APPLICATION NOTE

Gas Chromatography

AUTHOR

Thomas Dillon
PerkinElmer, Inc., Shelton, CT USA

GC-FID: Analysis of Residual Solvents and Monomers in Plastic

Introduction

Plastics are in use all around us; in medical devices, food packaging, toys, aerospace materials, insulation, and innumerable other applications. Different types of plastics are used for these diverse applications, but all plastic manufacturing has a few common raw material needs, including monomers and solvents. Plastic manufacturing workflows are designed to use all monomer input in the formation of the plastic polymer, and to remove all solvents from the final plastic product. However, residual levels of monomers and solvents often remain in the finished product.

Residual monomers and solvents can present health risks to end users or impact the performance of the final product. For example, leaching of residual monomers or solvents from dental appliances can cause localized irritation, inflammation, and pain in the mouth, or even a systemic allergic reaction. Medical devices and materials are subject to broad regulatory requirements pertaining to patient safety. In non-medical applications—automotive and aviation materials, for example—residual monomers or solvents can result in reduced usable lifespan, safety concerns, or even catastrophic failure. It is vital that plastic manufacturers have an easy method to prepare plastic samples for analysis, and analytical instruments that provide high throughput and the sensitivity required for the most stringent purity requirements.

This application note details the PerkinElmer GC 2400™ System with Flame Ionization Detector (FID) for the analysis of residual solvents and monomers in plastics. The PerkinElmer 2400 GC System provides accuracy and sensitivity to meet the requirements for plastics analysis.

PerkinElmer®
For the Better

Instrumentation

The PerkinElmer 2400 GC System provides robust sample throughput and the sensitivity needed to meet manufacturer and regulatory requirements for plastics. The PerkinElmer SimplicityChrom™ Chromatography Data System (CDS) Software and the detachable touchscreen allow for intuitive, high-throughput laboratory workflows and the real-time monitoring of crucial data from any location.

Experimental

The consumable materials, hardware, and software used in the method are detailed in the following sections.

Materials and Reagents

The consumable materials and reagents used are listed in Table 1 along with the PerkinElmer part numbers.

Table 1: Consumables used.

Consumable	PerkinElmer Part No.
2 ml PureView™ Autosampler Vials	N9305088
Autosampler Vial Caps	N9306362
22 mL Clear Glass Crimp Top Vial	B0104236
Aluminum Cap with PTFE/Silicone Septa and Spring Kit	B0104241
20 mm Hand Crimper	N9302785
15:85 Graphite/Vespel Ferrules, 0.4 mm I.D.	09920104
Stainless Steel Oxygen/ Moisture/ Hydrocarbon Trap Kit	N9306110
Stainless Steel Moisture/Hydrocarbon Trap Kit (Air)	N9306117
Advanced Green Injection Port Septum	N9306218
Orange, Capillary Splitless Deactivated Glass Liners with Deactivated Wool	N9306236
5 µL Autosampler Syringe	N6402186

Hardware and Software

The GC 2400 System with FID Detector was used for the analysis of residual solvents and monomers in plastics. A PerkinElmer Elite-5 column was conditioned according to the recommended protocol in the PerkinElmer Capillary Column Quick Care Guide. Instrument control and data analysis were completed with the SimplicityChrom CDS Software.

Methods

The instrument parameters, calibration standards, and blanks used in the method are described in the following sections.

Instrument Conditions

The consumable materials and reagents used are listed in Table 1 along with the PerkinElmer part numbers.

Table 2: Chromatography Conditions.

GC Parameters			
Instrument	GC 2400 System		
Column	PerkinElmer Elite-5; 30 m x 0.25 mm x 0.25 µm (part no. N9316076)		
GC oven parameters	Initial	Ramp 1	Ramp 2
	75°C (2 min)	10° C/min to 120°C	50° C/min to 240°C
Injector	Capillary Split/splitless (CAP) with autosampler		
Detector	Flame Ionization Detector (FID)		
Injector Parameters			
Carrier/mode	Hydrogen, 35 cm/s		
Temperature	275° C		
Injection volume	2.0 µL		
Split flow	25 mL/min		
Septum purge	3 mL/min		
Detection Mode Time Managed MRM			
Type	FID		
Temperature	300° C		
Hydrogen	30 mL/min		
Air	400 mL/min		

Figure 1: The PerkinElmer GC 2400 System.

Calibration Standards

The consumable materials and reagents used are listed in Table 1 along with the PerkinElmer part numbers.

Six initial calibration (ICAL) standards and one calibration check standard (Cal Check Std) were prepared using the following solvents and monomers:

- 1,2-Dichlorobenzene (1,2-DCB)
- Methyl methacrylate (MMA)
- Toluene
- Styrene
- n-Octane as Internal Standard (IS)

The standards were prepared in 22 mL crimp vials according to the parameters listed in Table 3. The standards were prepared volumetrically to a final volume of 10 mL due to volatility of the compounds; they were prepared just before the analysis using a gas-tight syringe to fill the clear GC vial.

Table 3: Calibration Standards.

Standard	1,2-DCB	MMA	Toluene	Styrene	n-Octane (IS)
ICAL STD #1	9972 µL	1.0 µL	1.0 µL	1.0 µL	25 µL
ICAL STD #2	9967.5 µL	2.5 µL	2.5 µL	2.5 µL	25 µL
ICAL STD #3	9960 µL	5.0 µL	5.0 µL	5.0 µL	25 µL
ICAL STD #4	9945 µL	10 µL	10 µL	10 µL	25 µL
Cal Check Std	9945 µL	10 µL	10 µL	10 µL	25 µL
ICAL STD #5	9900 µL	25 µL	25 µL	25 µL	25 µL
ICAL STD #6	9825 µL	50 µL	50 µL	50 µL	25 µL

Sample and Blank Preparation

Plastic samples were prepared as follows:

- Approximately 0.8 g of plastic was dissolved in 10 mL of 1,2-DCB.
- The plastic was allowed to fully dissolve over 8-12 hours.
- 25 µL of n-Octane was added as an internal standard and vortexed for 5-10 seconds to ensure the plastic was fully dissolved.
- Using a gas-tight syringe, the sample was removed from the prep vial and transferred into the GC vial with no headspace.

A blank was prepared using only 1,2-DCB to confirm that the sample preparation procedure and GC system were free of interferences.

The standards, samples, and blank were then analyzed on the GC-FID using the SimplicityChrom software. The final concentration of each analyte was calculated by the software's Processing Method.

Results and Discussion

The solvents and monomers of interest—MMA, Toluene, and Styrene—were detected and quantitated as listed in Table 4.

The sample preparation and GC-FID methods used here produced linear calibrations with r^2 values of 0.996 or greater. The calibration check standard verified the calibration and demonstrated that the method blank was free of all interferences. The calibration curves for MMA, Toluene, and Styrene are presented in Figures 2, 3, and 4, respectively.

The retention times and quantitation results for the sample are detailed in Table 5. The chromatograms for the sample and the Cal Check Standard are shown in Figures 5 and 6.

Table 4: Calibration Standards Quantitation Results.

Standard	MMA	Toluene	n-Octane (IS)	Styrene
ICAL STD #1	187 µg/mL	173 µg/mL	1758 µg/mL	182 µg/mL
ICAL STD #2	468 µg/mL	433 µg/mL	1758 µg/mL	455 µg/mL
ICAL STD #3	936 µg/mL	865 µg/mL	1758 µg/mL	909 µg/mL
Cal Check Std	936 µg/mL	865 µg/mL	1758 µg/mL	909 µg/mL
ICAL STD #4	1872 µg/mL	1730 µg/mL	1758 µg/mL	1818 µg/mL
ICAL STD #5	4680 µg/mL	4325 µg/mL	1758 µg/mL	4545 µg/mL
ICAL STD #6	9360 µg/mL	8650 µg/mL	1758 µg/mL	9090 µg/mL

