22	1.21	1.21	1.20	1.20	1.17	0.2	0.4	0.2	1.1
B45	1.23	1.22	1.23	1.22	1.22	1.23*	0.2	0.3	0.3	0.4
B20	1.26	1.26	1.26	1.24	1.22	1.21*	0.2	0.4	0.3	1.3
B46	1.28	1.27	1.28*	1.27*	1.26*	1.27*	0.2	0.4	0.3	0.5
B28	1.29	1.27	1.27*	1.26*	1.25*	1.22*	0.2	0.4	0.3	1.3
B36	1.34	1.33	1.33	1.32	1.32	ND	0.2	0.4	0.4	0.5
B43	1.34	1.33	1.33	1.32	1.32	ND	0.2	0.4	0.3	0.5
B4	1.34	1.34	1.34	1.32	1.33	1.33	0.2	0.4	0.3	0.6
B5	1.36	1.35	1.35	1.33	1.34	1.34	0.2	0.5	0.4	0.6
B48	1.40	1.38	1.38	1.36	1.35	1.36	0.2	0.5	0.4	1.0
B29	1.54	1.52	1.53	1.52	1.52	1.51	0.6	0.5	0.5	0.7
B40	1.61	1.58	1.58	1.55	1.54	1.52	0.2	0.6	0.5	1.6
B30	1.68	1.65	1.66	1.62	1.62	1.62	0.3	0.7	0.5	1.1
B37	1.68	1.66	1.66	1.63	1.63	1.63	0.3	0.7	0.5	1.0
B31	1.72	1.69	1.69	1.67	1.67	ND	0.3	0.7	0.5	0.9
B49	1.76	1.74	1.75	1.74	1.75	1.81	0.3	0.5	0.4	1.0
B38	1.82	1.82	1.82	1.80	1.81	1.83	0.3	0.6	0.4	0.6
B32	2.41	2.45	2.41	2.50	ND	ND	0.5	3.0	2.5	3.2
B34	2.43	2.55	2.52	2.64*	2.81*	ND	0.5	3.2	2.9	5.0
B39	2.47	2.69*	2.65*	2.72*	2.85*	3.00*	0.3	3.4	3.9	6.3
B33	2.50	2.55*	2.52*	2.60*	2.66*	2.75*	0.9	3.3	2.8	4.1
B35	2.73	2.86	2.83	3.06	3.25	ND	0.6	3.8	3.4	6.3

B47 was used as an internal standard, the values show retention times (min). Each value is the average of three analytical runs. ND: not detected. *: The order of detection differs from the Class I columns.

perature control program. These results suggest that the RRTs of the additives detected during the heat-up period in the column temperature control program were similar for all columns, however, the RRTs of the additives detected during the holding period varied depending on the column.

The CVs of the RRTs of the additives assayed using the Classes I and II columns were significantly lower than those of all fifteen columns. In particular, the CVs of the additives detected in the middle stage (RRT = 0.75 – 2) were similar to those obtained using the three lots of Inert Cap. Slight variation of the RRTs and the order of detection of certain additives were observed using the Classes III-V columns. Specifically, some additives were not detected on the 007-5MS and HT5 columns.

Given that the RRT of most of the additives was similar in the analyses using the fifteen columns, the properties of these columns are deemed equivalent in terms of the retention time of the additives. Therefore, for identification assay via GC analysis, these columns are deemed to be compatible with each other. Specifically, the eleven kinds of columns, classified into Classes I and II, exhibit largely equivalent performance. On the other hand, for analysis of the additives using the four kinds of columns classified in Classes III-V, the column identity might be important in terms of the detection order, and the physicochemical properties or structure of the analytes.

3.3. Comparison of Sensitivity

The detection sensitivity of the columns for the additives was analyzed based on the relative peak areas (RPAs) of the additives to the peak area of the internal standard. However, the data for twenty-two additives could not be compared because their RPA was small (less than 0.1) and because some additives were detected as multi-peaks. The RPAs and corresponding CVs for each additive are shown in **Tables 7 and 8**. To determine the effects of the inevitable basic conditions of the instrument, such as contamination in the injection port or ion source, column fixing or MSD tuning, etc., the analyses were performed using three lots of Inert Cap columns as well as performing the retention time comparisons.

