Determination of Formic Acid in Acetic Acid for Industrial Use by Agilent 7820A GC

Application Brief

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With rising prices of crude oil and a future shortage of oil and gas resources, people are relying on the development of the coal chemical industry.

Acetic acid is an important intermediate in coal chemical synthesis. It is used in the production of polyethylene, cellulose acetate, and polyvinyl, as well as synthetic fibres and fabrics. The production of acetic acid will remain high over the next three years. In China, it is estimated that the production capacity of alcohol-to-acetic acid would be 730,000 tons per year in 2010.

The purity of acetic acid determinates the quality of the final synthetic products. Formic acid is one of the main impurities in acetic acid. Many analytical methods for the analysis of formic acid in acetic acid have been developed using gas chromatography. For example, in the GB/T 1628.5-2000 method, packed column and manual sample injection is used with poor separation and repeatability which impacts the quantification of formic acid.

In this application brief, a new analytical method was developed on a new Agilent GC platform, the Agilent 7820A GC System. The GC was configured with a micro thermal conductivity detector (μTCD) which provides an easy to use method for the determination of formic acid in acetic acid. To achieve a better separation, an Agilent J&W DB-FFAP (30 m × 320 μm, 0.25 μm) capillary column was used as the analytical column.

Highlights

- The Agilent 7820A GC coupled with a μTCD provides a simple method for analysis of formic acid in acetic acid.
- ALS and EPC ensure good repeatabiltiy and ease of use which makes the 7820GC appropriate for routine analysis in QA/QC labs.
- Using a capillary column as the analytical column ensures better separation of formic acid in acetic acid compared to the China GB method.

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Experimental

Analytical conditions

Inlet	150 °C, Split ratio: 10:1
Injection volume	1 µL
Column	Agilent J&W DB-FFAP, 30 m × 320 µm, 0.25 µm (p/n 123-3232)
Carrier gas	He, Constant flow: 1.5 mL/min
Oven	80 °C (3 min) 8 °C/min 150 °C (5 min)
Detector	µTCD: 200 °C, Reference gas: 15 mL/min, Makeup gas: 6.5 mL/min FID: 300 °C, H_2: 30 mL/min; Air: 350 mL/min; Makeup flow (N_2): 60 mL/min
Autosampler	Agilent 7693A automatic liquid sampler
Data analysis system	EZChrom Elite Compact

Results

Figure 1 shows the chromatogram of the analysis of formic acid in acetic acid at 1% (weight to weight) on the Agilent 7820A GC. From the chromatogram it could be seen that formic acid elutes after acetic acid on the Agilent J&W DB-FFAP column, which is confirmed by the Agilent 6890 GC and the Agilent 5975C series GC/MSD. In this experiment, a flame ionization detector (FID) was also used to confirm the detection of formic acid. The results are shown in Figure 2.

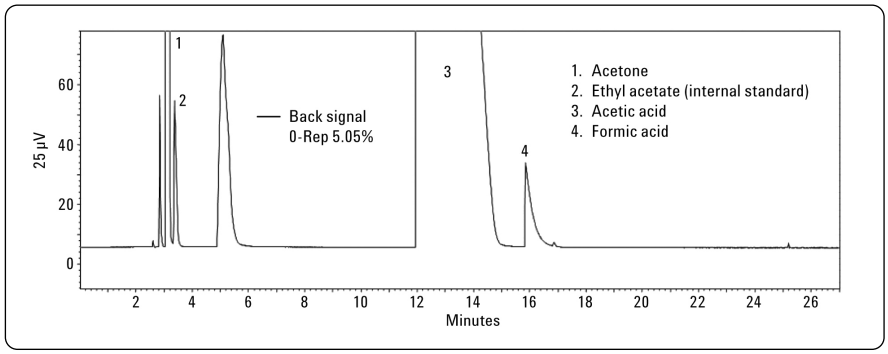

Figure 1. Chromatogram of formic acid analysis in acetic acid on the TCD channel.

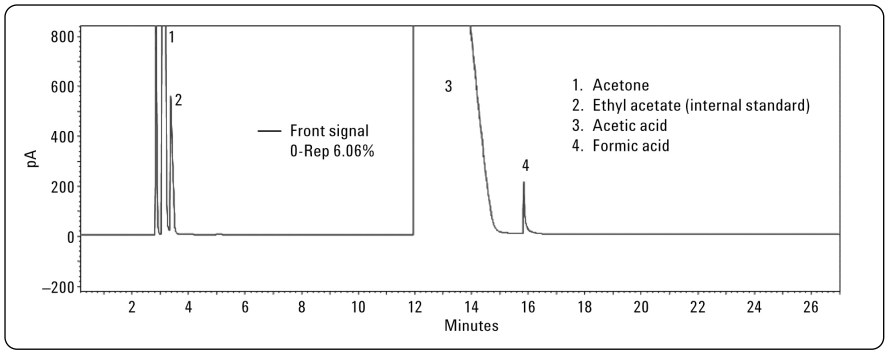

Figure 2. Chromatogram of formic acid analysis in acetic acid on the FID channel.

Agilent μTCD is a proprietary designed single-filament flow switching detector. This design eliminates the need for a reference column, by exposing the filament to column effluent and reference flows at a frequency of 5 Hz. There is no other reference column that assures a stable baseline even with a ramped temperature program. Compared to the typical TCD, the smaller volume cell of μTCD provides higher sensitivity.

The detection limits of formic acid on μTCD was tested using a series of diluted formic acid samples. The method has a signal-to-noise of 6.8 for a formic acid concentration of 0.05 wt%. A method precision of 1.75% RSD for five injections was also calculated for the 0.05 wt% concentration. These excellent results were attributed to the Electronic Pressure Control (EPC) and precision auto sampler that are the key features of the Agilent 7820A GC.

In this method, ethyl acetate was used as the internal standard according to the GB/T 1628.5-2000. Concentrations of 0.1%, 0.5%, 1%, 10% (weight to weight) formic acid in acetic acid standard solution were made with ethyl acetate as an internal standard. The results show that from 0.1% to 10% (weight to weight), formic acid response to concentration was linear with an $r^2 = 0.9917$.

Conclusions

The Agilent 7820A GC coupled with a μTCD provides a simple method for analysis of formic acid in acetic acid. Use of a capillary column ensures good separation of impurities and acetic acid in both high concentrations and low concentrations. The stable and sensitive μTCD is a good choice for formic acid analysis compared to an FID which has a relatively low response. The 7693A autosampler with a capacity of 16 sample vials and 7820A GC EPC control ensure good repeatability and ease of operation, which is suitable for the fast growing coal to chemical industry and routine analysis labs where feedstock and intermediate quality control is important. The data processing system, EZChrom Elite Compact software specially designed for Agilent 7820 GC, is easy to use and provides commonly used report templates.

References

1. GB/T 1628.5-2000. Determination of formic acid in acetic acid by gas chromatography.

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