

## 「Application Note」

# Analysis of Residual Solvent in Food Packaging Material by Headspace-GC

- GC Application



### Abstract

In the market, there are fancy and colorful food packaging used for drawing customer's attentions. The organic solvents based printing inks are mostly used on these packaging and these may affect the flavor of food and cause harmful effect to human health.

The use of residual solvents in food packaging is strictly regulated to prevent potential migration to the food. Korea Ministry of Food and Drug Safety regulates the residual toluene in packaging material to be less than 2 mg/m<sup>2</sup>. And, the rest of residual solvents are regulated by Korea Food Industry Association (KFIA) of which the total amount should be less than 6 mg/m<sup>2</sup>.

In this study, the analysis of residual solvent in food packaging material was carried out using a headspace autosampler-GC instead of manual headspace extraction to improve efficiency and repeatability.

## Headspace Instruments



**Fig 1. Versa Headspace-ChroZen GC**

### **Versa Headspace Autosampler (P/N: SC150800200)**

Static Headspace

Injection Type : Loop type

Injection Volume: 1 mL, standard / 100  $\mu\text{l}$ ~ 5 mL(option)

Sample Capacity : 20 vials (22 mL)



**Fig 2. PAL Headspace-ChroZen GC**

### **ChroZen PAL RSI Autosampler (P/N: 6501011600)**

Static and Dynamic Headspace

Injection Type : Gas-tight Syringe(Heated) type

Syringe Volume: 1 mL / 2.5 mL, standard / 5 mL

Sample Capacity : 180 vials (20 mL, 4 tray holders)

\* In case of the need of multiple headspace such as static/dynamic headspace and high throughput up to 180 samples, ChroZen PAL RSI autosampler is recommended.

## Instruments and Software

- Versa Headspace-ChroZen GC System

Item	Description	Part No.
Oven	ChroZen GC Mainframe Assembly with UPC Detector Board Unit	6701012502
Inlet	Capillary Inlet Assembly for ChroZen GC	6701012550
Detector	FID Assembly for ChroZen GC	6701012590
Autosampler	Versa Static Headspace Vial Sampler 230V.	SC150800200
CDS	YL-Clarity software for single instrument of YoungIn Chromass GC	5301011020
Column	DB-5MS (30 m, 0.32 mm, 0.25 $\mu$ m)	-
ACC	Start-up kit (without GC capillary column)	1601011110
	Vials, 22mL headspace, w/20mm top, 125 per pack.	SC144440024
	Crimp Cap. For headspace septa and 20mm top headspace vials, 125 per pack.	SC144436000
	Butyl rubber septa without teflon face. For 20mm top headspace vials, 125 per pack. Recommended for temperatures up to 100 °C. Color coded grey.	SC144385043

## Sample and Standard Preparation

- **Sample Preparation**

- ① Cut the sample into a size of 10 cm x 8 cm (0.008 m<sup>2</sup>) and cut again into smaller pieces.
- ② Put the small pieces of cut sample into a 20 mL headspace vial and seal with a cap.
- ③ Inject 1  $\mu$ l of tetra-hydrofuran as an internal standard through the cap.
- ④ Place the vials in the tray of headspace autosampler.

- **Standard Preparation and Calibration**

- ① Transfer 1.0 mL each from 5 stock solvent solution (toluene, methanol, ethyl acetate, methylethylketone, isopropylalcohol) in a storage vial and mix them.  
⇒ Standard mixture A
- ② Add 2.0 mL of heptane into 2.0 mL of standard mixture A and mix it.  
⇒ Standard mixture B

- ③ Add 2.0 mL of heptane into 2.0 mL of standard mixture B and mix it.  
⇒ Standard mixture C
- ④ Add 2.0 mL of heptane into 2.0 mL of standard mixture C and mix it.  
⇒ Standard mixture D
- ⑤ Add 2.0 mL of heptane into 2.0 mL of standard mixture D and mix it.  
⇒ Standard mixture E
- ⑥ Transfer 2.5  $\mu\text{l}$  from each standard mixture into 5 sealed 20 mL headspace vials each.
- ⑦ Inject 1  $\mu\text{l}$  of tetra-hydrofuran(IS) through the cap.
- ⑧ Run the headspace-GC and draw calibration curve by internal standard calibration with 5 standard points.

Solvent	Specific Gravity
Toluene	0.867
Methanol	0.792
Iso-propyl alcohol	0.785
Methyl ethyl ketone	0.805
Ethyl acetate	0.902

**Table 1. Specific gravity of solvents**

< Equation for calculating concentration of residual solvent >

$$\text{Concentration of solvent} = \frac{\text{Injection volume of solvent} \times \text{Specific gravity}}{\text{Area of sample}}$$

e.g. Concentration of toluene in standard mixture A =  $\frac{0.5 \mu\text{l} \times 0.867}{0.008 \text{ m}^2} = 54.188 \text{ mg/m}^2$

	Standard Mixture A	Standard Mixture B	Standard Mixture C	Standard Mixture D	Standard Mixture E
Toluene	54.188	27.094	13.547	6.773	3.387
Methanol	49.500	24.750	12.375	6.188	3.094
IPA	49.063	24.531	12.266	6.133	3.066
MEK	50.313	25.156	12.578	6.289	3.145
EA	56.375	28.188	14.094	7.047	3.523

