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INFRARED ABSORPTION BY SULFURIC ACID VAPOR

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ABSTRACT

A sample cell has been designed and built to contain hot H_2SO_4 vapor for spectroscopic analysis. The cell is heated to between 190° and 250°C by an electric furnace designed specifically for use with the sample cell. A small bulb connected to the main compartment of the sample cell contains liquid H_2SO_4. The partial pressure of the H_2SO_4 vapor is controlled by regulating the temperature of the liquid H_2SO_4, which is at lower temperature than any other part of the cell that is in contact with the vapor. Transmission curves have been scanned for a variety of samples over the 7.8 μm - 12.8 μm region with spectral slit-widths between 0.5 cm^{-1} and 4 cm^{-1}. At wavelengths of maximum absorption near 8.2 μm and 11.4 μm, the absorption coefficient is approximately 0.0004 (ppm - meters)$^{-1}$.

CONTENTS

		Page
Abstract		iii
Figures		vi
I	Introduction	1
II	Summary	4
III	Conclusions	5
IV	Recommendations	7
V	Experimental Apparatus and Methods	10
VI	Results of Transmission Measurements	18

FIGURES

Number		Page
1	Optical diagram of the apparatus used to study hot H_2SO_4 vapor	11
2	Two views of the sample cell	12
3	End view showing the location of the silicon windows and the method of sealing them	14
4	Side view of the furnace with the sample cell in place	15
5	Spectral curves of transmittance for three samples of H_2SO_4	19
6	Spectrum of H_2SO_4 vapor	20
7	Spectra of H_2SO_4 and H_2O between 7.85 μm and 8.45 μm	21
8	Spectra of H_2SO_4 and CO_2 between 11 μm and 12.5 μm	26

SECTION I

INTRODUCTION

Under a previous contract,[1] Aeronutronic Ford Corporation designed, fabricated, tested and delivered a cross-stack monitor for either HF, HCl, CO, NO or SO_2 that is based on the gas-filter correlation (GFC) principle. The instrument was delivered to EPA in June 1974, and extensive tests performed by EPA personnel since that time have shown that it meets design specifications for HF, CO, NO, and SO_2. Field evaluations have not been performed for HCl, but no problems are anticipated. During the field tests it was determined that a few minor modifications to the instrument would simplify the operation and improve the performance. It was originally intended that the instrument would also measure SO_3 in addition to any of the other 5 gas species, but during the design phase it was determined that the GFC technique is not suitable for SO_3 because of the difficulties in maintaining a known constant amount of this gas in a small cell.

The original objectives of the project reported herein were to (1) make recommended modifications to the existing instrument to simplify operation and to improve its performance, and (2) retrofit the existing instrument with a dispersive correlation accessory that would allow either SO_2 or SO_3 to be measured. The gas of primary importance was SO_3; however, the capability of measuring SO_2 was to be included because the additional cost would be small. Furthermore, the expected performance for SO_2 was better than that obtained with the instrument when employing the GFC technique.

Objective number 1 listed in the previous paragraph has been completed. During the early part of the present contract, EPA scientific personnel investigated the complex chemical reactions involving SO_3 that occur in the stacks of stationary sources. The results of this investigation indicated that the typical effluent probably contains more H_2SO_4 than SO_3. Much of the SO_3 that is formed probably reacts with the H_2O that is present and forms H_2SO_4. Thus, it was decided that the need for an H_2SO_4 monitor was greater that that for an SO_3 monitor.

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1. Burch, D. E., and D. A. Gryvnak. "Infrared Gas Filter Correlation Instrument for In-Situ Measurement of Gaseous Pollutants." Prepared by Philco-Ford Corporation for EPA under Contract No. 68-02-0575. EPA Report No. EPA-650/2-74-094. Also, Burch, D. E., and D. A. Gryvnak. "Cross-Stack Measurement of Pollutant Concentrations Using Gas-Cell Correlation Spectroscopy." Chapter 10 of Analytical Methods Applied to Air Pollution Measurements, Stevens, R. K. and W. F. Herget, (eds.). Ann Arbor, Ann Arbor Science Publishers Inc., 1974.

Therefore, the work on the dispersive correlation accessory for SO_2 and SO_3 was stopped, and the effort was diverted to an investigation of a cross-stack method of monitoring hot H_2SO_4 vapor. Possible problems in maintaining the chemical integrity of a sample as it is drawn through a probe to a remote monitor make this type of instrument much less desirable than a cross-stack instrument that does not disturb the sample.

Alpert[2] has published very high resolution spectra of H_2SO_4 vapor in the spectral region from 1312 to 1549 cm^{-1}. The H_2SO_4 vapor appeared as an impurity in Alpert's samples of HCN, the gas species of primary interest to him. Alpert's spectral curves show approximately 700 separate absorption lines of H_2SO_4, and the positions of more than 400 of them have been measured and tabulated to the nearest 0.001 cm^{-1}. These data are the only data known to the authors that show individual H_2SO_4 absorption lines. The intensities of the lines are quite irregular, and the spacings between adjacent lines vary with an average of approximately 0.3 cm^{-1}. Because of the strong absorption by H_2O, SO_2 and SO_3 in the same spectral region, this H_2SO_4 band is of little value for monitoring purposes.

Chackalackal and Stafford[3] have published low resolution spectra of hot H_2SO_4 vapor in the spectral region 400 - 4000 cm^{-1}. The vapor was produced by heating the azeotropic mixture (98.3%) of liquid H_2SO_4. Some SO_3 was also present in the vapor over the hot liquid; the percentage of SO_3 depends on the temperature and on the amount of excess H_2O. The sampling method employed by these workers made it impossible to determine the amount of vapor present in the sample. Thus, these data provide helpful information on the positions of the strong H_2SO_4 bands and on their relative intensities, but they do not provide quantitative information on the band intensities. The spectral resolution is also quite coarse and the positions of the strong absorption features can not be determined as accurately as is desired.

After the present study was well under way, we learned of a paper by Stopperka and Kilz[4] that also presents infrared spectral data on hot H_2SO_4 vapor. These data are similar to the earlier data of Chackalackal and Stafford,[3] although the later data are probably somewhat more quantitative and less subject to errors due to impurities. Both References 3 and 4 show four strong absorption

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2. Alpert, B. D. Studies in High Resolution Infrared Spectroscopy. Ph.D. Dissertation. (Appendix B contains H_2SO_4 data.) Ohio State University, Columbus, Ohio, 1970.

3. Chackalackal, S. M. and F. E. Stafford. Infrared Spectra of the Vapors above Sulfuric and Deuteriosulfuric Acids. Journ. Amer. Chem. Soc., 88:4, 723-728, Feb. 20, 1966.

4. Stopperka, V. K. and F. Kilz. Die Zusammersetzung der Gasphase über dem flüssigen System H_2O–H_2SO_4 in Abhängigkeit von der Temperatur. Z. anorg. u allgem. Chem., