Sensor Gas Chromatograph

Ammonia/Trimethylamine Analyzer
Model: ODNA-P2

Technical Information

FiS

FiS Inc.
Japan
October, 2011

Contents

1. Sensor Gas Chromatograph (SGC)   ODNA-P2

1. Measurement principle

ODNA-P2 is a gas chromatograph using a semiconductor gas sensor as a detector. Ammonia/trimethylamine is separated from a gas mixture through chromatography, and measured with a semiconductor gas sensor showing high sensitivity to ammonia/trimethylamine.
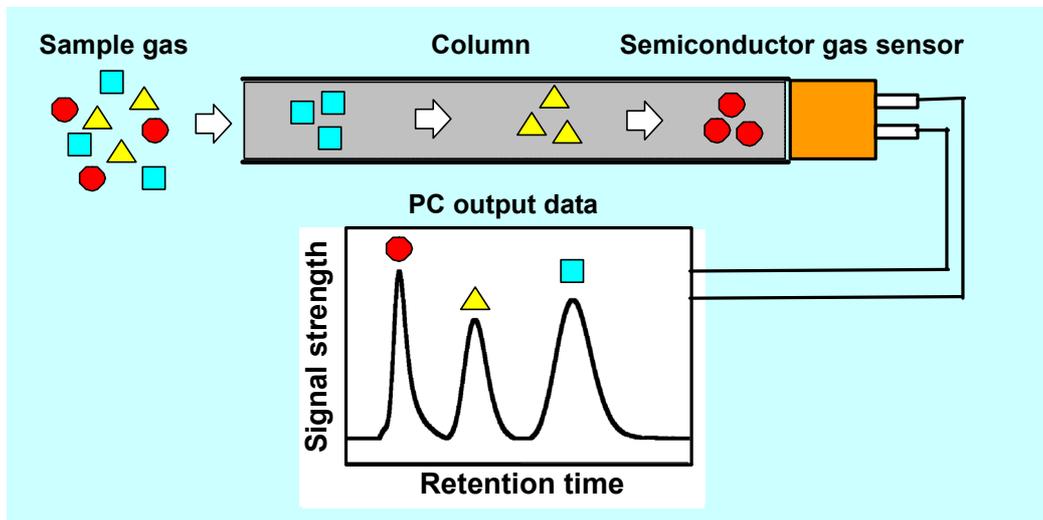

Fig. 1 Measurement principle

2. Features

- Ammonia can be measured from 50ppb to 100ppm, trimethylamine from 50ppb to 10ppm.
- Injecting the sampling gas with a syringe will automatically start the measurement and the measurement will be completed in 4 or 8 minutes.
- Automatic and continuous sample gas injector is optionally available.
- The following measurement is ready in one minute from the previous measurement completion.
- No high-pressure gas cylinder is needed since ambient clean air is used as carrier gas.
- In case you are concerned about very small amounts of gas in ambient air, the SGC can be optionally modified to connect to a cylinder.
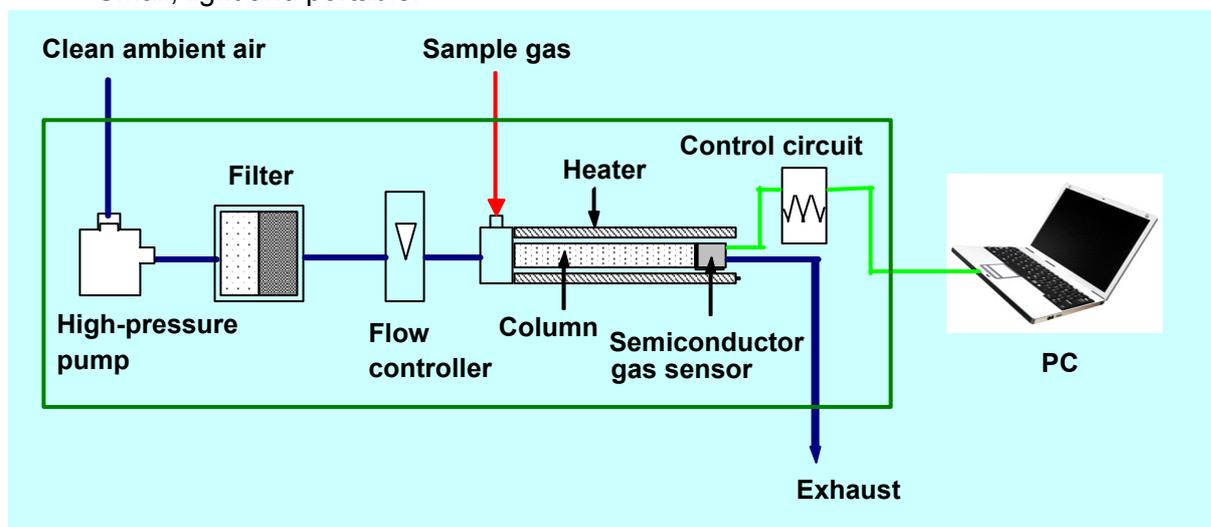- Small, light and portable.

Fig. 2 Block diagram

3. Quantitative measurement

Fig. 3 shows the SGC chromatogram of standard ammonia and trimethylamine.

Fig. 4 shows the relation between the peak height (signal strength) and concentration in Fig. 3.

The relation should be linear in log-log scale because of semiconductor characteristics.

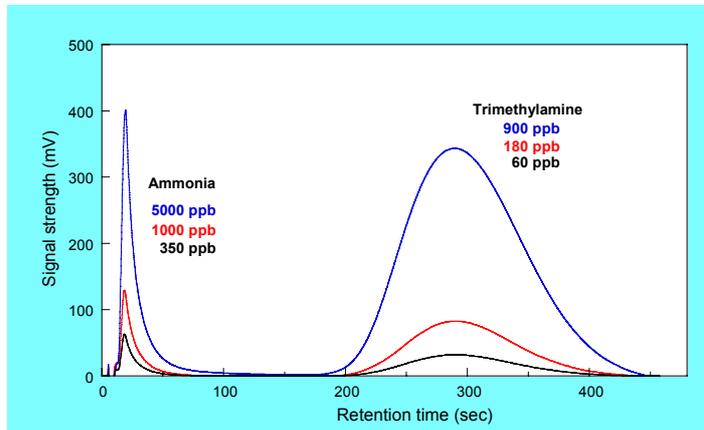Other concentrations can be calculated based on this relation.
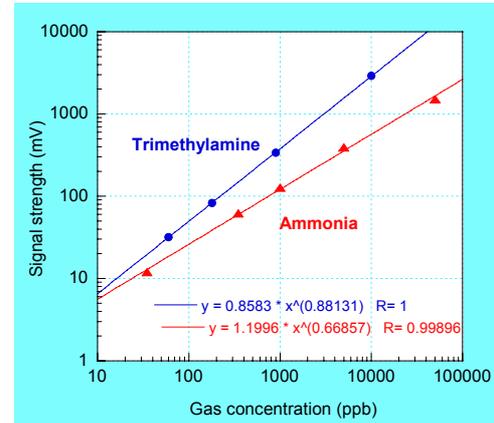

Fig. 3 Standard chromatogram

Fig. 4 Gas conc and signal strength

4. Data analysis

Measured data is analyzed with our original software "SGC Analyzing Software" which is supplied with the device.