In the case of the three lots of Inert Cap columns, the CVs of the RPA were less than 10%. However, for the analyses of the thirteen kinds of additives, such as A25, A27, A31, B4, B10, B11, B17, B19, B22, B24, B25, B28, and B40, the CVs were larger than 10%. The lubricants and antioxidants were strongly influenced by the inevitable basic conditions of the instrument.

The CVs for the analyses of the additives using all columns were mostly below 20%. However, the CVs for fourteen additives, such as A25, A31, A45, B4, B22, B23, B27, B28, B36, B37, B40, B42, B43, and B46, were larger than 20%, and these additives were mainly antioxi-

dants or ultraviolet absorbers. The sensitivities towards these additives varied based on the type of column. The RPAs of a number of additives obtained using the 007-5MS and HT5 columns were significantly different from those obtained with other columns.

For the analysis using the HT5 column, the RPAs of nine plasticizers (A2, A4, A8, A12, A13, A32, A33, A34, and A43) were significantly lower than that achieved with the other columns, although the RPA of A25 was relatively high. The RPA of the amide type lubricants (B1-B5) were relatively low when Class Iib columns were used, but were notably high for the analyses using the 007-5MS and HT5 columns. Furthermore, the RPAs of the hydrocarbons (B14-20) were slightly high when the 007-5MS column was used for analysis. Most of the antioxidants and ultraviolet absorbers were detected at low levels using HT5. Although the RPAs of the antioxidant B37 were high when the DB-5HT and ZB-5HT columns were used, these values were low when the Rxi-5, Inert Cap, CP-Sil, VF-5ms, and TR-5MS columns were used. The RPAs of six antioxidants (B25-28, B31, and B40) and an ultraviolet absorber B41 were high using the DB-5HT and ZB-5HT columns.

The maximum/minimum ratio (Max/Min) of the RPAs was less than 2 for the plasticizers and lubricants, except for the fatty acids (B10-12). Therefore, the limit of determination or detection for these additives using an internal standard method would be similar, even if the analyses were performed using columns other than those specified in the published analytical methods. However, the Max/Min of the antioxidants and ultraviolet absorbers were 1.8-4.7, thus, the sensitivity may vary depending on the columns.

4. Conclusions

In this study, ninety-six kinds of additives used for food contact plastics, which have various chemical properties, were analyzed by GC/MS using fifteen kinds of non-polar capillary columns. These columns were categorized into five classes based on their chromatogram patterns and the degree of peak separation. This study suggests that the performance of most non-polar capillary columns is equivalent. Therefore, it is implied that all of the columns used in this study may be applicable in the identification of the additives used in food contact plastics. However, the RRTs of additives detected during the holding period of the column temperature control program varied depending on the type of column.

The detection sensitivity for certain antioxidants or ultraviolet absorbers may vary depending on the type of column used in the analysis. Thus, it is suggested that the sensitivities or limits of detection should be confirmed in the analysis of these additives. Moreover, certain additives such as fatty acids and some antioxidants, two plas-

Table 7. Relative peak areas of additives in test Solution A.

