A Gas Filter Correlation Monitor for CO, CH₄, and HCl

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SUMMARY

A fast-response instrument for monitoring the atmospheric constituents CO, CH₄, and HCl (using a modified nondispersive infrared technique) was designed, assembled, and tested. This gas filter correlation method uses a sample of gas to provide a selective filter for radiation absorbed in a gas mixture containing the specified gas. Depending on the spectral-line broadening, temperature, and optical depth of the gas selected, exceptionally high spectral resolution may be attained. A description of the single-beam rotating-cell system and its specific application is presented along with the signal processing circuit. Calibrations of the instrument show that the technique can be used to measure CO, CH₄, and HCl concentrations as small as 5 ppm-m. A field version was employed to measure diurnal variations of CO and CH₄ and the interfering effects of other atmospheric gases were analyzed.

INTRODUCTION

The use of optical techniques for real-time monitoring of trace gases in the lower atmosphere requires an instrument of high-spectral resolution and high sensitivity while maintaining a relative fast response time. A number of optical instruments have been developed for this purpose in recent years, including both dispersive and nondispersive absorption methods, fluorescent methods, Raman-scattering methods, and laser absorption. A new version of the gas filter correlation analyzer, using a nondispersive infrared technique, was selected for this study because of the ease of adapting the instrument to a number of important trace gases and because the complexity of fine-line data analysis is significantly reduced compared with that of dispersive techniques.

To illustrate the adaptability, three gases (CO, CH₄, and HCl) were selected for this study because of their importance in current tropospheric studies. Among the trace gases present in the lower atmosphere, CH₄ and CO have relatively high concentrations (about 1.5 ppm for CH₄ and from 0.01 to over 10 ppm in urban areas for CO). Although accurate production rates are not available, most atmospheric CH₄ is thought to result from anaerobic bacterial decomposition of organic matter. Human activities can be related to CH₄ emission by the increased turnover of organic matter, geothermal production, and the production of coal fields, natural gas, and petroleum. Furthermore, the oxidation of CH₄ is probably the most important natural source of atmospheric CO; thus, both CH₄ and CO are key gases in the overall study of air-pollution sources and sinks.

HCl is of concern because of the large amount of this toxic gas released from solid rocket motors into ground exhaust clouds. Results of the HCl version of this study are being applied to an airborne version of the instrument for use in mapping HCl in an exhaust cloud mixing with the atmosphere.
It is important to point out that each application of the gas filter correlation technique has its own unique problems. The instrument described in this report is used for a specific application which includes a constant light source and a known path length. Major modifications are necessary to use this relatively simple version of the gas filter correlation technique in more complicated situations such as those encountered in airborne or satellite operations where the radiation from the Earth's surface is used as an energy source.

**SYMBOLS**

- \( P_r \) resolving power
- \( R \) reference gas
- \( S \) specifying gas
- \( V_s \) voltage output of detector with specifying cell in path
- \( V_r \) voltage output of detector with reference cell in path
- \( \Delta V = V_s - V_r \)
- \( \lambda \) wavelength
- \( \lambda_1 \) wavelength of first spectral line

**APPARATUS AND METHOD**

Infrared Measurement Techniques

All infrared (IR) analyzers require a source, a detector, an absorbing medium, and a method of wavelength selection. The nondispersive infrared technique does not use prisms or gratings, but uses a sample of gas to provide a selective filter for radiation absorbed in a gas mixture containing the specified gas. When two similar cells, one containing the specifying gas and one containing a nonabsorbing gas or a vacuum, are placed alternately in a light beam, the radiation measured by the detector collecting the beam is differentially modulated only at the wavelengths at which the specifying gas absorbs. If the optical depth of the specifying gas cell is increased to where the radiation is very strongly absorbed at these characteristic wavelengths, then any small additional absorption of the beam by the specified gas located outside the cells will cause a small decrease in the modulated signal since the additional absorption will produce a larger signal change when the nonabsorbing cell is in the beam than when the specifying cell is in the beam. The difference in signal change is a result of the nonlinear characteristic of molecular energy absorption of light. For a given concentration of specified gas outside the cell, the largest signal change would occur if the optical depth of the specifying cell could be increased to where no additional outside absorption
is detected when the specifying cell was in the beam. In this manner, the specifying cell acts as a selective gas filter for radiation absorbed by a mixture of atmospheric constituents.

