# Acetaldehyde Residue Detection by Agilent 7820 GC with FID in Polyethylene Terephthalate Bottles

Application Note


## Authors

Jiaomei Yang [Agilent intern]
Suli Zhao
Agilent Technologies
Shanghai 200131
China



## Abstract

A method was developed for the analysis of acetaldehyde (AA) residue in polyethylene terephthalate (PET) bottles using an Agilent 7820 GC with flame ionization detection (FID). This method achieved a low detection limit with a linear range of 0.5–5.0 µg and an $R^2$ of 0.9993. AA residue in the range of 0.03 µg/g was identified in pure water bottle samples by this method.


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## Introduction

AA is known to be present in PET bottles, and can leach into the bottle contents. This can alter the taste of the beverage, especially if it is carbonated. EU Council Directive 98/83/EC regulated the AA content in water intended for human consumption. The European Union Scientific Committee for Food (EU SCF) has set a limit of AA residue in food to 6.8 µg/g.

The AA content of PET is generally determined using headspace gas chromatography. Since AA detection depends upon temperature and time, consistent instrument conditions must be defined for lab-to-lab comparisons. This application note describes the development of a method for determining AA content in PET water bottles using headspace gas chromatography with an Agilent 7820 GC with FID detection. According to the method, the bottle is ground into powder under liquid nitrogen, then heated prior to injection into the GC.

## Experimental

### Reagents and chemicals

Acetaldehyde, pure grade, and methanol, HPLC grade were purchased from J&K.

### Equipment and materials

This method was developed with an Agilent 7820 GC and an Agilent 7697A Headspace Sampler (p/n G4556) with an FID detector. The carrier gas was controlled by a 7820 EPC system using a split/splitless inlet. The 7697A transfer line was installed through an inlet septum. The 7820A GC liner was a direct 2 mm id (p/n 5181-8818). The 7697A Headspace Sampler used 20-mL vials (p/n 5190-2288), a 20-mm crimper (p/n 5040-4669), and a headspace Al crimp cap (PTFE/Si sep, 20 mm, p/n 5183-4477).

### Sample preparation

PET bottles were cut into 6-mm pieces. The PET pieces were kept under liquid nitrogen and ground into a powder. The powder was weighed into 20-mL headspace sample vials for injection onto the GC.

*Table 1. Instrument Conditions*

| **Headspace sampler conditions** | |
|---|---|
| Instrument | Agilent 7697A Headspace Sampler |
| Oven temperature | 90 °C |
| Loop | 100 °C |
| Transfer line | 110 °C |
| Vial equilibration | 30 minutes |
| Injection time | 0.5 minutes |
| GC cycle | 46 minutes, 20 mL |
| Fill mode | default |
| Fill pressure | He, 15 psi |
| Extraction mode | single extraction |
| **GC conditions** | |
| Instrument | Agilent 7820 GC |
| Inlet | 200 °C; split: 10:1 |
| Carrier gas | He, flow mode: 1.5 mL/min |
| Injection volume | 1 mL from 1 mL headspace loop |
| Column | CP-PoraBOND Q FUSED SILICA (25 m × 0.25 mm L.D. df = 3 µm) |
| Oven temperature | Hold at 60 °C for 4 minutes, Then 60 °C to 100 °C at 5 °C/min, hold for 10 minutes, Then 100 °C to 250 °C at 50 °C/min, hold for 10 minutes |
| FID | 250 °C ; $H_2$: 30 mL/min; make up + constant flow: He, 25 mL/min; air: 400 mL/min |
| FID signal | 20 HZ |

## Results and Discussion

### Calibration

Calibration was performed with a 1.0 mg/mL solution of AA dissolved in pure water. Solution in the amounts of 0.1, 1, 10, 20, 35, and 50 µL were injected into six separate 20-mL headspace vials. The calibration curve was constructed with these six calibration standards, resulting in a correlation coefficient ($R^2$) of 0.9998. The calibration curve is shown in Figure 1.