Application	No.	Class I					Class II					Class III		Class IV	Class V	CV (%)		Max/Min		
		HP-5 MS	DB-5	ZB-5	DB-5 HT	ZB-5 HT	Rxi-5 HT	DB-5 MS	Rxi-5	Inert Cap	CP-Sil	VF-5 ms	TR-5 MS	BPX-5	007-5 MS	HT5	Inert Cap 3 lots		All column	
Is	A17	6.1 × 10 ⁶		3.9 × 10 ⁶		5.0 × 10 ⁶		4.7 × 10 ⁶		3.2 × 10 ⁶		7.9 × 10 ⁶		5.8 × 10 ⁶		4.2 × 10 ⁶		-	-	-
			5.2 × 10 ⁶		5.5 × 10 ⁶		4.6 × 10 ⁶		3.4 × 10 ⁶		4.7 × 10 ⁶		5.4 × 10 ⁶		3.6 × 10 ⁶					
Plasticizer	A1	0.25	0.35	0.31	0.25	0.25	0.27	0.29	0.34	0.31	0.31	0.30	0.29	0.31	0.29	0.24	4	12	1.5	
	A2	0.17	0.22	0.22	0.19	0.18	0.19	0.20	0.22	0.21	0.20	0.20	0.21	0.20	0.21	0.13	4	11	1.7	
	A3	0.27	0.36	0.30	0.28	0.24	0.28	0.33	0.38	0.35	0.35	0.28	0.28	0.34	0.30	0.27	2	13	1.6	
	A4	0.16	0.19	0.20	0.18	0.17	0.17	0.18	0.19	0.18	0.18	0.18	0.18	0.18	0.18	0.12	2	9	1.7	
	A6	0.32	0.37	0.31	0.34	0.35	0.33	0.40	0.36	0.39	0.37	0.31	0.30	0.36	0.34	0.39	3	9	1.3	
	A8	0.19	0.15	0.13	0.18	0.19	0.15	0.18	0.15	0.15	0.15	0.13	0.15	0.16	0.15	0.11	4	15	1.6	
	A9	0.61	0.44	0.45	0.60	0.63	0.51	0.47	0.45	0.47	0.47	0.48	0.49	0.47	0.51	0.42	3	13	1.5	
	A12	0.53	0.62	0.64	0.56	0.53	0.55	0.58	0.63	0.58	0.54	0.61	0.66	0.58	0.60	0.41	6	10	1.5	
	A13	0.57	0.62	0.68	0.57	0.54	0.57	0.57	0.63	0.59	0.58	0.59	0.65	0.57	0.62	0.47	5	8	1.4	
	A14	0.75	0.84	0.85	0.75	0.71	0.77	0.79	0.84	0.83	0.80	0.76	0.80	0.76	0.79	0.70	4	6	1.2	
	A15	0.79	0.85	0.88	0.80	0.76	0.80	0.81	0.87	0.85	0.83	0.83	0.83	0.84	0.83	0.75	3	4	1.2	
	A16	0.81	0.85	0.80	0.79	0.78	0.79	0.79	0.80	0.84	0.81	0.78	0.80	0.81	0.82	0.74	1	3	1.1	
	A18	0.40	0.40	0.38	0.43	0.45	0.43	0.41	0.39	0.43	0.41	0.39	0.43	0.40	0.45	0.48	2	6	1.3	
	A20	0.28	0.29	0.25	0.30	0.29	0.28	0.29	0.28	0.30	0.29	0.26	0.25	0.28	0.27	0.28	3	5	1.2	
	A21	0.68	0.67	0.65	0.72	0.73	0.69	0.73	0.64	0.76	0.72	0.64	0.74	0.74	0.67	0.68	5	6	1.2	
	A22	0.66	0.53	0.65	0.64	0.65	0.56	0.59	0.51	0.60	0.56	0.50	0.41	0.53	0.55	0.56	5	12	1.6	
	A25	0.16	0.15	0.13	0.17	0.17	0.16	0.20	0.16	0.20	0.19	0.13	0.13	0.17	0.14	0.27	11	22	2.0	
	A26	1.09	0.90	0.86	1.11	1.12	1.04	1.10	0.94	1.09	1.07	0.89	0.91	1.02	1.14	1.05	7	9	1.3	
	A27	0.18	0.17	0.15	0.19	0.19	0.18	0.23	0.18	0.24	0.22	0.15	0.15	0.21	0.17	0.20	12	15	1.6	
	A31	0.24	0.13	0.17	0.28	0.30	0.22	0.28	0.18	0.26	0.25	0.16	0.20	0.20	0.20	0.20	20	22	2.2	
A32	0.34	0.38	0.38	0.42	0.41	0.32	0.36	0.37	0.38	0.36	0.38	0.36	0.38	0.40	0.17	3	16	2.5		
A33	0.34	0.33	0.33	0.37	0.37	0.33	0.33	0.33	0.35	0.33	0.31	0.34	0.34	0.36	0.26	2	8	1.4		
A34	0.15	0.15	0.13	0.15	0.15	0.13	0.15	0.15	0.14	0.13	0.12	0.14	0.14	0.14	0.09	1	12	1.7		
A35	0.41	0.32	0.32	0.42	0.43	0.35	0.39	0.33	0.41	0.38	0.30	0.32	0.35	0.34	0.31	10	12	1.4		
A36	0.65	0.77	0.76	0.64	0.58	0.68	0.71	0.74	0.72	0.71	0.69	0.69	0.70	0.71	0.67	1	7	1.3		
A38	1.01	1.03	0.98	1.07	1.08	1.04	1.04	1.03	1.07	1.04	1.03	0.96	1.03	1.01	1.05	2	3	1.1		
A39	0.32	0.27	0.27	0.33	0.33	0.28	0.30	0.27	0.29	0.27	0.27	0.27	0.27	0.30	0.23	3	9	1.2		
A40	0.37	0.46	0.49	0.39	0.37	0.42	0.40	0.48	0.43	0.44	0.46	0.45	0.44	0.46	0.36	3	9	1.4		
A41	0.39	0.33	0.32	0.41	0.41	0.35	0.41	0.33	0.39	0.37	0.32	0.32	0.35	0.35	0.31	8	11	1.3		
A42	0.63	0.90	0.84	0.78	0.71	0.75	0.83	0.89	0.87	0.86	0.82	0.81	0.82	0.76	ND	6	9	-		
A43	0.39	0.39	0.35	0.43	0.44	0.36	0.39	0.38	0.39	0.36	0.35	0.39	0.38	0.39	0.28	3	10	1.6		
Other	A45	0.12	0.15	0.16	0.15	0.14	0.29	0.17	0.17	0.16	0.15	0.14	0.13	0.15	0.14	ND	7	25	-	
	A46	1.01	1.11	1.09	1.22	1.20	1.10	1.06	1.11	1.10	1.11	1.09	1.09	1.05	1.02	ND	4	5	-	
	A47	0.42	0.32	0.42	0.47	0.41	0.42	0.37	0.35	0.39	0.38	0.40	0.41	0.33	0.27	ND	10	13	-	

