Fast On-Site Mine Safety Analysis by the Agilent 490 Micro GC

Application Note

Micro Gas Chromatography, Mine gas analysis, Mine safety

Authors

Darren Brady
Safety in Mines Testing and Research Station (SIMTARS)/Queensland Government
Goodna, Australia

Remko van Loon
Agilent Technologies, Inc.
Middelburg, the Netherlands

Abstract

Numerous mine disasters with loss of many lives continue to occur today. This fact dramatically emphasizes the importance of fast and accurate determination of the mine atmosphere for an early warning of hazards in day-to-day mine operations or after an accident has happened. This application note describes a method for fast on-site analysis of mine gases in less than 100 seconds using the Agilent 490 Micro GC equipped with four independent column channels.

Introduction

We all can recall the news bulletins reporting a mine accident and, in some cases, the many lives that are lost. Therefore, to have an early warning for multiple safety reasons, fast analysis of the mine atmosphere is extremely important for day-to-day mining activities. Moreover, a complete overview of the gases in the mine, after an accident, is essential to determine if the mine is safe for a rescue team to enter.

First, it is necessary to check for explosive gases in a mine environment. During the formation of coal beds, some gases, mainly methane and some ethane and hydrogen were trapped in the coal. When these coal beds are mined, the gases are released. Methane and other explosive gases, when mixed in certain ratios with oxygen from the air, are highly explosive. To prevent explosion hazards, it is necessary to monitor flammable gases such as methane, hydrogen, and the C2 hydrocarbons.

A second reason for mine gas analysis is that the absence of carbon monoxide and the right oxygen and carbon dioxide levels in the mine atmosphere are critical for the safety of the mine workers and rescue teams.

Third, analyzing the gases in the mine can predict spontaneous combustion or detect a fire in an early stage. Spontaneous combustion could happen when internal heat, produced by chemical reactions in the coal, is generated faster than it can be lost to the surrounding environment. Hydrogen and ethylene are formed when temperatures rise above 100 °C. The presence of low concentrations of these components gives an indication of fire or elevated temperatures in an early stage. This increases the chance of successfully dealing with the problem.

The Safety in Mines Testing and Research Station (SIMTARS), based in Queensland Australia has been providing and supporting gas monitoring systems based on gas chromatographs to the mining industry for over 20 years and offer their services, support, and training to mining companies to reduce the risks of mine explosions and help them after a mine disaster. For the three reasons given, SIMTARS is using the Agilent 490 Micro GC to provide a complete, fast, and on-site analysis of the gases collected from the underground mine.

Micro GC setup and conditions

The 490 Micro GC (p/n G3581A) used for the analysis of mine gas consists of a quad cabinet (Figure 1), and is equipped with four column independent column channels. Each column channel is a complete, miniaturized GC with electronic carrier gas control, micro-machined injector, narrow-bore analytical column, and micro-thermal conductivity detector (µTCD).

Figure 1. Agilent 490 Micro GC with quad channel cabinet housing.

The first channel installed, is equipped with a 10 meter CP-MolSieve 5Å column, running on argon as carrier gas for the analysis of helium, hydrogen, oxygen, and nitrogen. Channels 2 and 3 are identical and, like the first channel, are equipped with a 10 meter CP-MolSieve 5Å column. However, these channels have the optional backflush functionality and run on helium carrier gas, for the analysis of methane and carbon monoxide. Ethane and ethylene are analyzed on a fourth channel using a 10 meter a PoraPLOT U column. Table 1 shows the analytical conditions for all channels.

Agilent EZChrom Chromatography Data Software is used for data acquisition, and SIMTARS EZGas Professional software, specifically written for the mining industry, is used for calibration and result generating. The analysis results are exported to Segas Professional, a software package developed by SIMTARS, for additional combustibility calculations, combustion ratios and trend analysis.

Fast mine safety analysis in less than 100 seconds

The first column channel, equipped with a CP-Molsieve 5Å column, is used to analyze permanent gases, including helium, hydrogen, oxygen and nitrogen. Figure 2 shows a chromatogram where the compounds of interest are well separated.

Table 1. Analytical Conditions for Quad Channel Micro GC

	Channel 1 CP-Molsieve 5A 10 m	Channel 2 CP-Molsieve 5A 10 m	Channel 3 CP-Molsieve 5A 10 m	Channel 4 PoraPLOT U 10 m
Column temperature	80 °C	80 °C	80 °C	60 °C
Carrier gas	argon, 120 kPa	helium, 150 kPa	helium, 150 kPa	helium, 100 kPa
Injector temperature	50 °C	50 °C	50 °C	50 °C
Injection time	100 ms	110 ms	110 ms	90 ms
Back flush time	no backflush	10	10	no backflush
Detector sensitivity	auto	auto	auto	auto
Invert signal	yes	no	no	no
Sample line temperature	40 °C			
Sampling time	70 seconds			

