



## 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.

## 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 |

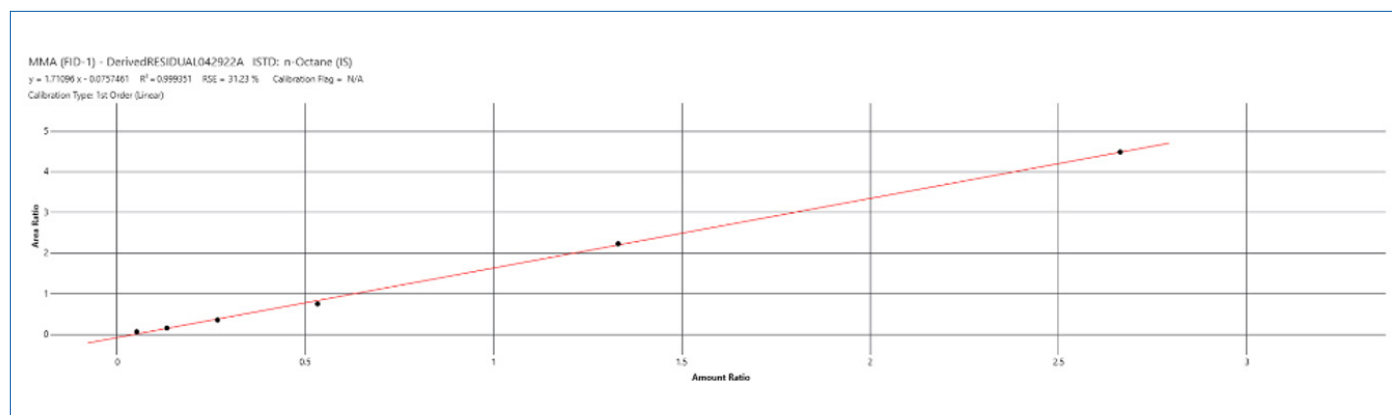


Figure 2: MMA Calibration Curve.

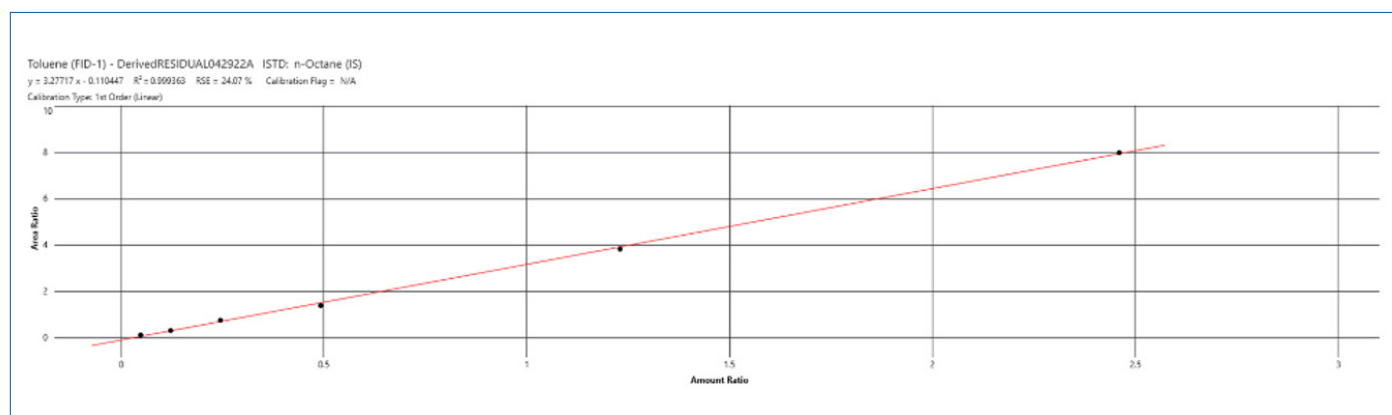


Figure 3: Toluene Calibration Curve.