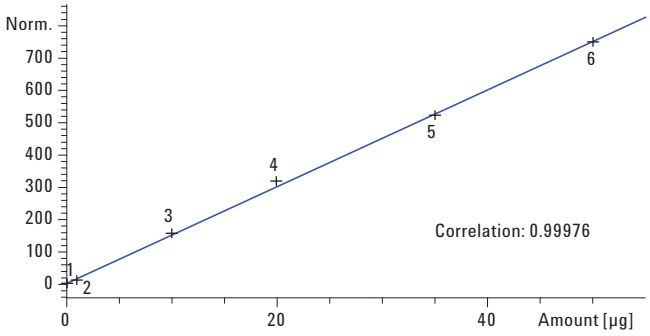


*Figure 1. Calibration curve of AA.*

Figure 2 shows the resulting chromatogram of the calibration run with a 20-µL injection of a 1.0 mg/mL AA solution. As the figure illustrates, the AA retention time was 9.45 minutes.

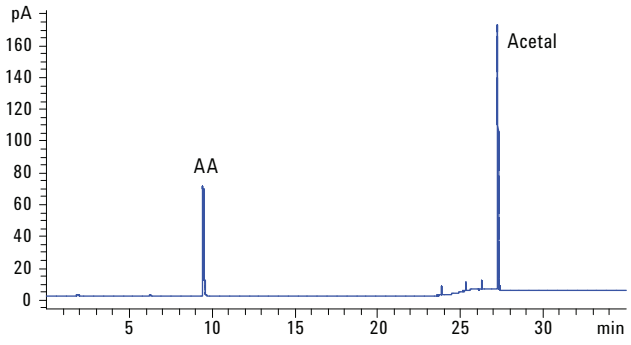


*Figure 2. 1.0 mg/mL calibration solution chromatogram.*

### Repeatability

Seven 20 µL injections of the 1.0 mg/mL AA solution determined the repeatability of the system for AA detection. Table 2 shows retention time and area RSD% of the seven injections. Figure 3 shows an overlay of the seven chromatograms. As the figure illustrates, repeatability of the method is excellent.

*Table 2. RSD% of AA*

| Name (n = 7) | RSD (%) | |
|---|---|---|
| AA | R.T | Area |
| | 0.01 | 2.05 |

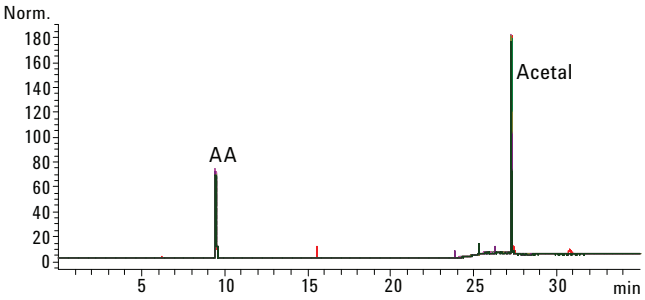


*Figure 3. Overlay of seven chromatograms.*

### Real sample

The method was verified by analyzing 0.4 g of ground sample powder under the same experimental conditions. Figure 4 shows an overlay of the chromotograms produced from analysis of the real sample (blue trace), and the real sample spiked with 1 uL of standard (red trace). As the figure illustrates, both samples had the same retention time. The AA content in the sample was detected at 0.3 µg/g, which is lower than EU SCF regulation limits.

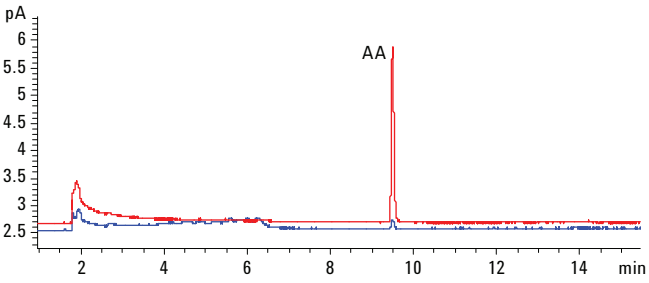


*Figure 4. Contrast chromatograms of real sample (blue) and standard added sample (red).*

## Conclusion

A method was developed for the detection of AA in PET bottles using an Agilent 7820 GC system with FID detection, and an Agilent 7697 Headspace Sampler. This method has excellent linearity and repeatability, and can reach the low detection limits set forth by the EU SCF for the regulation of AA in food. Therefore, it has been determined that this is a reliable method for the analysis of AA in PET.

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