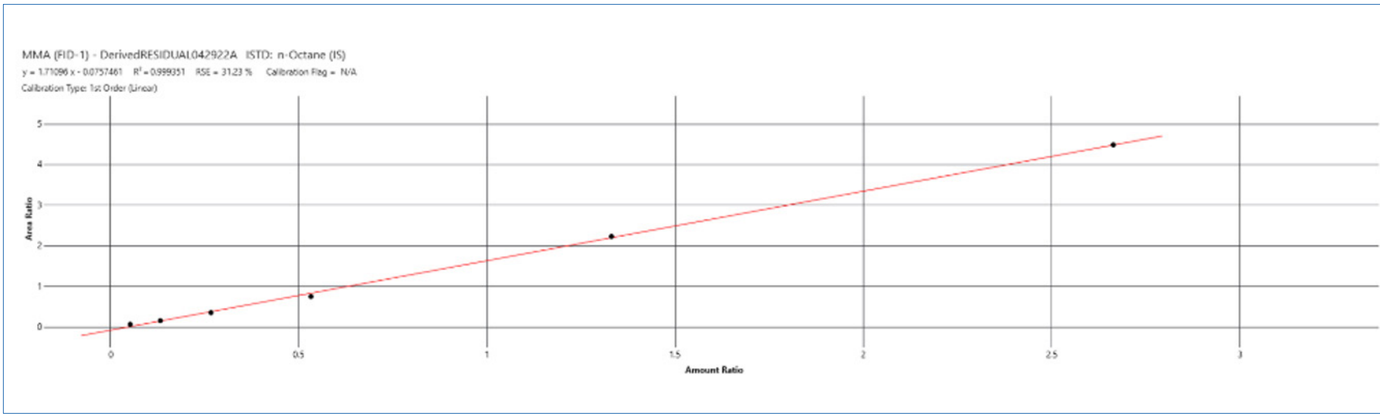

Figure 2: MMA Calibration Curve.

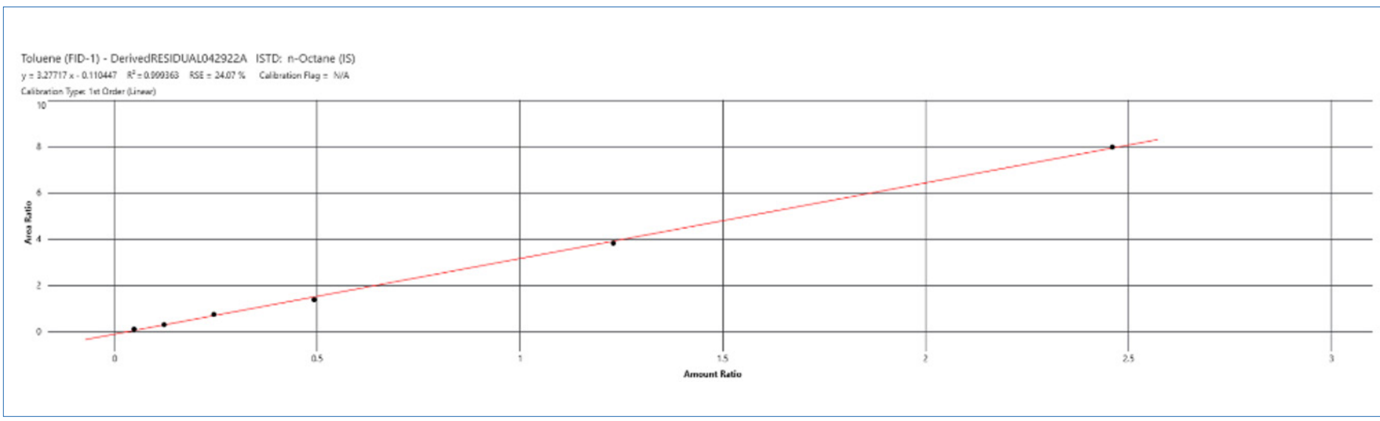

Figure 3: Toluene Calibration Curve.