- General gas chromatograph (GC) uses a peak area for quantitative measurement. But SGC uses the peak height. This method has almost no effect of interference gases of which retention time is near to the target gas. The measurement accuracy and reproducibility is the same as those of peak area calculation.
- The baseline of the measured chromatogram is corrected to obtain the precise peak height. Small incline of the baseline will not influence the measurement accuracy.
- Other gas peaks may appear before the target gas peak. Such peaks cannot be separated by the column used in the SGC. If the sample gas includes a large amount of such gases, their peak may override the target peak. In order to solve this, the interference gas pattern can be separated from the target pattern as shown in Fig. 5.
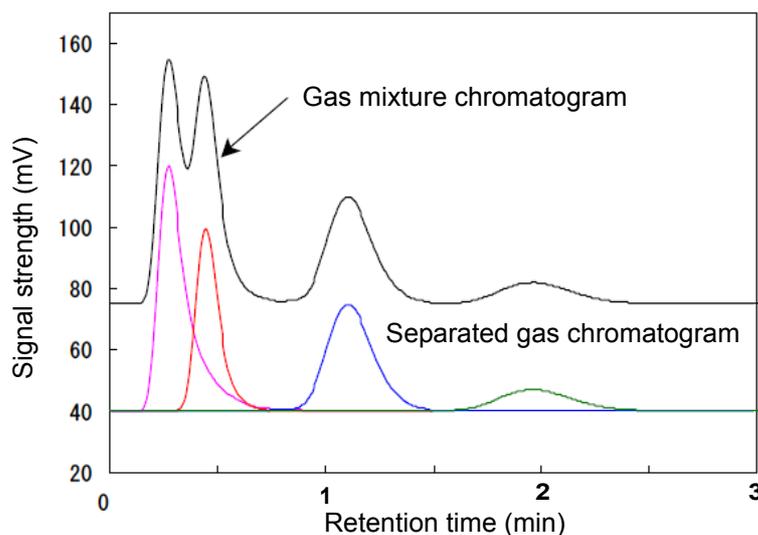

Fig. 5 Chromatogram of gas mixture and separated gases

Semiconductor gas sensor

SGC uses an SB type semiconductor gas sensor which is developed by FIS. The semiconductor gas sensor uses metal oxides such as tin dioxide for gas sensing material. The electric resistance of this material is changed when the gas is adsorbed on its surface. The sensor makes use of this property for gas detection. The SB sensor has features suitable to the detector of gas chromatograph; small size, low power consumption, high sensitivity, and quick response. Especially, the SB sensitivity is much higher than the sensitivity of the detector used in general GC. This feature has realized highly sensitive GC with a small amount of sample gas.
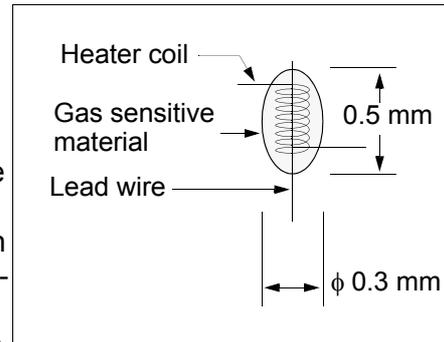
Terms

Gas chromatograph

Gas chromatography is a technology to separate mixture gas into each component with a column and carrier gas. The instrument used to perform gas chromatography is called a gas chromatograph. The resulted chart is called a chromatogram.

Column

A tubing filled with filler material having different adsorbing capability. The material and heating temperature (column temperature) are selected according to the target gas.

Detector

Device to detect the separated gases and change to an electric signal. SGC uses a semiconductor gas sensor as a detector.

Carrier gas

Gas always passing through a column and moving the sample gas. Usually, inert gas such as hydrogen, helium, and nitrogen is used. SGC uses air because the semiconductor gas sensor as a detector needs oxygen.

Baseline

A part of a line on the chromatogram showing only carrier gas without sampled gas.

Peak, Peak height

A mountain shape output wave on the chromatogram is called a peak. The distance between the top of the peak to the baseline is called peak height.

Retention time

Time required for the specified compound in the sampled gas to detect from the time of injection. Retention time determines the kind of detection gas.

Sensor output (Vs)

Voltage to which electric conductivity change of highly sensitive semiconductor gas sensor is converted.

Signal strength

Voltage equal to "Vs(0) - Vs" where Vs(0) is the Vs when the measurement starts.

