Sub 1 mg/kg (1 ppm) Detection of Carbon Disulfide with the Polyarc System

Application Note

Low-Level Detection

Author

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Abstract

The analysis of low levels of carbon disulfide (CS_2) is important in the pharmaceutical, food, environmental, and other industries, but there exist few simple, low-cost, methods for analysis because the ubiquitous flame ionization detector (FID) is insensitive to CS_2. Here, the Polyarc System (i.e., Polyarc/FID) is shown to be a low-cost solution for low-level analyses of carbon disulfide down to a minimum detection limit of 0.14 mg/kg (0.14 ppm).

Introduction

Carbon disulfide (CS_2) is a toxin known for its neurotoxicity, damage to organs through repeated exposure, and its reproductive toxicity. Therefore, it is typically analyzed at low-levels when there is suspected contamination in the environment, workplace, pharmaceuticals, or other products. Typical GC/FID methods are unable to analyze CS_2 because it lacks C-H bonds, which are necessary for production of a signal in a flame ionization detector (FID). Because of this, researchers must resort to using a different detector such as a mass spectrometer (MS) to analyze carbon disulfide, which is more expensive and requires a second detector. In this application note, the analysis of trace levels of carbon disulfide with the Polyarc System is demonstrated.

The Polyarc system converts all organic molecules to methane (CH_4) through a two-step reaction, allowing the FID to "see" molecules that were previously invisible to the FID such as CS_2. The two-step reaction that occurs in the Polyarc for CS_2 is as follows:

$$CS_2 \xrightarrow[\text{Oxidation}]{3\cdot O_2} CO_2 + 2\cdot SO_2 \xrightarrow[\text{Methanation}]{10\cdot H_2} CH_4 + 2\cdot H_2S + 6\cdot H_2O$$

where every mole of CS_2 is converted to one mole of methane and non-carbonaceous byproducts. The FID has a very high sensitivity to methane (~1 picogram of carbon per second) and thus the Polyarc/FID combination is able to analyze the same low levels of CS_2 down to 1 pg C/s.

Figure 1. Polyarc system installed in the back detector position next to an FID on an Agilent 7890 GC.

Experimental

An Agilent 7890A GC equipped with a split/splitless inlet (Agilent G3454-64000), capillary-optimized FID, mass spectrometer (Agilent 5973), and Polyarc® reactor (ARC PA-RRC-A02) were used for the analysis.

Helium (99.999%, Praxair) was used for carrier and FID makeup. Air (zero grade, Praxair) and H_2 (99.999%, Praxair) were supplied to the ARC electronic flow control module (PA-MFC-A09) and to the FID. The effluent of the GC column was connected to an Agilent 3-way CFT splitter (G3183-60500). The MS was connected to the splitter via a retention gap column (Agilent, 160-2635-5, 0.61 m, 0.1 mm ID). The inlet capillary to the Polyarc® was connected directly to the splitter according to Figure 1. The splitter was controlled by an EPC (with restrictor frit removed) set to 4 psig.

Samples were prepared for GC analysis by serial dilutions of CS_2 (Sigma Aldrich, >99%) in 1-propanol (Sigma Aldrich, 99.9%) to create a solution with 1.24 mg/kg (1 ppm) of CS_2 in 1-propanol. Extreme care should be taken when working with carbon disulfide because of its toxicity.

GC conditions

Front inlet	Split/splitless
Inlet temperature	165 °C
Inlet linter	Agilent 18740-80190
Carrier gas	He; 11 sccm constant flow
Septum purge flow	3 sccm
Oven	30 °C (hold 20 min), 250 °C post run (5 min)
Column	DB-5 UI (30 m × 0.25 mm × 1 µm film)
Syringe	10 µL
Injection volume	0.5 µL

FID conditions

Temperature	165 °C
H_2	1.5 sccm
Air	350 sccm
Makeup	20 sccm (He)

Polyarc® System conditions

Setpoint	293 °C
H_2	35 sccm
Air	2.5 sccm

Results and Discussion

A GC method was optimized for the analysis of low levels of CS_2 (see experimental information). Notably, a high column flow rate of 11 sccm He produces very sharp peaks (1 s width) which allow for very low-level detection. Three injections of 1.24 mg/kg carbon disulfide in 1-propanol were performed, and the results are tabulated below, with the chromatograms shown in Figure 2.