Resolving Power

Resolution attained with the gas filter correlation technique may be exceptionally high but depends on the spectral-line broadening, temperature, and optical depth of the specifying gas selected. The definition of resolving power of a dispersive instrument is given by

$$P_r = \frac{\lambda}{\Delta \lambda}$$

where $\Delta \lambda$ is the smallest difference in wavelength of identical spectral lines that can be discriminated and $\lambda$ is the average of the wavelengths $\lambda_1$ and $\lambda_1 + \Delta \lambda$. By use of the Rayleigh criterion, two lines are resolved when the maximum of one falls on the first minimum of the other. For the nondispersive gas filter technique, wavelength selection results from the absorption by the specifying gas and the resolving power is determined by the width of the rotational lines within the gas absorption band spectra. Only energy at wavelengths within these narrow intervals results in a change in the analyzer output.

The Rayleigh criterion is attained when

$$\Delta \lambda = 1.2 \lambda_{1/2}$$

where $\lambda_{1/2}$ is the line half-width. The natural line widths in the rotational bands of interest in this study are in the order of 0.001 to 0.01 Å and the widths produced by the Doppler effect are from 0.01 to 0.1 Å (ref. 1). Since the nondispersive gas filter technique uses a number of absorption lines which differ in half-width and whose values are of questionable accuracy, the actual resolving power of this type of instrument is generally not calculated. However, because the resolution is a function of the line half-width, the resolving power of a gas filter correlation analyzer should be equal to or better than the typical dispersive instrument whose resolution is determined by slit-width limitations. Since line width is a function of pressure and optical depth, the selectivity can be increased by decreasing the partial pressure and optical length of the specifying gas cell. Gas filter correlation also offers the advantage of using the contribution of a large number of absorption lines of a band system without decreasing resolution; thereby the radiation level is increased above that generally available when using conventional spectroscopic instruments.

Gas Filter Optical Design

Although nondispersive infrared techniques have been applied to gas measurements for over three decades (refs. 2 to 7), the need for higher sensitivities in air-pollution monitoring of trace gases has recently accelerated the
development of these instruments. Improvements are being accomplished by modifications of the optical systems and by advances in the electronic signal processing. Since instruments are needed that can measure concentrations of parts per million over a 1-m path length of light (ppm-m), the electronic-processing system must detect and measure a signal difference in the range of one part in a thousand.

The basic elements of the gas-filter correlation analyzer designed for this experiment are shown schematically in figure 1. The basic instrument includes (1) a parallel IR beam provided by an IR source and a parabolic mirror; (2) an IR lens; (3) a rotating cell, partitioned so that half of the cell acts as a specifying cell (filled with the gas of interest - CO, CH₄, or HCl) and the other half is evacuated or filled with a transparent gas such as N₂ and acts as a reference cell; (4) a rotating chopper, with nine evenly spaced slots on the reference cell side and ten evenly spaced slots on the specifying side, and permanently attached to the rotating cell so that the fast light chop will remain synchronized with the slower modulation chop from specifying cell to reference cell (see fig. 2 for details of chopper blade); (5) a narrow band-pass filter; (6) an IR detector; and (7) an electronic signal processor.

Radiation from the source passes through the calibration cell (or the atmosphere) where it is spectrally absorbed by the gas specified (CO, CH₄, or HCl) and other absorbing gases some of which may cause interference effects. The light beam, after being focused by the IR lens onto the filtered detector, is alternately passed through the specifying cell and the reference cell and is chopped as the cell rotates. When the radiation passes through the reference cell, it is not attenuated further; but when it passes through the specifying cell, it is strongly attenuated in the spectral interval of the absorbing specified lines. Therefore, a modulated chopped signal is generated by the detector as shown in figure 1. The magnitude of the change of this modulated signal is related to the concentration of the gas to be detected in the calibration cell or in the atmosphere.

