

# Ultra Fast Determination of VOCs in Packaging Materials by Headspace GC

Thermo Fisher Scientific Inc., Milan, Italy

## Key Words

- TRACE GC Ultra
- TRACE TR-1701 Column
- Volatile Organic Compounds
- Ultra Fast GC
- Headspace Analysis

## Introduction

To prevent excessive amounts of residual solvents (VOCs) in the final packaging of material products, whose presence would seriously compromise their quality, an efficient on-line control over the whole process is required. Due to the high speed of production, however, only very limited analytical times are allowed for an effective real-time monitoring, which is able to guarantee a uniform quality of each specific lot. Conventional headspace GC analyses of packaging materials require about 30-60 minutes, a time equivalent to the printing of thousands of meters of flexible materials.

In this application, rapid headspace analysis of VOCs in packaging materials is performed using a Thermo Scientific TRACE GC Ultra™ equipped with the Ultra Fast Module option (UFM). GC analysis of various residual solvents in different packaging materials was achieved in only 1-2 minutes with a 10-20 fold speed increase with respect to conventional GC methods. The analysis time is compatible with the production flow rate of the packaging material.

## Instrumentation

A TRACE GC Ultra equipped with SSL injector, Fast Flame Ionization Detector (FID) and Ultra Fast Module option (UFM) was used (Figure 1). Featuring direct resistive heating of capillary columns, the UFM achieves very fast temperature programming rates (as high as 20 °C/s), not otherwise attainable with the use of conventional air circulating GC ovens. The fused silica's very low thermal mass also allows rapid cool-down cycles (around one minute from 350 °C to 50 °C), thus further decreasing the overall run-to-run time.

The module, containing the capillary column, the heating element, and the temperature sensor, is housed in the GC oven and connected to standard GC split-splitless injector and GC detectors, just as any normal capillary column. Capillary columns with different lengths and diameters featuring different stationary phases can be incorporated in the UFM module.

In this application, all samples were analyzed using a 5 m, 0.1 mm i.d., 0.2 µm film thickness Thermo Scientific TRACE™ TR-WAX or a 10 m, 0.1 mm i.d., 0.1 µm film thickness Thermo Scientific TRACE TR-1701 column, incorporated in the UFM module. Injections were performed using the Thermo Scientific TriPlus™ static Headspace Autosampler (Figure 2). In addition to advanced robotic control of vial and syringe operations over a 3D space, the TriPlus™ Autosampler is a

user-friendly, versatile and high performance instrument, also capable of working in liquid injection mode.



Figure 1: TRACE GC Ultra with the Ultra Fast Module



Figure 2: TriPlus Autosampler installed on a TRACE GC Ultra

## Methods

Calibration was performed introducing and evaporating known amounts of standard mixture into headspace vials having 20 mL capacity. Packaging material samples were prepared by cutting slices of 100 cm<sup>2</sup> from different packaging products (uncoated, coated and laminated) of different layer composition. A cut was performed on the region of average ink coating of the single film. Flexographic ink samples commercially available for general purpose were prepared introducing 20 µL directly into the 20 mL vial. Up to six samples were simultaneously incubated. In such a way, the waiting times between consecutive GC runs were eliminated. Analytical conditions are reported beside each chromatogram.

## Results

### Linearity and Repeatability

Calibration data are reported in Figures 3 and 4. A good calibration curve linearity was found for all components.

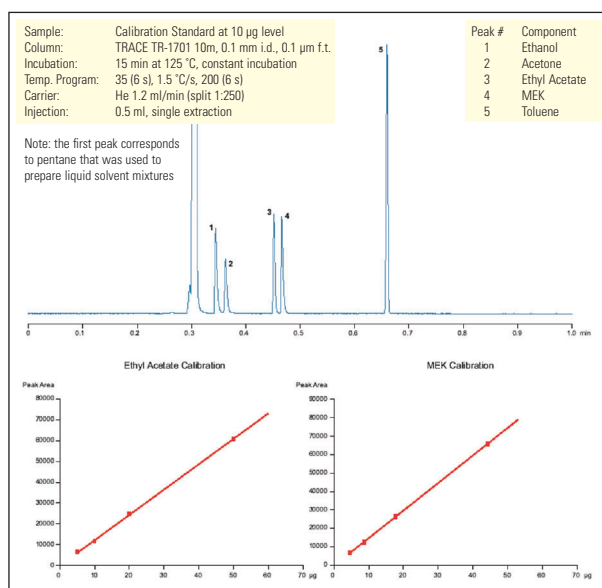


Figure 3: Chromatogram of the calibration standard containing five solvents and calibration curves for two of the components

Data obtained from five repetitions of the 10 µg calibration level (Figure 4) shows excellent retention times and peak areas repeatability. The comparison between the

standard deviation of the retention times and the peak width at half height of the mixture components, proves that the system allows safe and unambiguous identification of the compounds with a 99.7 % level of probability.

### Food Packaging Analysis

Figures 5A and 5B show the analysis, performed in only one minute, of two different films used in food packaging. Some solvents were identified and quantified down to 0.1 mg/m<sup>2</sup>.