Injection 1

Analyte	Ret time (min)	Area
CS_2	1.24	68377
1-Propanol	2.77	$1.07 \cdot 10^{11}$

Injection 2

Analyte	Ret time (min)	Area
CS_2	1.25	69253
1-Propanol	2.81	$1.08 \cdot 10^{11}$

Injection 3

Analyte	Ret time (min)	Area
CS_2	1.24	70126
1-Propanol	2.75	$1.06 \cdot 10^{11}$

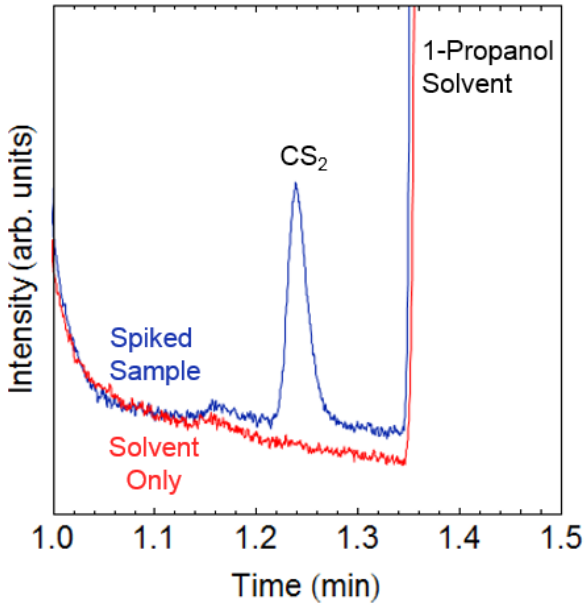

Figure 2. Polyarc/FID chromatogram for the analysis of 1.24 mg/kg CS_2 in 1-propanol (blue) and a solvent-only blank (red).

The signal-to-noise ratio, defined as two times the peak height divided by the noise, for the analysis of 1.24 mg/kg CS_2 was determined to be S/N=27 (Figure 3). Thus, the minimum detection limit (MDL) with this method is approximately 0.14 mg/kg CS_2 (assuming an MDL of 3 x the S/N).

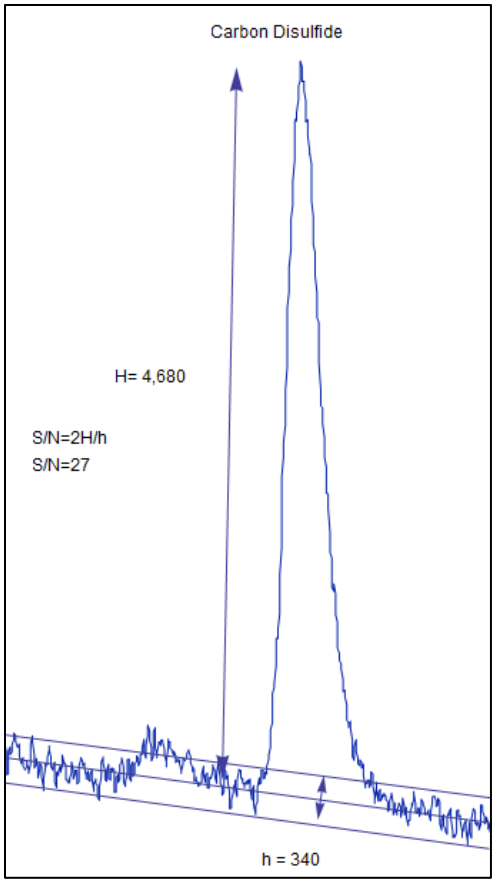

Figure 3. Signal-to-noise determination for the analysis of 1.24 ppm CS_2 in 1-propanol using the Polyarc System.

Conclusions

The Polyarc System is capable of detecting sub-ppm (1 mg/kg) levels of carbon disulfide, as a result of the catalytic conversion to methane before detection in the FID. This is in sharp contrast to traditional FIDs, for which carbon disulfide is invisible. The results shown here use 1-propanol as a solvent, but this method could be translated to other solvent systems, if necessary, with method development.

Contact Us

For more information or to purchase a Polyarc® system, please contact us at 612-787-2721 or contact@activatedresearch.com.

Please visit our website for details and additional technical literature, www.activatedresearch.com.

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