2. Basic performance

1. Accuracy

Measured concentration accuracy immediately after the calibration is +/-15%.
Fig. 6 shows the measurement results 3 or 5 times immediately after the calibration, indicating an accuracy of +/-15%.
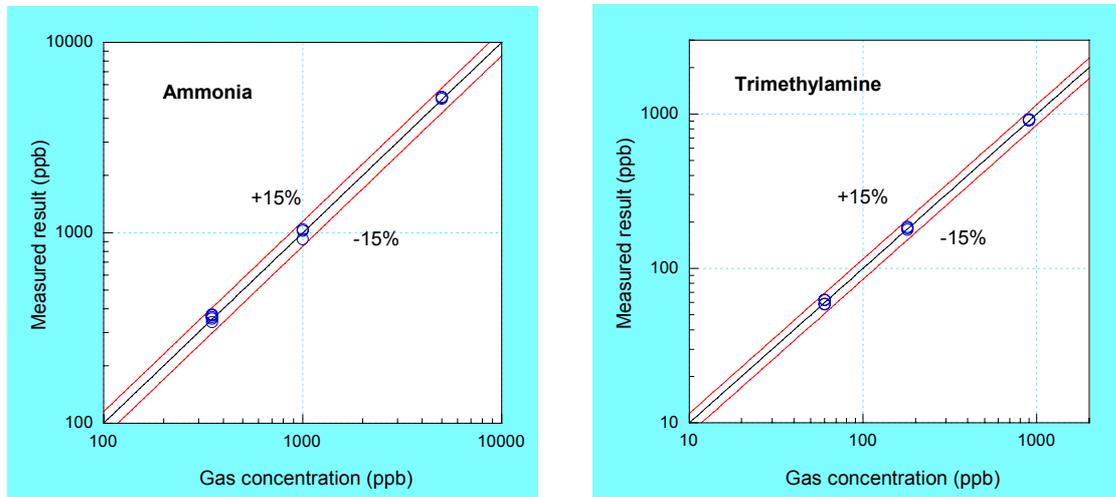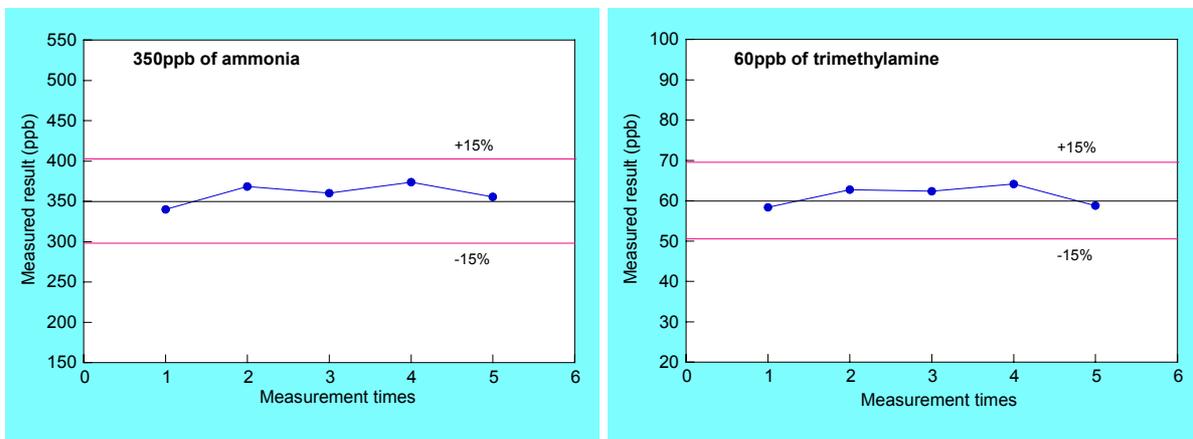
**Fig. 6 Gas conc and measured result**

2. Reproducibility

350 ppb of standard ammonia and 60 ppb of trimethylamine were continuously measured with manual gas injection 5 times after the calibration. Fig. 7 shows the results, indicating good reproducibility.

