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Application News

GC Brevis™ GC-2050

Residual Solvents Analysis from Flexible Packaging Material using Brevis GC-2050

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User Benefit

- The small-sized instrument reduces the occupied space in the laboratory.
- The automatic sampling system achieves easy operation in headspace sampler system HS-20 NX
- Nitrogen that is easy to obtain was employed as carrier gas.

■ Introduction

Flexible packaging material includes materials made with such as polyethylene, polypropylene, and polyolefin. They are widely utilized especially in food packing and containers. Manufacturing these materials involves a variety of organic solvents for printing and gluing, and thus, testing for these residual solvent is remarkably important especially for food relevant products.

Gas chromatography (GC) with Headspace sampler (HS) is useful for this type of analysis because the residual solvents are highly volatile. This application news introduce the example of the residual solvents analysis in flexible packing materials with HS-20, referring the manual published from Flexible Packaging Hygiene Association.

■ Instruments

Our new gas chromatograph Brevis GC-2050 is notably compact sized and can reduce the occupied space in the laboratory. It is one of the best options for analysis of residual solvents in soft packaging material when it is connecting to HS-20.

Fig. 1 HS-20 NX＋ Brevis™ GC-2050

■ Quantification for Headspace Sampler

Headspace sampler can deal with both liquid and solid samples. When the sample shape is liquid, chemical equilibrium between the sample liquid and its vaporized gases can be achieved some time after the lid is hermitically sealed. This equilibrium headspace gases can be analyzed with GC for quantification of the target solvents.

On the other hand, when the sample shape is solid, generated gases are analyzed under the specific condition. When a sample is induced into the vial and the lid is sealed tightly, the gas compounds would be vaporized from the solid sample in the vial. This generated gases should be analyzed for quantification.

In this article, the sample was soft packaging material that is solid, and the generated gases from the sample was analyzed.

■ Analysis of the Standards

In this experiments, typical 21 residual solvents were selected as target. 0.5 g of these chemical standards were weighed and mixed for creating the standard solution. 0.4, 1, 2, and 4 µL of this standard solution was introduced with a micro syringe into each vial for calibration curve sample. Calibration curve was created with the peak area and the weight of the standard solution, followed by calculating each compound weight from their density.

Table 1 Analytical Condition

Model	:	Brevis GC-2050
Autosampler	:	HS-20 NX (Loop)
[HS-20 NX]		
Oven temp.	:	80 °C (30 min)
Sample Line temp.	:	150 °C
Transfer Line temp.	:	150 °C
Vial Stirring	:	OFF
Vial Pressurization Time	:	1.0 min
Pressure Equilib. Time	:	0.1 min
Loading Time	:	0.5 min
Load Equilib. Time	:	0.1 min
Injection Time	:	1.0 min
Needle Flush Time	:	5.0 min
Vial Pressure	:	100 kPa (N2)
Injection Volume	:	1.0 mL
Needle Flush Time	:	5.0 min
[GC-2050]		
Carrier Gas	:	N2
Carrier Gas Control	:	Constant Linear Velocity (25 cm/sec)
Column	:	SH-WAX (P/N 227-75893-60) (60 m × 0.25 mm I.D., 0.25 µm)
Column temp	:	50 °C (3 min) – 10 °C/min – 200 °C (5 min)
Detector temp	:	250 °C
FID H2 Flow Rate	:	32 mL/min
FID Make up Flow Rate	:	24 mL/min
FID Air Flow Rate	:	200 mL/min

■ Result of the Standard Solution

After the vials that the standard solution contain were heated to 80°C for 30 min, headspace sampler automatically introduced the gases into GC. The obtained chromatogram showed 21 organic solvent peaks that were clearly separated. Table 2 shows the calculated weight per unit area of each compound. The calibration curve referring to it resulted in sufficient linearity. The calibration curve of i-Propanol, that was the most abundant compound in the material sample, was shown in Fig. 3 as an example.

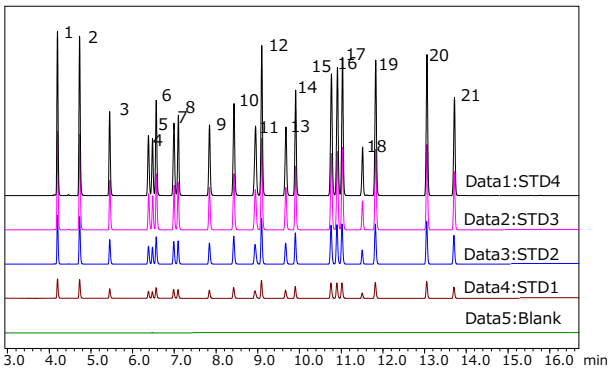
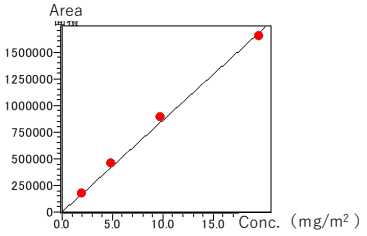


Fig. 2 Chromatogram of Standard Solution

Table 2 Weight per unit area of each compound (mg/m²)