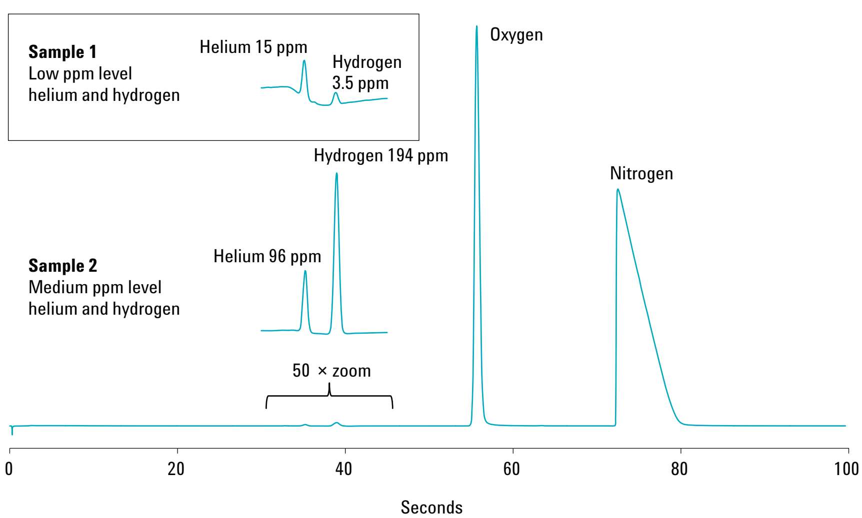

Figure 2. Chromatogram for helium, hydrogen, oxygen and nitrogen separation on the first column channel.

The molecular sieve channel is running on argon as the carrier gas, which enables the determination of low concentrations of helium and hydrogen. All other compounds will have an increased detection limit by approximately a factor of 10, compared to helium, when argon is used as a carrier gas. However, oxygen and nitrogen are present at percentage levels in the mine atmosphere, which allows the use of argon carrier gas for detection of these gases. Concentration results for hydrogen, oxygen, and nitrogen are used by SIMTARS for combustibility calculations.

Helium, naturally available in our atmosphere at low ppm concentrations, is analyzed on this channel as well. On a molecular sieve column, helium and hydrogen elute close together. Analysis of helium prevents it from being incorrectly reported as hydrogen. This can result in the erroneous conclusion that spontaneous combustion is occurring. From time to time, helium is also used as a tracer gas to determine gas movements in the underground mine.

Channel two also includes a 10 meter MolSieve 5Å, this time with helium as the carrier gas. This channel is used for the analysis of methane and carbon monoxide. Figure 3 shows a chromatogram for two different samples, one containing a medium level for carbon monoxide (~ 200 ppm) and the other with a very low level of carbon monoxide. In this chromatogram, excellent separation and analysis of methane and carbon monoxide in less than 100 seconds is obtained.

The typical limit of detection for the µTCD, specified by Agilent, is 1 ppm for early eluting components on a Wall Coated Open Tubular (WCOT) column and 10 ppm on Porous Layer Open Tubular (PLOT) and micro-packed column types. The CP-MolSieve 5Å column is a PLOT type column, however when it comes to carbon monoxide at low levels, the exact concentration is of less importance for SIMTARS than the trend. Even a slight increasing trend of the chromatogram's base line at the carbon monoxide retention time is monitored for early indications of spontaneous combustion in the mine.

This MolSieve 5Å channel is equipped with back flush functionality to ensure moisture, carbon dioxide, and the C2 hydrocarbons are backflushed to vent, to maintain the separation efficiency of the molecular sieve column. Moisture and carbon dioxide tend to adsorb quickly to the Molsieve 5Å stationary phase changing its chromatographic properties. This could result, over time, in retention shifts and loss of separation.

For SIMTARS, the analysis of methane for explosion risk reasons and carbon monoxide for combustion identification are of high importance, especially when the Micro GC is taken into the field after a mine disaster. Therefore, this column channel is duplicated to the third position of the instrument to allow optimized operation for the analysis of each, and to have a back up available at all times. When one column is reconditioned, the other column can still be used for analysis.