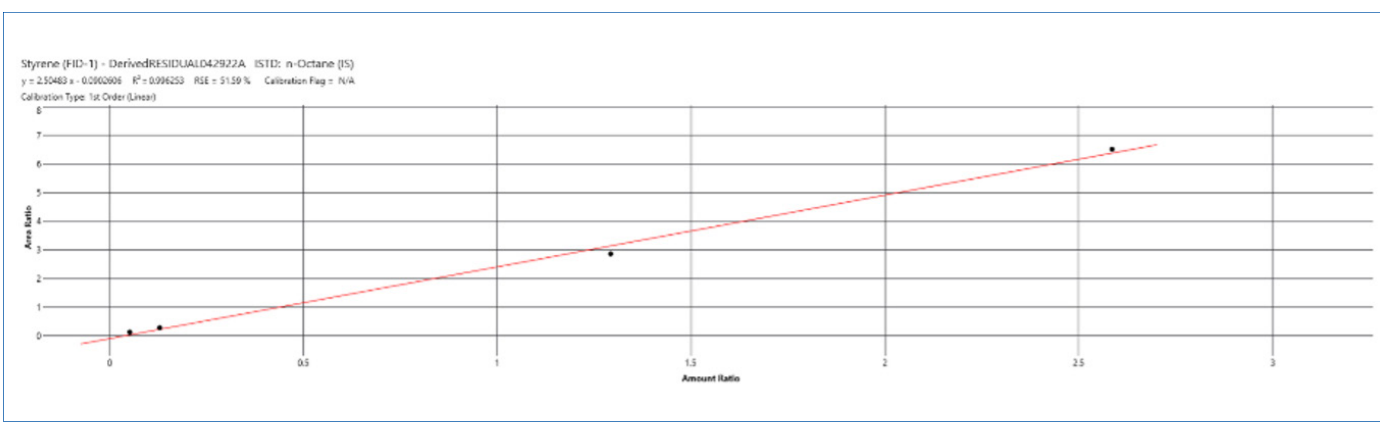

Figure 4: Styrene Calibration Curve.

Table 5: Sample Results.

Analyte	Retention Time (min)	MMA µg/ml	Toluene µg/ml
MMA	2.146	227.9883	485.0814
Toluene	2.509	399.9139	850.8807
Styrene	3.680	149.1866	317.4183

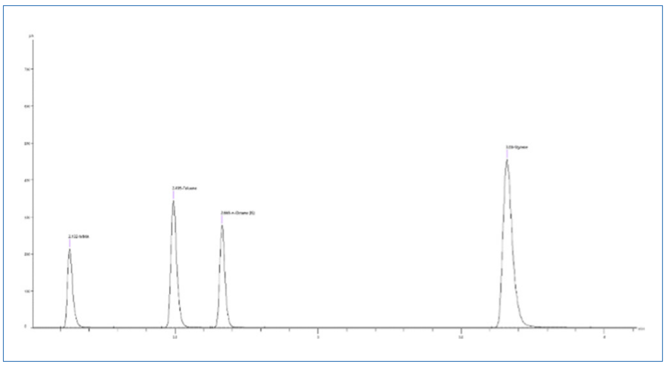

Figure 5: Cal Check Std Chromatogram.

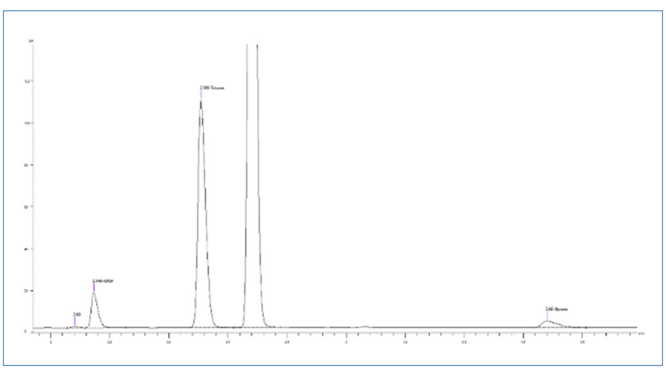

Figure 6: Sample Chromatogram.

Conclusion

Accurate and sensitive analysis of monomers and solvents used in plastic products was successfully completed using the GC 2400 System with FID Detector. As long as plastics are manufactured, there will be a need for robust methods and instrumentation such as those described here to deliver the quality control data required by plastic manufacturers.

The GC 2400 provides reliable and reproducible runs for high throughput laboratories. Data acquisition and analysis was performed with the SimplicityChrom CDS Software, which provides a practical, customizable user experience with multifunctionality and accessibility options. In addition, the detachable touchscreen provides versatility and portability which optimizes time and ultimately lab productivity..

PerkinElmer, Inc.
940 Winter Street
Waltham, MA 02451 USA
P: (800) 762-4000 or
(+1) 203-925-4602
www.perkinelmer.com

783192 PKI