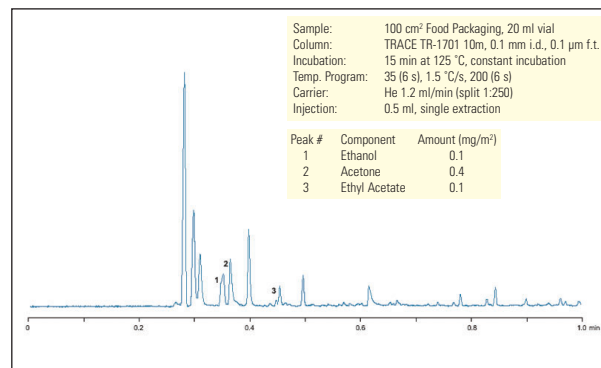


Figure 5A: Metallized polypropylene film analysis

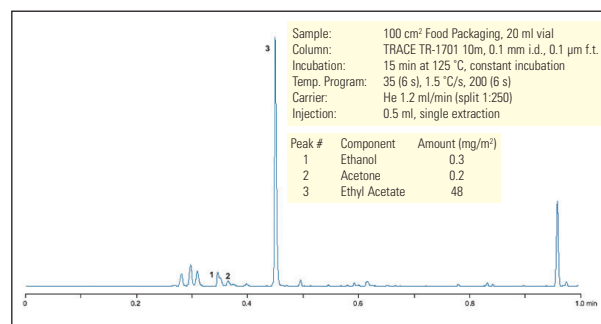


Figure 5B: Metallized polyester/polyethylene film analysis

### Flexographic Ink Analysis

Analyses of solvents released by two different commercial inks used for general purpose packaging are shown in Figures 6A and 6B. Solvents concentration was in the range of 0.1 % to 5 %.

#### RETENTION TIMES, AREA REPEATABILITY AND LINEARITY OF CALIBRATION

PEAK	PW 1/2 (MS)	RETENTION TIMES			PEAK AREAS		R <sup>2</sup>
		MEAN (SEC)	SD (MS)	6XSD(1)	MEAN	RSD %	
Ethanol	240	20.66	32	192	12235	1.95	0.9992
Acetone	240	21.77	30	180	7754	2.01	0.9989
Ethyl Acetate	200	27.1	26	156	11595	1.12	0.998
MEK	200	28.00	32	192	12513	1.87	0.9998
Toluene	180	39.61	18	108	29800	1.59	0.9998

(1) In a ± 3 SD span there is a probability of 99.7 % for a measure to fall inside this range

Figure 4: Repeatability data calculated over a set of five repetitions of the 10 µg calibration level and linear correlation coefficient for all the calibration curves.

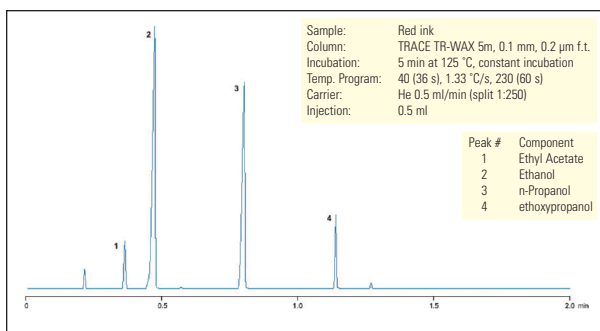


Figure 6A: Sample 1 - Red ink analysis

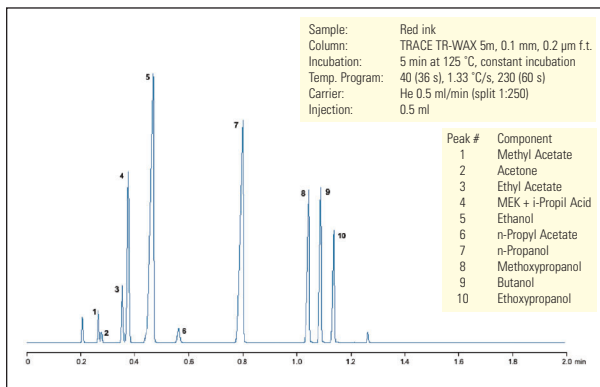


Figure 6B: Sample 2 - Red ink analysis

## Conclusions

Rapid Headspace analysis of residual solvents (VOCs) in packaging materials can successfully be performed with high precision and accuracy, using the TRACE GC Ultra equipped with the Ultra Fast GC option. The combination of the Ultra Fast GC technique with TriPlus HS Autosampler, featuring optimization of the incubation times, allows the analyst to achieve a total analysis cycle time of about five minutes per sample, in line with the requirements of a production flow rate of packaging materials.

## References

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Authors: Riccardo Facchetti, Andrea Cadoppi

In addition to these offices, Thermo Fisher Scientific maintains a network of representative organizations throughout the world.

### Australia

+61 2 8844 9500

### Austria

+43 1 333 50340

### Belgium

+32 2 482 30 30

### Canada

+1 800 532 4752

### China

+86 10 5850 3588

### Denmark

+45 70 23 62 60

### France

+33 1 60 92 48 00

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