Application Brief

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Endrin and DDT Stability Study for Drinking Water Method EPA 525.2 on the Intuvo

Technology advantage: Agilent Intuvo 9000 GC with MSD

Introduction

The organochlorine pesticides endrin and 4,4'-DDT are commonly used to determine flowpath inertness and cleanliness in gas chromatography (GC). Exposed active sites, residual matrix, or septum debris with high temperature can cause the decomposition of 4,4'-DDT to 4,4'-DDD and 4,4'-DDE, and the isomerization of endrin to endrin ketone and endrin aldehyde[1,2,3]. While DDT is thermally stable at temperatures typically used for environmental analysis by GC, degradation requires the presence of an active surface (for example, matrix or debris) to catalyze the dechlorination reactions[4]. However, endrin isomerization may occur at high temperatures in the absence of a catalyst or debris[5,6]. Therefore, take care in setting the appropriate Guard Chip and bus temperatures when analyzing for endrin with the Agilent Intuvo 9000 GC.

Due to the lability of endrin and 4,4'-DDT, several United States Environmental Protection Agency (US EPA) methods specify their use to verify system inertness preceding quantitative analysis. For example, US EPA Method 525.2 for the determination of organic compounds in drinking water requires a degradation limit no greater than 20 % for each compound. If the limit is exceeded, the system is deemed unsuitable for analysis, and corrective maintenance is required[7].

This application brief demonstrates that the Intuvo 9000 GC can meet the instrument performance check criteria established by US EPA Method 525.2.

Experimental

Instrumentation

- Agilent Intuvo 9000 GC
- Agilent 5977 MSD with an Inert EI
- Agilent DB-UI 8270D
  30 m × 0.25 mm, 0.25 m column
  (122-9732-INT)
- Agilent Ultra Inert double taper splitless liner (5190-4007)

Sample preparation

The instrument performance check (IPC) solution was prepared by diluting a standard solution containing DFTPP, 4,4'-DDT, and endrin (GCM-160A, ULTRA Scientific) to a final concentration of 5 ng/µL in methylene chloride.

Results and discussion

A repetitive series of injections were made consisting of 3–5 injections of the IPC, followed by 10 blank ethyl acetate injections, and ending with 3–5 injections of the IPC. The series was repeated until 310 blank injections were made, for a total of 404 injections. For each injection of the IPC solution, the percent degradation was calculated for 4,4'-DDT and endrin as specified in Method 525.2.

Figure 1 shows the average percent degradation per injection number with error bars representing one standard deviation. The calculated degradation for each measurement was well below the 20 % limit for both probes. For all measurements, the average percent degradation was 0.91 and 3.71 % for 4,4-DDT and endrin, respectively. The key to achieving these results is temperature programming the Guard Chip to match the column temperature (or using track oven mode), and keeping the bus temperature setting between 245–270 °C.

Instrument conditions

Parameter	Value
Injection volume	1 µL
Inlet	Split/Splitless 250 °C Pulsed splitless 30 psi until 0.5 minutes Purge 50 mL/min at 0.5 minutes Septum purge switched flow mode 3 mL/min
Guard chip	40 °C for 1 minute, 25 °C/min to 160 °C 3 minutes, 6 °C/min to 260 °C
Column temperature	40 °C for 1 minute, 25 °C/min to 160 °C 3 minutes, 6 °C/min to 260 °C
Bus temperature	260 °C (default)
Flow	1.2 mL/min constant flow
Transfer line temperature	260 °C
Drawout plate	6 mm (option)
Ion source temperature	260 °C
Quadrupole temperature	180 °C