A narrow band-pass filter is selected which passes only a small portion of the absorption band of interest so that the detector views a very limited part of the wavelength interval being emitted by the source. In this manner the effects of interfering gases and source variations with wavelength are minimized.

To eliminate variations of the output signal due to source intensity fluctuations, the modulated signal change is electronically divided by the detector output when the specifying cell is in the light path. This signal, which does not vary with the specified gas concentration in the atmosphere, can be detected by a lock-in amplifier since it has a chop frequency different than the reference cell chop.

A photograph of the optical system is shown in figure 3. The rotating split cell has a 15-cm inside diameter and the inside separation between the end windows (optical path) is 2.54 cm. Undiluted specifying gas is generally used in the cell and results in an optical thickness of 2.54 atm-cm. (1 atm = 101.3 kPa.) The IR lens has a focal length of 20 cm and a diameter of 4 cm. This optical system is portable and is mounted on a tripod for ease
of alinement. A quartz lamp is used as a light source for the $\text{CH}_4$ and $\text{HCl}$ instruments and a glowing silicon carbide rod is used for the CO instrument.

Signal Processing Circuit

The electronic system designed for processing the IR detector output is shown in figures 4 and 5. Infrared radiation chopped at three frequencies (21 Hz (cell chop), 420 Hz (specifying cell chop), and 378 Hz (reference cell chop)) falls on the PbSe detector. The output of the detector is amplified by a low-noise preamplifier located close to the detector to maintain the highest possible signal-noise ratio and then is split into inputs to the 21-Hz and 420-Hz circuit channels.

The 21-Hz input, which contains the information on variations in outside specified gas concentration, is passed through a band-pass filter with variable gain to remove the 420-Hz and 378-Hz components of the signal and to reduce the noise level. The output of the filter then goes into the null-and-add circuit shown in figure 4 which is used to electronically null the effects of absorption within the specifying cell. Nulling the 21-Hz signal is necessary in order to obtain a zero output signal for zero specified gas concentration in the optical path in order to increase the sensitivity of the phase sensitive detector for the measurement of very small signals resulting from very small gas concentrations.

The output of the null circuit is then fed to the 21-Hz phase sensitive detector which is synchronized to the input signal by gate pulses from the 21-Hz phase control circuit which are derived from the 21-Hz reference signal detectors. The reference signal detectors obtain their signals from separate choppers attached to the rotating cell. A phase-sensitive detector and phase-control circuit combination constitute a lock-in amplifier which produces a dc output that is proportional to the root-mean-square (rms) value of the signal input amplitude.

Demodulation of the 420-Hz signal is accomplished by sending the amplified IR detector pulse directly into the 420-Hz lock-in amplifier system which eliminates both the 21-Hz and the 378-Hz signals. The dc voltage output is therefore proportional to the input amplitude of the specifying cell chopped signal. Range selection steps of input voltage to output voltage from $10^{-1}$ to $10^{-5}$ are provided to span a large range of gas concentrations. The phase-sensitive detectors are followed by low-pass filters with selectable time constants permitting longer integration times for low-level signals. The dual-channel dc signals are then fed into a divider circuit to produce the system output which is monitored by a strip recorder. Panel meters are provided to read the output of each phase-sensitive detector and the divider output and controls are available on the front panel and on an inside panel so that each component of the system is adjustable.

Details of the null-and-add circuit are shown in figure 5. The synchronous 21-Hz gated signal from the 21-Hz phase control circuit is fed through a phase shifter and into an amplitude modulator circuit. Modulation of the 21-Hz square-wave amplitude is controlled by the dc output of the 420-Hz phase
sensitive detector which detects only the variation of the light source seen by the optical system. This modulated signal is then modified into a sine wave by using a 21-Hz band-pass filter with variable gain which serves as the zero control. For proper nulling, the sine wave is shifted 180° out of phase with the 21-Hz sine wave obtained from the IR detector and then both are fed into the add circuit where the 21-Hz signal offset can be effectively balanced. When an electronic zeroing scheme is utilized, it is essential that the time constants for both the signal to be nulled and the nulling signal be as identical as possible. If the time constants are not matched, the 21-Hz phase sensitive detector will sense a phase shift in the add circuit when a fast fluctuation of the light source occurs. The output signal resulting from this phase shift may be larger than the specified gas concentration signal when the sensitivity range of the instrument is set at a high level. A photograph of the electronic processing system is shown in figure 6.