No.	Compound	STD1	STD2	STD3	STD4
1	n-Hexane	1.96	4.90	9.80	19.60
2	Cyclohexane	1.96	4.90	9.79	19.58
3	Acetone	1.97	4.91	9.83	19.65
4	Ethyl acetate	1.98	4.95	9.89	19.78
5	Methanol	1.98	4.94	9.88	19.76
6	MEK	1.95	4.88	9.76	19.53
7	i-Propanol	1.96	4.90	9.80	19.60
8	Ethanol	1.95	4.88	9.77	19.53
9	Propyl acetate	1.97	4.94	9.87	19.74
10	MiBK	1.98	4.94	9.89	19.78
11	N-Propanol	1.95	4.87	9.74	19.48
12	Toluene	1.98	4.94	9.88	19.75
13	Butyl acetate	1.95	4.87	9.75	19.49
14	i-Butanol	1.96	4.89	9.79	19.57
15	Ethyl benzene	1.98	4.94	9.88	19.77
16	p-Xylene	1.95	4.89	9.77	19.54
17	m-Xylene	1.96	4.90	9.81	19.62
18	2-Methoxy ethanol	1.99	4.97	9.94	19.88
19	o-Xylene	1.97	4.93	9.87	19.74
20	Styrene	1.95	4.88	9.76	19.53
21	Cyclohexanone	1.96	4.91	9.81	19.63



Y = aX + b
a = 85884.7
b = 0
R^2 = 0.9980243
R = 0.9990116
RSS = 5.072276e+009

External standard
Linear
Forced to zero
Weight: none

Fig. 3 Calibration curve of i-Propanol
(1.96, 4.90, 9.80, 19.6 mg/m²)

<Reference>
Manual for handling instruments with manufacturing of flexible packaging materials
6th edition (Oct, 2017)
Flexible Packaging Hygiene Association

Repeatability for minimum calibration point were also evaluated. The relative standard deviation (%RSD) was less than 3% for all the compounds (n=3).

Table 3 Peak area repeatability (%RSD)

No.	COmpounds	1st	2nd	3rd	Mean	RSD(%)
1	n-Hexane	337,380	325,333	341,522	334,745	2.05%
2	Cyclohexane	351,074	338,344	355,696	348,371	2.11%
3	Acetone	180,257	172,488	181,439	178,061	2.23%
4	Ethyl acetate	142,804	137,376	144,460	141,547	2.14%
5	Methanol	126,078	119,184	125,753	123,672	2.57%
6	MEK	213,567	204,944	215,555	211,355	2.18%
7	i-Propanol	179,964	172,619	181,710	178,098	2.21%
8	Ethanol	171,891	164,234	173,057	169,727	2.31%
9	Propyl acetate	177,410	171,083	179,736	176,076	2.08%
10	MiBK	244,347	235,704	247,613	242,555	2.07%
11	N-Propanol	211,049	202,243	212,888	208,727	2.23%
12	Toluene	372,088	358,152	376,439	368,893	2.11%
13	Butyl acetate	190,326	183,935	192,815	189,025	1.98%
14	i-Butanol	247,805	238,353	250,401	245,520	2.11%
15	Ethyl benzene	355,974	343,573	360,395	353,314	2.02%
16	p-Xylene	353,231	340,618	357,182	350,344	2.02%
17	m-Xylene	354,848	342,406	359,004	352,086	2.00%
18	2-Methoxy ethanol	110,689	106,468	111,819	109,659	2.10%
19	o-Xylene	358,581	346,076	362,893	355,850	2.00%
20	Styrene	362,423	349,375	366,356	359,385	2.02%
21	Cyclohexanone	246,664	238,128	250,022	244,938	2.04%

■ Result of the material sample

All the five materials (A, B, C, D, and E) were cut into a piece. Each piece size was 5cm×16cm. A single piece of A and B placed into a vial, respectively. Three pieces of C, D and E were placed into a vial, respectively, as these generated less quantity of gases.

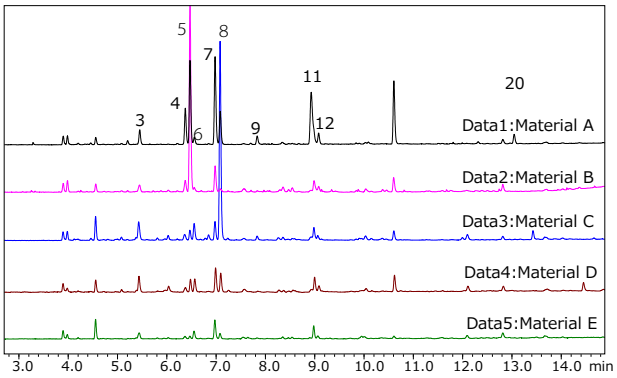


Fig. 4 Chromatogram of the material samples

Table 4 Quantification result (mg/m²)

No.	Compound	Material A	Material B	Material C	Material D	Material E
3	Acetone	0.017	0.01	0.008	0.008	0.003
4	Ethyl acetate	0.056	0.02	0.004	0.003	-----
5	Methanol	0.14	0.319	0.005	0.007	-----
6	MEK	0.007	-----	0.006	0.005	0.003
7	i-Propanol	0.107	0.031	0.008	0.01	0.008
8	Ethanol	0.042	-----	0.082	0.008	0.002
9	Propyl acetate	0.012	-----	-----	-----	-----
11	n-Propanol	0.087	0.015	0.005	-----	0.004
12	Toluene	0.007	0.004	0.001	0.001	-----
20	Styrene	0.006	-----	-----	-----	-----

■ Summary

This article introduced the analysis of residual solvents in flexible packaging materials, using HS-20 and Brevis 2050 system with Nitrogen carrier. Column separation, calibration curve and repeatability all showed a sufficient result.

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