Fig. 7 Reproducibility
Manual measurement

6

220 ppb of standard trimethylamine was continuously measured with an optional automatic gas injector 90 times (measurement interval: one minute). Fig. 8 shows the results. The average is 223ppb and 3 sigma is 4ppb meaning about 2% of the concentration. The data indicates good accuracy and reproducibility.
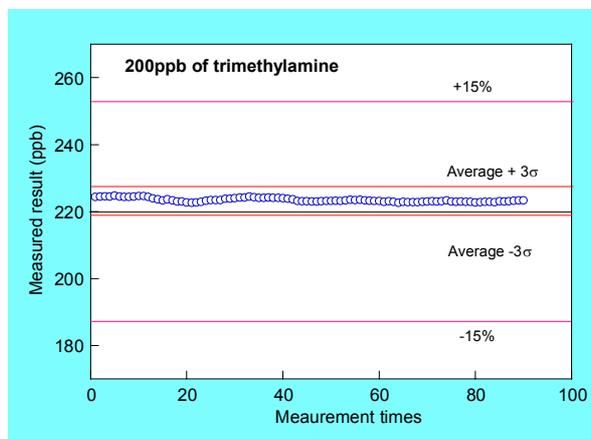
Fig. 8 Reproducibility
Continuous auto-measurement

3. Interference gases

ODNA-P2 responds to some other gases than ammonia and trimethylamine.
Table 1 shows the retention time for gases. The signal strength is the relative value if the strength of ammonia is 100.
Please use the retention time and signal strength in the table just as a reference because they depend more or less on the individual device.
If other gas than ammonia and trimethylamine exists and its peak position is near to those of ammonia and trimethylamine, it may influence the accuracy. Long retention time gas may influence the baseline stability for the following measurements, not the current measurement.

Table 1: Retention time and signal strength for various gases

Gas	Chemical formula	Measured conc (ppm)	Retention time (sec)	Signal strength	Remarks
Hydrogen	H_2	5	11	129	
Oxygen	O_2	0	13	10	* Note 1
Formaldehyde	HCHO	1	11	108	
Sulfur dioxide	SO_2	10000	11	103	
Ethylene	C_2H_4	1	13	104	
Ammonia	**NH_3**	**5**	**24**	**100**	
Hydrogen sulfide	H_2S	10	72	172	
Methylamine	$NH_2(CH_3)$	8	141	137	
Methyl mercaptan	CH_3SH	1	181	114	
Acetaldehyde	CH_3CHO	1	181	143	
Trimethylamine	$N(CH_3)_3$	1	257	78	

*Note 1: The carrier gas used in SGC includes 21% oxygen corresponding to the ambient air. Then, the semi-conductor gas sensor's output indicates this oxygen concentration when no sample gas exists. If the oxygen concentration is largely different from 21%, the corresponded oxygen peak will appear. If the oxygen concentration in the sampled gas is much larger than 21%, the peak height will be higher. If it is much lower than 21%, the peak height will be lower. This data indicates the output for no oxygen, such as 100% nitrogen.

1. Initial stabilization time after power-on

It takes 10 to 30 minutes for the READY lamp to turn on immediately after power-on. This time is required for the stabilization of column temperature and sensor. It would be better to power on the SGC for more than 1 hour before measurement for higher accuracy.

2. Carrier gas amount

The retention time largely depends on the carrier gas amount. If the retention time largely shifts, measurement accuracy will be lower, and peak position may not be detected. If the carrier gas flow rate is shifted +/- 3cc from the initial setting, adjust the flow rate. Confirm the initial setting in the CD supplied with the device.

3. Ambient temperature

Rapid temperature change such as power-on the air-conditioner will cause the baseline drift. Use the SGC under as small a temperature change as possible. Large baseline drift will cause the SGC to be in a WAIT status. Start the measurement after the status is READY.

4. Ambient atmosphere

Since the ambient air is used as a carrier gas, the measurement accuracy for the sulfide will become lower if large amount of interference gas co-exists in the ambient atmosphere. Avoid such atmosphere for the measurement. Do not worry about the influence of temporarily existing gas such as sprayed gas.
SGC enters a WAIT status when detecting the polluted atmosphere. Start the measurement after the status is READY.

5. Measurement after a long time of non operation

When you use the SGC after a long time of non operation, the measurement data may show a bit lower results. If the non operation time is more than two weeks, power on the SGC for several hours on the previous day of the measurement to obtain higher accuracy.