A17 was used as an internal standard (Is), the values show peak areas. Each value is the average of three analytical runs. ND: not detected.

Table 8. Relative peak areas of additives in test Solution B.

Application	No.	Class I					Class II					Class III		Class IV	Class V	CV (%)		Max/Min		
		HP-5 MS	DB-5	ZB-5	DB-5 HT	ZB-5 HT	Rxi-5 HT	DB-5 MS	Rxi-5	Inert Cap	CP-Sil	VF-5 ms	TR-5 MS	BPX-5	007-5 MS	HT5	Inert Cap 3 lots		All column	
Is	B47	2.9×10^6		2.4×10^6		3.1×10^6		2.7×10^6		3.4×10^6		5.4×10^6		4.3×10^6		2.8×10^6		-	-	-
		3.8×10^6		3.9×10^6		3.0×10^6		2.3×10^6		4.3×10^6		3.2×10^6		2.1×10^6						
Lubricant	B1	0.68	0.73	0.82	0.77	0.78	0.87	0.73	0.76	0.69	0.70	0.65	0.71	0.73	0.89	0.99	4	12	1.5	
	B2	0.33	0.34	0.39	0.38	0.41	0.39	0.34	0.36	0.32	0.32	0.30	0.35	0.36	0.45	0.49	5	14	1.6	
	B3	0.63	0.66	0.72	0.71	0.71	0.75	0.65	0.67	0.61	0.60	0.56	0.61	0.67	0.82	0.91	4	13	1.6	
	B4	0.25	0.25	0.24	0.37	0.40	0.34	0.31	0.24	0.22	0.22	0.21	0.25	0.28	0.32	0.40	12	23	1.9	
	B5	0.62	0.67	0.70	0.80	0.85	0.77	0.77	0.61	0.56	0.55	0.52	0.62	0.71	0.80	1.03	10	19	2.0	
	B6	0.48	0.47	0.46	0.47	0.48	0.48	0.46	0.50	0.48	0.47	0.45	0.48	0.45	0.55	0.48	4	5	1.2	
	B7	0.21	0.21	0.23	0.25	0.28	0.26	0.20	0.20	0.20	0.20	0.20	0.23	0.21	0.28	0.22	4	13	1.4	
	B8	0.22	0.23	0.29	0.24	0.29	0.23	0.20	0.21	0.19	0.20	0.16	0.18	0.19	0.22	0.27	2	17	1.8	
	B9	0.51	0.50	0.48	0.52	0.55	0.52	0.50	0.53	0.50	0.48	0.47	0.53	0.48	0.56	0.50	3	5	1.2	
	B10	0.13	0.13	0.18	0.15	0.16	0.16	0.14	0.15	0.16	0.15	0.13	0.13	0.14	ND	0.11	17	13	-	
	B11	0.12	0.11	0.15	0.13	0.14	0.14	0.12	0.11	0.13	0.13	0.11	0.10	0.11	ND	0.08	12	15	-	
	B12	0.11	0.10	0.13	0.14	0.15	0.14	0.12	0.10	0.11	0.11	0.11	0.09	0.10	ND	0.08	10	18	-	
	B14	0.34	0.36	0.42	0.34	0.32	0.35	0.36	0.41	0.38	0.37	0.37	0.40	0.36	0.42	0.35	7	8	1.3	
	B15	0.36	0.35	0.39	0.33	0.33	0.36	0.38	0.40	0.38	0.38	0.34	0.38	0.35	0.44	0.32	8	9	1.4	
	B16	0.39	0.36	0.40	0.37	0.37	0.36	0.39	0.41	0.38	0.38	0.35	0.38	0.36	0.46	0.34	8	7	1.4	
	B17	0.42	0.42	0.41	0.42	0.44	0.37	0.40	0.40	0.36	0.41	0.38	0.42	0.40	0.45	0.37	13	6	1.3	
	B18	0.43	0.35	0.42	0.44	0.48	0.39	0.43	0.38	0.36	0.40	0.38	0.47	0.44	0.43	0.35	5	10	1.4	
	B19	0.32	0.30	0.31	0.39	0.41	0.38	0.33	0.31	0.32	0.31	0.29	0.35	0.29	0.31	0.37	11	12	1.4	