Spectral Characteristics of Gases

Features of the characteristic transmission spectra used in this study are shown for each of the three gases in figures 7, 8, and 9. These measurements were obtained by using an optical spectrograph which was scanned over the wavelength range of interest to measure the intensity of the IR beam after it passed through the interference filter and specifying gas. The CO absorption band is presented in figure 7 and includes part of the P and R branches of the CO band near 4.7 μm. The CH₄ absorption band near 3.3 μm is presented in figure 8 and includes P and R branch lines and the entire unresolved Q branch which is not present in the CO and HCl bands. Characteristic features of the HCl band near 3.4 μm are seen in figure 9. This band-pass filter is slightly broader than the others and includes only one R branch line and seven lines of the P branch. Doublets exist for each line of the band as a result of the isotopes HCl₁₃⁵ and HCl₁₃⁷. Undiluted instrument grade CO, CH₄, and HCl gases were used in the specifying cell; as a result the optical thickness was 2.5 atm-cm. Under these conditions, the radiation from the source is strongly absorbed at the characteristic wavelengths of each gas.

The line resolution shown in figures 7, 8, and 9 is a result of the spectrograph used and does not indicate the gas filter correlator resolution which is much greater. Resolution of the gas filter can be increased by decreasing the optical thickness of the specifying gas which results from decreased line broadening in the specifying gas. Increased resolution becomes important if interfering gas absorption lines exist within the band-pass filter wavelength range and overlap the specifying gas lines.

RESULTS AND TESTING

Calibration

To calibrate the instrument for a particular gas, the specifying cell must first be charged with the gas to be measured and the proper interference filter
installed in the detector assembly. The instrument is then electronically zeroed and the gain controls are adjusted for a full-scale reading over the sensitivity range of interest.

Certified standard gas mixtures containing known amounts of CO, CH₄, or HCl in prepurified N₂ are then admitted to the calibration cell shown in figure 1 with the IR source moved close to the instrument to eliminate atmospheric absorption of the gas to be measured. Gas concentration and instrument output are recorded to obtain the calibration data presented for CO in figure 10, for CH₄ in figure 11, and for HCl in figure 12.

The lower detectable concentration for each of the gases is about 5 ppm-m and occurs when the signal-noise ratio reaches unity, a time constant of 1 second being used. Lower detectable concentrations may be achieved by using longer time constants and much lower gas concentrations than 5 ppm may be measured if longer light paths are used. The upper detectable concentration is reached when the optical depth in the sampling path approaches the optical depth in the specifying cell. Measurements of trace gas concentrations above several hundred ppm are unnecessary in pollution measurements, but the calibration indicates that the instrument is sensitive above 6000 ppm-m and is fairly linear up to 300 ppm-m.

Interfering Gases

Interfering effects were evaluated for other gases that may be present in the atmosphere including CO₂, CO, H₂O, CH₄, C₂H₄, C₂H₆, C₃H₈, C₄H₁₀, and N₂O. These tests consisted of admitting known concentrations of the interfering gas into the calibration cell and observing instrument signals which would indicate equivalent concentrations of the specified gas. The results for CO are presented in figure 13 and only N₂O is found to cause interference. The CO calibration curve is also included in this plot for comparative purposes. Since an N₂O band is known to overlap the R branch of the CO band, this effect, although small, was expected. By using figures 10 and 13, it can be estimated that equivalent signals are obtained for about 1000 ppm-m of N₂O and 10 ppm-m of CO. If this ratio is extrapolated down to the anticipated midrange concentration for atmospheric CO, then 100 ppm of N₂O gives an equivalent signal as 1 ppm of CO. Since the measured atmospheric concentrations of N₂O are generally less than 500 ppb (ref. 8), the equivalent CO concentration would be less than 5 ppb. The resulting error would be insignificant for CO atmospheric measurements.

Results of the interfering gas study for the CH₄ instrument version are shown in figure 14 along with the CH₄ calibration curve. Positive interference effects were found for C₂H₄ and C₂H₆ but negative correlations were found for C₃H₈ and C₄H₁₀. Positive interference results from absorbing gas lines that overlap the specified gas lines in the filter wavelength interval and give rise to a decrease in the transmission through the reference cell but do not significantly affect the transmission through the specifying cell. Negative interference results from absorbing gas lines that do not overlap the specifying gas lines in the filter wavelength interval and are an effect of the nulling procedure. In this case, the transmission through both cells is decreased but by
different amounts since the electronic zeroing circuit has reduced the detector output when the reference cell is in the IR path in order to null the instrument output.

As in the CO instrument, it can be shown that the interfering effects of the hydrocarbons are small when the instrument is set up to measure atmospheric CH₄. Not only do the positive and negative interfering signals tend to cancel each other out, but the total hydrocarbon concentration of the atmosphere, excluding CH₄, is generally an order of magnitude less than the CH₄ concentration. Typical values measured (ref. 9) are as follows:

<table>
<thead>
<tr>
<th>Gas</th>
<th>Value, ppm</th>
</tr>
</thead>
<tbody>
<tr>
<td>CH₄</td>
<td>2.355</td>
</tr>
<tr>
<td>C₂H₆</td>
<td>0.0636</td>
</tr>
<tr>
<td>C₂H₄</td>
<td>0.0636</td>
</tr>
<tr>
<td>C₄H₁₀</td>
<td>0.0276</td>
</tr>
<tr>
<td>C₃H₈</td>
<td>&lt; 0.0000</td>
</tr>
</tbody>
</table>

If the interfering signal of each interfering hydrocarbon measured is weighted according to the ratio of that specific gas concentration to the CH₄ concentration listed above and then added, the total result will approximate the line of long dashes shown in figure 14. For a 2.4-ppm concentration of CH₄, the error would amount to about +1 percent or an equivalent CH₄ concentration of 24 ppb.

Although some overlap exists between the HCl and CH₄ bands, no CH₄ interference was found on the HCl version of the instrument. Of the gases tested, C₃H₈, C₂H₆, and C₄H₁₀ did produce interference effects and all indicate a negative correlation as shown in figure 15. Again the results of these signals, when weighted according to their atmospheric concentration, would be insignificant for an HCl sensor used to measure this gas in the ppm range.

Interference effects of H₂O are difficult to determine for a long-path instrument because the H₂O saturation levels of air limit the upper obtainable concentration. Two tests were performed to check H₂O effects. One consisted of introducing moist air into a 1-m calibration cell while monitoring the instrument output. The air was saturated by passing it through a steam chamber and then cooling it to room temperature. Since the optical path may be many times longer than 1 m, the H₂O concentration is much less for this test than the maximum that could be encountered in the field depending on the humidity and the path length. The second test consisted of simply increasing the path length from 5 to 100 m during a typical humid day and observing the difference in the indicated gas concentration due to H₂O in the path when the change in path length is accounted for. No H₂O interference effects were observed in any version of the instrument. When very long paths are considered or if measurements are taken in extremely humid weather, the second test should be repeated as a standard operating procedure.
Field Measurements

Variations of the CO and CH₄ concentrations were monitored over approximately one diurnal cycle to check the adaptability of the instrument to field operations. Zeroing was accomplished by moving the IR source as close as possible to the instrument and electronically zeroing the output. The IR source was then moved back to obtain a path length of 50 m and the intensity of the source was adjusted so that the peak-to-peak signal measured by the IR detector remained constant. A calibration check was made by using a 3.3-cm cell placed in the light path. Gas concentrations were then continuously monitored for the time period shown in figures 16 and 17.

The CO concentration measurements indicate a continuous rise between 6 and 8 p.m. on March 8, 1977, to over 3 ppm, and then a slow decrease to below 1 ppm during the night. A similar behavior was observed on the morning of March 9, 1977. These measurements were taken at Langley Research Center which is located about 13 km north and east of the center of the urban area comprising Hampton and Newport News, Virginia. The wind was continuously from the south and southwest over this period of measurement. Under these conditions, the maximum values of CO concentration are attributed to the result of the evening and morning "rush" hour emissions from automobiles in the urban areas to the south of Langley Research Center.

Also included in figure 16 are 1-hour averages of the CO concentration measured independently with a commercial IR absorption instrument at the Industrial Park, Hampton, Virginia. This site is located approximately 13 km southwest of the Langley site. The agreement of the two sets of data is excellent and the time lag is to be expected since the wind was moving the air mass containing the CO from Hampton to Langley.

Variations of CH₄ concentration are presented in figure 17 and indicate a large CH₄ event to have occurred between 4 and 8 a.m., December 10, 1976. These measurements were also taken at Langley Research Center near a saltwater marsh. The wind speed during the time of high CH₄ concentrations was effectively calm and from 1 to 5 km/hr during the rest of the period. Because of the wind-related effect, the high CH₄ concentrations are assumed to be due to local natural sources resulting from anaerobic bacterial decomposition of organic matter. No independent measurements of CH₄ were available for comparison.

CONCLUDING REMARKS

A fast-response instrument for monitoring the atmospheric constituents of CO, CH₄, and HCl (using a modified nondispersive infrared technique) was designed, assembled, and tested. This gas filter correlation method uses a sample of gas to provide a selective filter for radiation absorbed in a gas mixture containing the specified gas. Depending on the spectral-line broadening, temperature, and optical depth of the gas selected, exceptionally high spectral resolution may be attained. A description of the single-beam rotating-cell system is presented along with the signal processing circuit. The instrument described is used for a specific application which includes a fixed-light
source and a known path length. Calibrations of the instrument show that the technique can be used to measure CO, CH₄, and HCl concentrations as small as 5 ppm-m. A field version was employed to measure typical diurnal variations of CO and CH₄ and the effects of atmospheric interfering gases are analyzed.

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REFERENCES


Figure 1.— Gas-filter correlation technique.
Figure 2.- Details of light beam chopping blade which is permanently attached to rotating specifying cell.
Figure 3.- Photograph of optical system.
(V_R - V_S) + Δ(V_R - V_S) \quad ΔV = Δ(V_R - V_S)

Figure 4.- Schematic of signal processing circuit for gas filter correlation analyzer.
Figure 5.- Block diagram of null-and-add circuit used to zero or balance effects of absorption within specifying cell.
Figure 6. - Photograph of signal processing system.
Figure 7.- Transmission of IR beam through 4.69-μm interference filter and CO specifying cell at band near 4.7 μm.
Figure 8.- Transmission of IR beam through 3.42-μm interference filter and CH₄ specifying cell at band near 3.3 μm.
Figure 9. Transmission of IR beam through 3.56-μm interference filter and HCl specifying cell at band near 3.4 μm.
Figure 10.- Instrument calibration for CO at 1 atmosphere. Solid line is faired through points.
Figure 11.- Instrument calibration for CH$_4$ at 1 atmosphere. Solid line is faired through points.
Figure 12.- Instrument calibration for HCl at 1 atmosphere. Solid line is faired through points.
Figure 13.- A comparison of absorption interfering effects of N$_2$O on CO gas filter correlation analyzer with CO calibration. Solid lines are faired through points.
Figure 14.- A comparison of absorption interfering effects of C₂H₄, C₂H₆, C₃H₈, and C₄H₁₀ on CH₄ gas filter correlation analyzer with CH₄ calibration. Solid lines are faired through points.
Figure 15.- A comparison of absorption interfering effects of C$_3$H$_8$, C$_2$H$_6$, and C$_4$H$_{10}$ on HCl gas filter correlation analyzer with HCl calibration. Solid lines are faiired through points.
Figure 16.- Measurement of variation of CO concentration with time. Solid line denotes gas filter correlation analyzer 1 m off ground over a 50-m path at Langley Research Center. Circles denote commercial IR absorption instrument, Industrial Park, Hampton, Va.
Figure 17.- Variation of CH$_4$ measured with gas filter correlation analyzer 1 m off ground over a 50-m path at Langley Research Center, Hampton, Va.
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