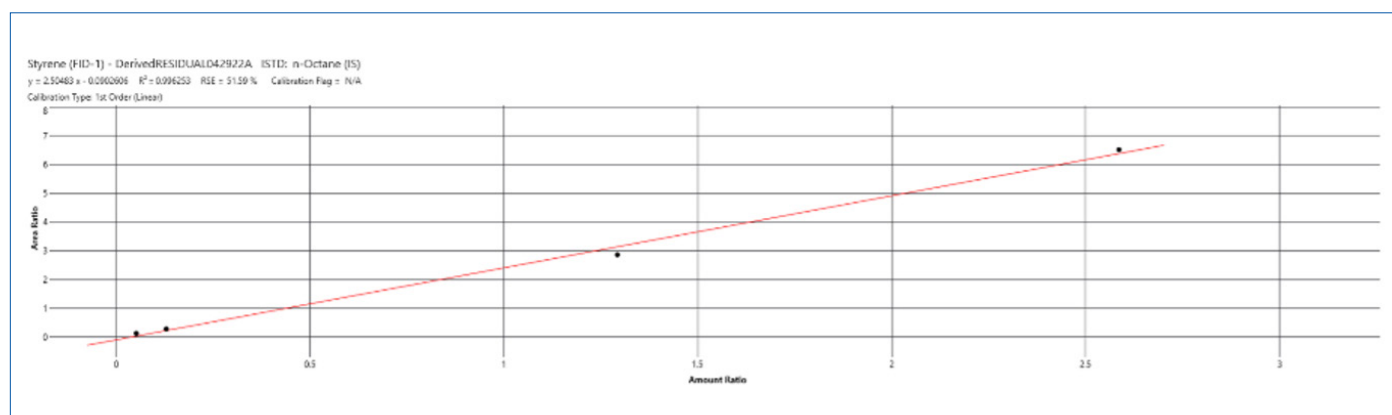


Figure 4: Styrene Calibration Curve.

Table 5: Sample Results.

| Analyte | Retention Time (min) | MMA $\mu\text{g/ml}$ | Toluene $\mu\text{g/ml}$ |
|---------|----------------------|----------------------|--------------------------|
| MMA     | 2.146                | 227.9883             | 485.0814                 |
| Toluene | 2.509                | 399.9139             | 850.8807                 |
| Styrene | 3.680                | 149.1866             | 317.4183                 |

## 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..

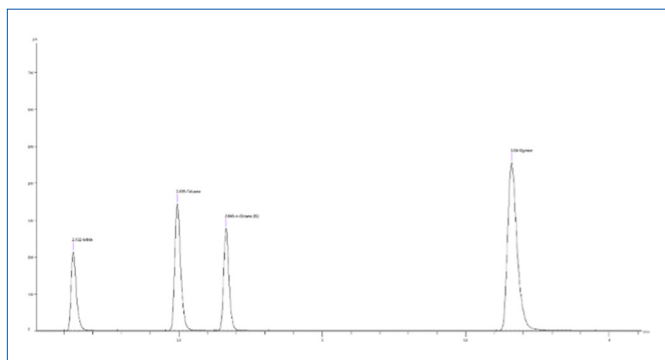


Figure 5: Cal Check Std Chromatogram.

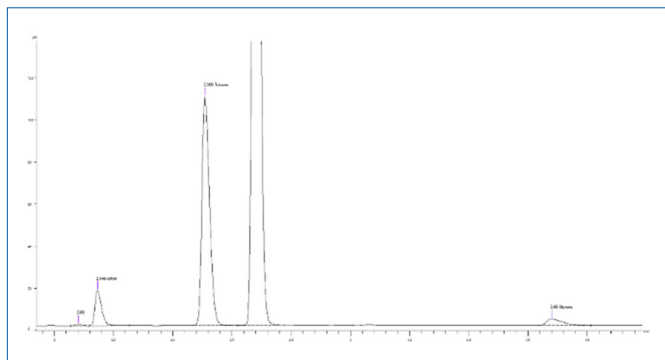


Figure 6: Sample Chromatogram.