	B20	0.35	0.32	0.32	0.40	0.43	0.39	0.38	0.32	0.34	0.31	0.29	0.33	0.34	0.38	0.38	6	11	1.5	
	Antioxidant	B21	0.45	0.50	0.54	0.46	0.49	0.48	0.47	0.54	0.53	0.50	0.51	0.58	0.48	0.55	0.13	3	12	4.5
B22		0.73	0.80	0.95	0.83	0.88	0.81	0.75	0.88	0.89	0.81	0.83	1.02	0.77	0.97	0.54	16	40	1.9	
B23		0.72	0.77	0.93	0.89	0.94	0.81	0.76	0.86	0.87	0.78	0.78	1.01	0.77	0.98	0.53	10	22	1.9	
B24		0.48	0.50	0.58	0.62	0.67	0.53	0.54	0.50	0.54	0.47	0.51	0.62	0.49	0.45	ND	12	14	-	
B25		0.36	0.34	0.33	0.40	0.41	0.37	0.40	0.35	0.33	0.34	0.30	0.35	0.37	0.41	<0.1	12	15	>4	
B26		0.61	0.59	0.65	0.85	0.94	0.74	0.62	0.60	0.64	0.58	0.56	0.68	0.58	0.74	0.20	7	12	4.7	
B27		0.29	0.27	0.29	0.38	0.40	0.35	0.29	0.28	0.30	0.27	0.27	0.34	0.27	0.30	<0.1	8	25	>4	
B28		0.19	0.20	0.18	0.23	0.25	0.21	0.21	0.19	0.20	0.18	0.16	0.22	0.21	0.21	0.12	12	25	2.1	
B31		0.30	0.31	0.30	0.42	0.44	0.30	0.41	0.24	0.31	0.23	0.27	0.38	0.34	0.27	ND	4	16	-	
B36		0.28	0.29	0.26	0.34	0.35	0.28	0.30	0.25	0.28	0.24	0.23	0.31	0.30	0.27	ND	4	21	-	
Ultraviolet absorber	B37	0.16	0.19	0.20	0.28	0.29	0.24	0.18	0.10	0.10	0.10	<0.1	0.10	0.14	0.14	0.22	5	22	>2	
	B40	0.22	0.22	0.21	0.30	0.35	0.23	0.29	0.18	0.23	0.19	0.16	0.27	0.24	0.24	0.18	11	24	2.2	
	B41	0.36	0.36	0.42	0.45	0.47	0.39	0.36	0.40	0.39	0.36	0.36	0.46	0.37	0.43	ND	4	10	-	
	B42	0.12	0.13	0.13	0.14	0.16	0.12	0.12	0.13	0.12	0.12	0.11	0.14	0.13	<0.1	<0.1	4	21	-	
	B43	0.37	0.35	0.38	0.59	0.63	0.43	0.40	0.29	0.34	0.31	0.35	0.44	0.37	0.15	ND	2	24	-	
	B44	0.41	0.41	0.45	0.52	0.54	0.46	0.43	0.43	0.42	0.40	0.37	0.50	0.43	0.51	0.16	4	15	3.4	
	B45	0.23	0.23	0.23	0.29	0.32	0.25	0.24	0.22	0.24	0.21	0.19	0.26	0.24	0.26	0.16	4	15	2.0	
	B46	0.57	0.55	0.53	0.69	0.74	0.60	0.59	0.53	0.58	0.51	0.46	0.62	0.58	0.61	0.38	4	30	1.9	
B48	1.53	1.46	1.51	2.01	2.23	1.76	1.57	1.42	1.51	1.36	1.32	1.81	1.50	1.77	1.22	4	17	1.8		

B47 was used as an internal standard (Is), the values show peak areas. Each value is the average of three analytical runs. ND: not detected.

ticizers, and two benzophenone type ultraviolet absorbers could not be detected using some columns. Judicious selection of the column used for analysis of these additives is thus recommended.

