An Economical EN71 Solution for VOCs Analysis from Toys Emission Using an Agilent 7667A Mini TD and an Agilent 5977E MSD System

Application Note

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Abstract

Following the European Standard EN 71 part 11, an economical solution for the determination of volatile organic compounds (VOCs) was developed on an Agilent 7820A GC with an Agilent 5977E GC/MSD System configured to an Agilent 7667A Mini Thermal Desorber. Excellent linearity was obtained with calibration ranging from 0.02 µg to 2 µg for 10 volatile target compounds, and from 0.05 µg to 5 µg for cyclohexanone and isophorone. A plastic toy and a cloth doll sample were spiked with target chemicals and tested using this method. The results showed superior repeatability.

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Introduction

The potential exposure and toxicological effects of volatile organic compounds present the greatest risk to children's health. The European Standard EN 71-11 was mandated by the European Commission to address the risks presented by organic chemical compounds in toys. The analysis of toys or toy materials should be performed according to the sampling procedures specified in the standard, and the method performance must achieve the accuracy, precision, and sensitivity described in the standard. The Agilent 5977E GC/MSD System is a bundle system that provides adequate sensitivity and outstanding repeatability for this routine analysis.

Experimental

Chemicals

All of the independent chemicals including methanol, methylene chloride, hexane, benzene, trichloroethylene, toluene, ethylbenzene, *p*-xylene, *m*-xylene, *o*-xylene, cyclohexanone, 1,3,5-trimethylbenzene, nitrobenzene, and isophorone were purchased from J&K Scientific ltd. Purity for each chemical was 99.5%. Methanol was used as solvent.

Calibration and sample preparation

The liquid standard solutions, comprising the six concentration levels of target compounds in methanol listed in Table 2, were prepared following the standard method EN71-11. Then each standard solution was introduced onto the Tenax-TA tube in a 80 mL/min flow of pure nitrogen gas using the calibration standard solution loading rig from Markes International.

The EN71 standard specifies that the toy samples must be extracted in a thermal extractor unit at 40 °C for 15 minutes with a 20 mL/min flow of nitrogen gas. The Adsorbent Tube Injector System (ATIS) from SUPELCO was used for this application development. The ATIS is a sample preparation device that provides a quick way to transfer the VOCs from the solid sample onto an adsorbent tube with a continuous flow of inert gas at a fixed temperature. The toy samples were cut into small pieces and placed into the glassware extractor. Prior to analysis, a 1-µL standards mixture of known concentration was spiked into the toy samples.

Instrument conditions

The optimized instrument conditions are listed in Table 1.

Table 1. Optimized Instrument Conditions for VOCs Emission Analysis

Agilent 7667A Mini TD		**Agilent 7820A GC with an Agilent 5977E GC/MSD System**	
Tube sorbent	Tenax TA	Inlet	250 °C
Tube temperature program	35 °C (0 minutes) to 300 °C (3 minutes) at 500 °C/min	Carrier	He, 1 mL/min
		Split ratio	50:1
Injection start time	2 minutes	Column	Agilent DB-624, 60 m × 0.25 mm, 1.4 µm (p/n 122-1364)
Transfer line	180 °C	Oven program	50 °C (3 minutes) to 220 °C (5 minutes) at 10 °C/min
Valve box	175 °C		
Purge	100 mL/min for 0.5 minutes	MS scan range	35–300
Cleaning	100 mL/min at 310 °C for 5 minutes		

Results and Discussion

The chromatogram in Figure 1 shows the lowest level of standards desorption result. As seen in the figure, all the peaks are symmetrical without tailing, and the responses satisfy the standard.

Excellent repeatability was proven by five runs of standard samples. Each standard sample was made by manually loading liquid standards into the tube. All of the RSD(%) results, both low level and high level standards (namely level 1 in Table 2) loading, are below 4%, which strongly demonstrates the superior reliability of the instrument system including the Thermal Desorber and GC/MS.

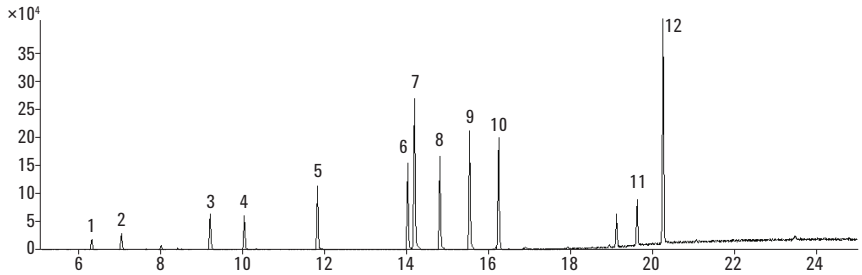

Figure 1. Chromatogram of level 1 liquid standards on a Tenax tube.