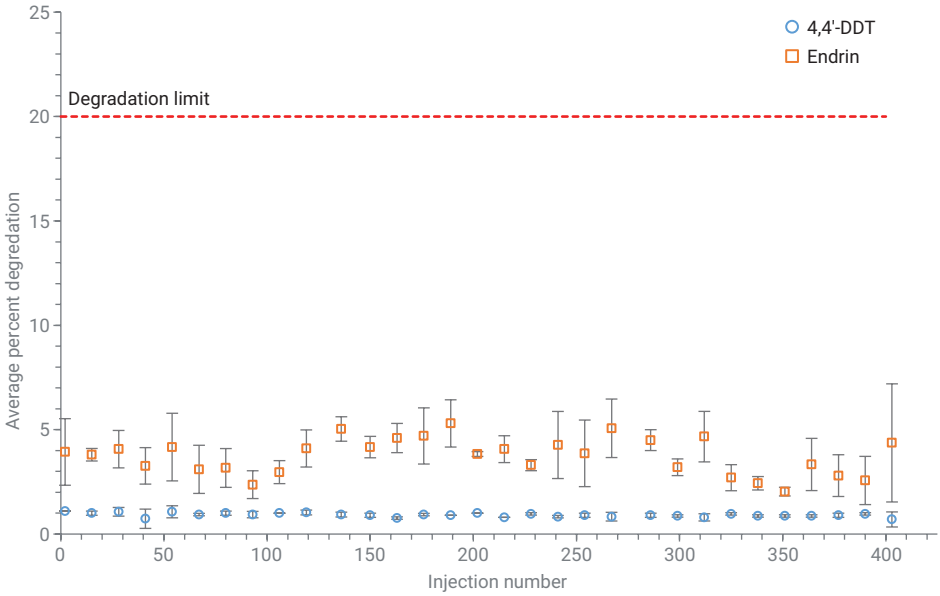

Figure 1. Degradation measurements for 4,4'-DDT and endrin.

Figure 2 shows a comparison of the first and last performance check solution injections. There is little difference between the two chromatograms. The small peak at 18.5 minutes in the last chromatogram was tentatively identified as an oxidation product of DFTPP. Oxidation of DFTPP likely occurred in the vial due to the prolonged exposure to light and air at ambient temperature while queued on the autosampler.

In addition to measuring system inertness, tuning stability was assessed based on the ion ratio criteria established in Method 525.2. For each injection of the performance check solution, the DFTPP tuning criteria was achieved.

Conclusions

The Intuvo 9000 GC demonstrates outstanding flowpath inertness from inlet to detector as measured using 4,4'-DDT and endrin. The system easily achieves the system inertness criteria specified in US EPA Method 525.2 for the analysis of organics in drinking water.

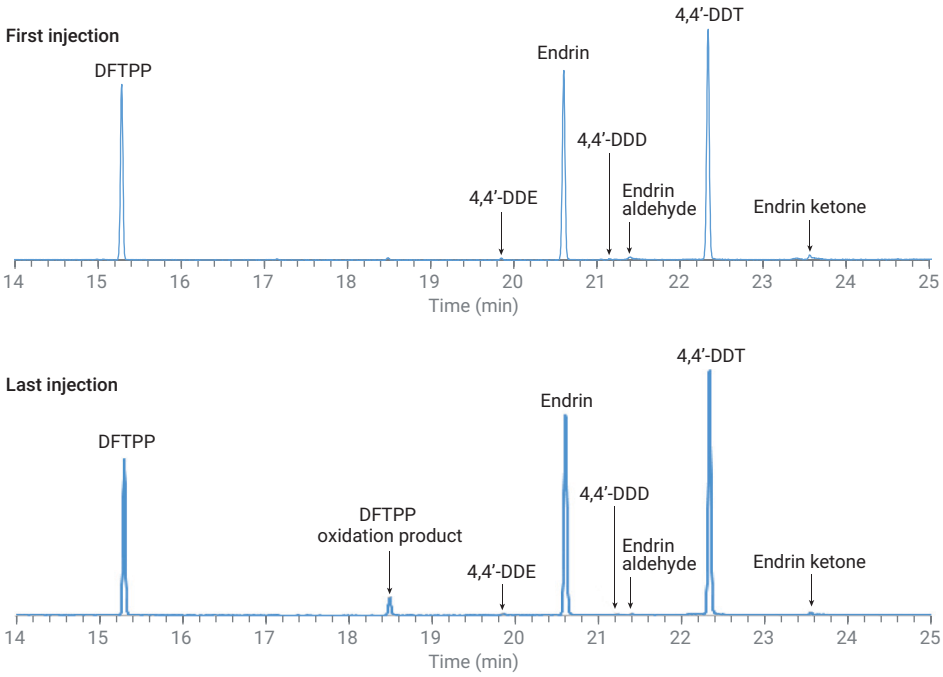

Figure 2. Chromatograms from first and last injection of the performance check solution.

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