REFERENCES

- [1] M. Peltzer, J. Wagner and A. Jimenez, "Migration Study of Carvacrol as a Natural Antioxidant in High-Density Polyethylene for Active Packaging," *Food Additives and Contaminants*, Vol. 26, No. 6, 2009, pp. 938-946.
- [2] J. H. Petersen and L. K. Jensen, "Phthalates and Food-Contact Materials: Enforcing the 2008 European Union Plastics Legislation," *Food Additives and Contaminants*, Vol. 27, No. 11, 2010, pp. 1608-1616.
[doi:10.1080/19440049.2010.501825](https://doi.org/10.1080/19440049.2010.501825)
- [3] A. Guart, F. Bono-Blay, A. Borrell and S. Lacorte, "Migration of Plasticizers, Phthalates, Bisphenol A, and Alkylphenols from Plastic Containers and Evaluation of Risk," *Food Additives and Contaminants*, Vol. 28, No. 5, 2011, pp. 676-685.
[doi:10.1080/19440049.2011.555845](https://doi.org/10.1080/19440049.2011.555845)
- [4] C. Simoneau, L. V. Eede and S. Valzacchi, "Identification and Quantification of the Migration of Chemicals from Plastic Baby Bottles Used as Substitutes for Polycarbonate," *Food Additives and Contaminants*, Vol. 29, No. 3, 2012, pp. 469-480.
- [5] L. L. Barnkob and J. H. Petersen, "Effect of Relative Humidity on the Migration of Benzophenone from Paperboard into the Food Simulant Tenax and Modeling Hereof," *Food Additives and Contaminants Part A*, Vol. 30, No. 2, 2013, pp. 395-402.
[doi:10.1080/19440049.2012.741717](https://doi.org/10.1080/19440049.2012.741717)
- [6] Y. Kawamura, K. Watanabe, K. Sayama, Y. Takeda and T. Yamada, "Simultaneous Determination of Polymer Additives in Polyethylene by GC/MS," *Journal of the Food Hygienic Society of Japan*, Vol. 38, No. 5, 1999, pp. 307-318.
[doi:10.3358/shokueishi.38.5_307](https://doi.org/10.3358/shokueishi.38.5_307)
- [7] Y. Kawamura, C. Tagai, T. Maehara and T. Yamada, "Additives in Polyvinyl Chloride and Polyvinylidene Chloride Products," *Journal of the Food Hygienic Society of Japan*, Vol. 40, No. 4, 1999, pp. 274-284.
[doi:10.3358/shokueishi.40.4_274](https://doi.org/10.3358/shokueishi.40.4_274)
- [8] Y. Kawamura, C. Tagai, T. Maehara and T. Yamada, "Simultaneous Determination Method of Additives in Polyvinyl Chloride," *Journal of the Food Hygienic Society of Japan*, Vol. 40, No. 3, 1999, pp. 189-197.
[doi:10.3358/shokueishi.40.3_189](https://doi.org/10.3358/shokueishi.40.3_189)
- [9] Y. Kawamura, R. Yonezawa, T. Maehara and T. Yamada, "Determination of Additives in Food Contact Polypropylene," *Journal of the Food Hygienic Society of Japan*, Vol. 40, No. 2, 2000, pp. 154-161.
[doi:10.3358/shokueishi.41.154](https://doi.org/10.3358/shokueishi.41.154)
- [10] Y. Abe, M. Yamaguchi, M. Mutsuga, Y. Hirahara and Y. Kawamura, "Survey of Plasticizers in Polyvinyl Chloride Toys," *Journal of the Food Hygienic Society of Japan*, Vol. 53, No. 1, 2012, pp. 19-27.
[doi:10.3358/shokueishi.53.19](https://doi.org/10.3358/shokueishi.53.19)
- [11] European Commission, "Commission Regulation 10/2011/EU of 14 January 2011 on Plastic Materials and Articles Intended to Come into Contact with Food," *Official Journal of the European Union*, 2011, pp. 1-89.