Table 2.. Calibration Levels, Linearity, and Repeatability of Target Compounds

No.	Compounds	Level 1 (µg)	Level 2 (µg)	Level 3 (µg)	Level 4 (µg)	Level 5 (µg)	Level 6 (µg)	Linearity (R^2)	RSD (%) Spike 1	RSD (%) Spike 2
1	Dichloromethane	0.02	0.04	0.1	0.2	1	2	0.9998	17.9%	29.2%
2	Trichloroethylene	0.02	0.04	0.1	0.2	1	2	0.9995	4.6%	3.1%
3	*n*-Hexane	0.02	0.04	0.1	0.2	1	2	0.9999	4.4%	2.4%
4	Benzene	0.02	0.04	0.1	0.2	1	2	0.9998	4.6%	3.5%
5	Toluene	0.02	0.04	0.1	0.2	1	2	0.9998	4.3%	3.6%
6	Ethylbenzene	0.02	0.04	0.1	0.2	1	2	0.9994	4.2%	2.5%
7	*m/p*-Xylene	0.04	0.08	0.2	0.4	2	4	0.9982	4.0%	3.8%
8	*o*-Xylene	0.02	0.04	0.1	0.2	1	2	0.9984	4.4%	3.8%
9	1,3,5-Trimethylbenzene	0.02	0.04	0.1	0.2	1	2	0.9983	4.0%	14.4%
10	Nitrobenzene	0.02	0.04	0.1	0.2	1	2	0.9994	3.0%	6.4%
11	Cyclohexanone	0.05	0.1	0.25	0.5	2.5	5	0.9989	3.4%	28.5%
12	Isophorone	0.05	0.1	0.25	0.5	2.5	5	0.9992	4.5%	24.2%

Two types of real samples, plastic toys and soft toys, were spiked and tested using this method. Figures 2 and 3 show the overlapped chromatograms for each type of sample. The detailed information of the target compounds regarding retention time, calibration linearity, and RSD(%) results for spiked samples are listed in Table 2.

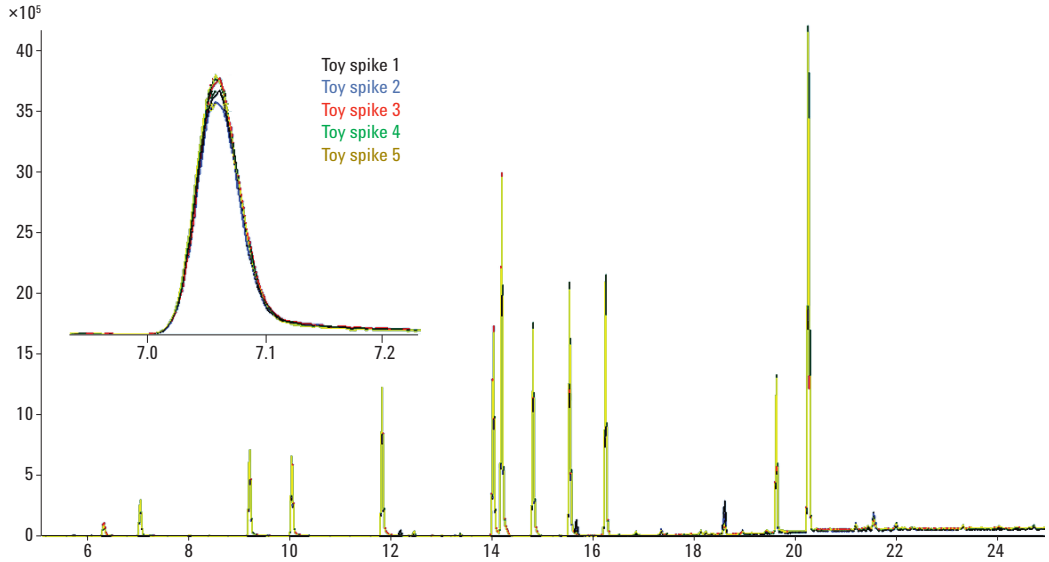

Figure 2. Overlapped chromatograms of five spiked plastic toy samples (Spike 1).

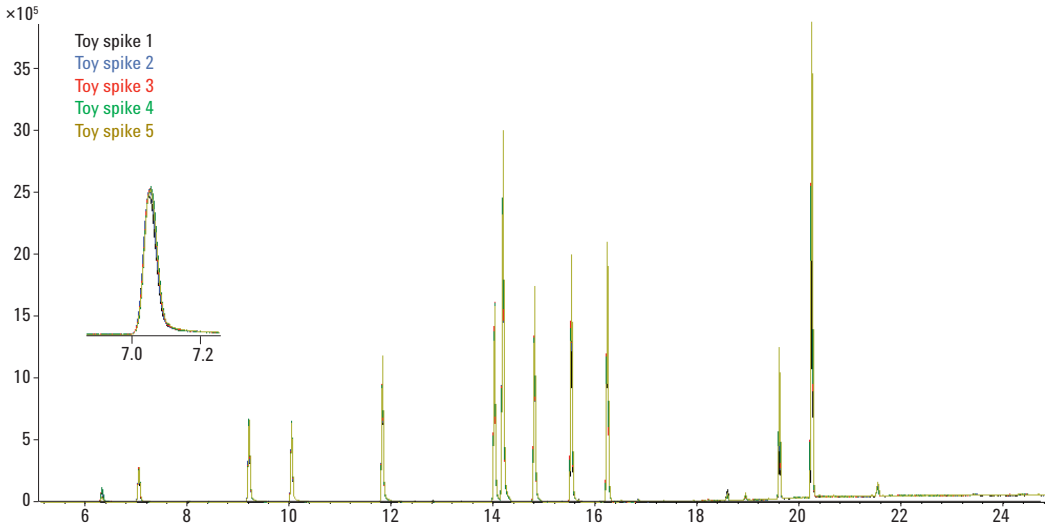

Figure 3. Overlapped chromatograms of five spiked soft toy samples (Spike 2).

Conclusion

Using an Agilent 7820A GC with an Agilent 5977E GC/MSD System configured to an Agilent 7667A Mini Thermal Desorber to search for and find volatile organic compounds in children's toys meets the requirements of standard EN71-11. The test results in this application note demonstrate adequate sensitivity, linearity, and outstanding repeatability for this routine analysis.

Reference

1. EN71-11:2005 Safety of toys-Part11 Organic compounds - Methods of analysis

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