**Table 2. Concentration of standard mixture (mg/m<sup>2</sup>)**

## Analytical Conditions

Headspace conditions	
Valve Oven Temp.	80 °C
Transfer Line Temp.	120 °C
Platen/sample Temp.	80 °C
Sample Equal. Time	30 min
Sample Mixer	On
Loop Volume	1 ml
GC Cycle Time	12 min
GC/FID conditions	
Column	DB-5 (30 m x 0.32 mm x 0.25 µm)
Inlet	Temperature: 230 °C Spilt Ratio: 30:1 Injection Volume: 1 mL Carrier Gas: N <sub>2</sub> (99.999 %)
Oven	Oven temperature program : 50 °C (3 min) → 15 °C/min → 110 °C
Detector	FID temperature: 250 °C

**Table 3. Headspace-GC/FID conditions**

## Calibration Curve

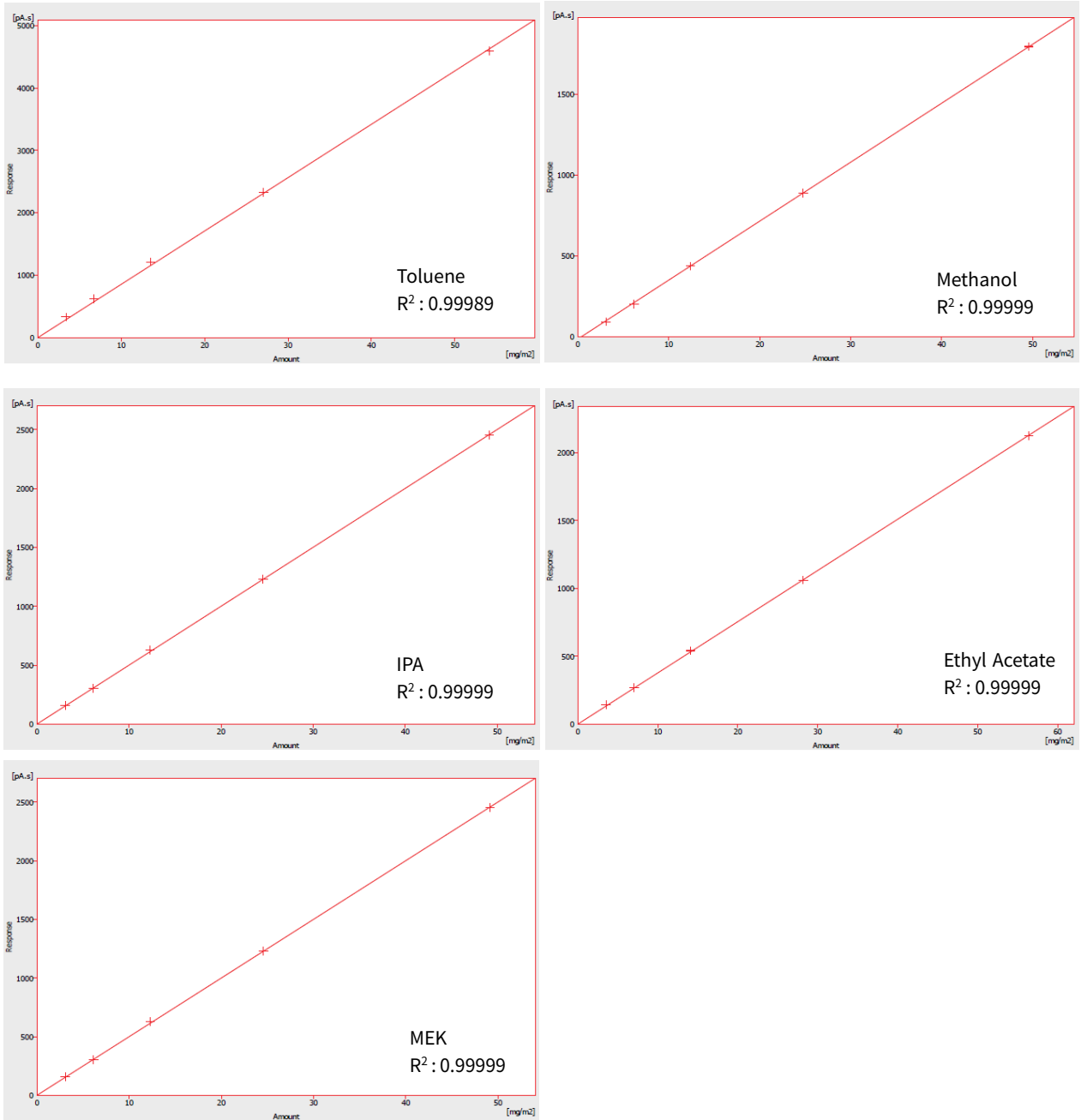
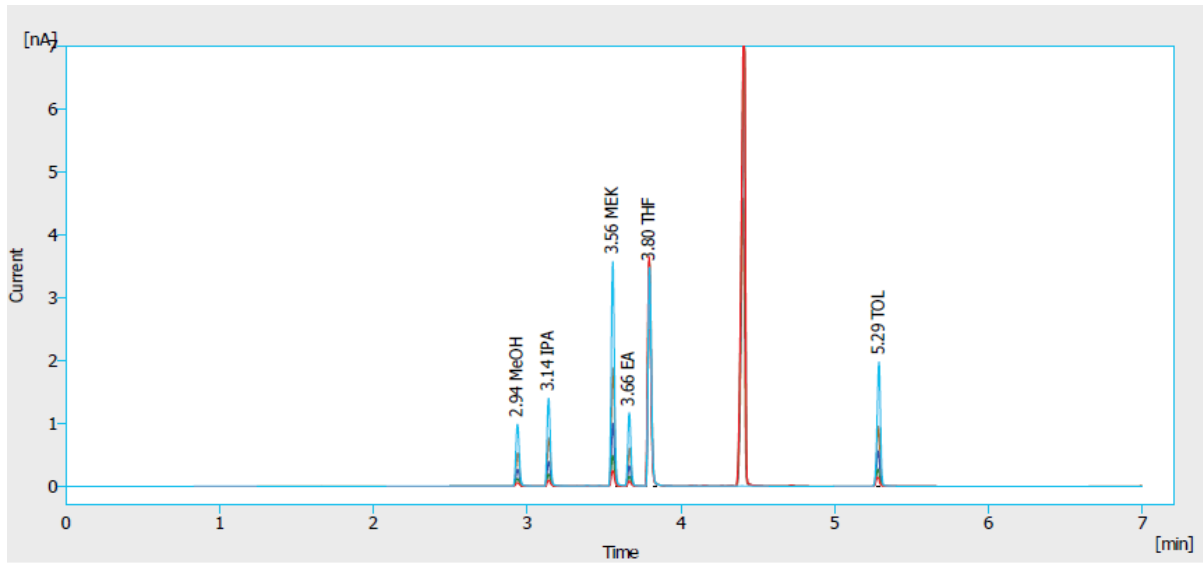
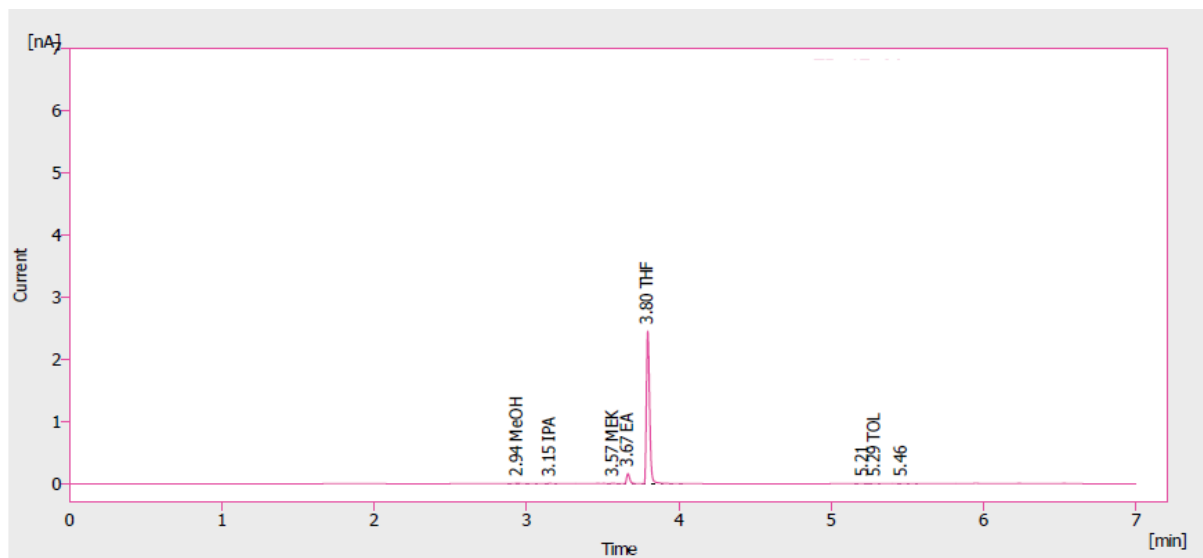


Fig 3. Calibration curve (5 standard points)

## Result



**Fig 4. Chromatogram overlay of standards**



**Fig 5. Chromatogram of sample**

Solvent	Amount (mg/m <sup>2</sup> )
Toluene	0.032
Methanol	0.360
Iso-propyl alcohol	0.115
Methyl ethyl ketone	0.059
Ethyl acetate	7.430

**Table 4. Amount of residual solvent in sample**

## Conclusion

The established test method using headspace-ChroZen GC to analyze residual solvent in food packaging ensures greater repeatability and higher productivity than the traditional test method.

## Reference

- Standards and Specifications for Food Utensils, Containers and Packages, Korea Ministry of Food and Drug Safety.
- Standards and Specifications of Residual Solvents in Food Packaging Materials, Korea Food Industry Association.
- USP 467, Standard Test Method for Residual Solvents



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