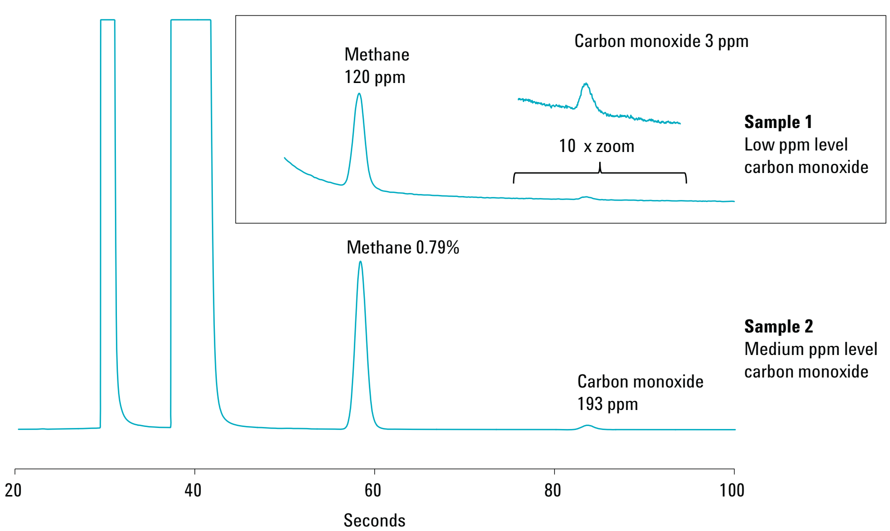

Figure 3. Chromatogram for methane and carbon monoxide on the second column channel.

The fourth channel, equipped with a 10 meter PoraPLOT U column and helium as the carrier gas, is used to analyze carbon dioxide, ethane, and ethylene. Figure 4 shows an example for baseline separation of these three components.

The right carbon dioxide level is of importance for the safety of the mine workers and rescue personnel. Moreover, the results for carbon dioxide and ethane as well, are used in the combustibility calculations by SIMTARS. Ethylene, like hydrogen, is formed when coal temperatures rise above 100 °C and, therefore, is used as an early warning for spontaneous combustion or a fire.

Excellent repeatability for quantity and retention time

Repeatability, reported as relative standard deviation, shows excellent results for both concentration and retention time as shown in Table 2. Typical values, based on quantity, are determined around 0.05% RSD for components that are present in the sample at percentage levels and between 0.1 to 0.6% for ppm level components. Retention time repeatability, for all components of interest, is calculated at 0.015% or lower.

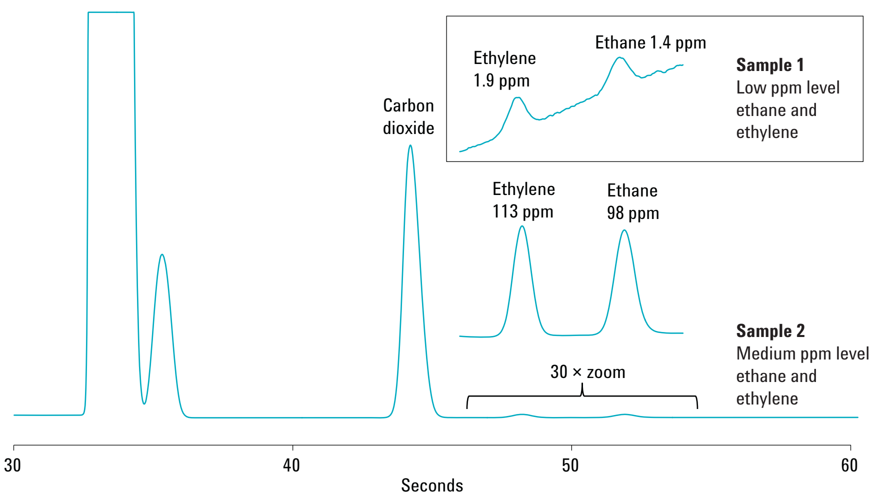

Figure 4. Chromatogram for carbon dioxide, ethane and ethylene on the fourth column channel.

Table 2. Typical Repeatability Figures (Population Size is 10) for the Agilent 490 Micro GC

Component	Column channel	Concentration average	Concentration unit	Concentration RSD (%)	Retention time average (seconds)	Retention time RSD (%)
Helium	1	102.8	ppm	0.10	35.22	0.015
Hydrogen	1	118.5	ppm	0.11	38.79	0.014
Oxygen	1	20.4	%	0.044	54.65	0.0088
Nitrogen	1	72.4	%	0.056	70.00	0.011
Methane	2 (and 3)	1.85	%	0.054	54.09	0.0087
Carbon monoxide	2 (and 3)	181.9	ppm	0.25	71.35	0.012
Carbon dioxide	4	1.91	%	0.040	43.92	0.014
Ethylene	4	110.8	ppm	0.61	48.01	0.013
Ethane	4	92.3	ppm	0.25	51.62	0.013

Conclusion

This application note clearly show that the 490 Micro GC is a powerful tool for accurate mine safety analysis.

The major reason for SIMTARS using the 490 Micro GC is that it provides a complete, fast and on-site analysis of the mine gases collected from underground. Moreover, the 490 Micro GC detects compounds that are not covered by the mine's continuous monitoring system.

The 490 Micro GC analyzes mine environment samples in less than 100 seconds resulting in multiple results per hour for accurate trend analysis and thus better informed decision making for the prevention of mine disasters.

In addition, the 490 Micro GC gives SIMTARS rapid and reliable results to determine, after a mine disaster, the status of the underground environment before deciding to send in a rescue teams.

For More Information

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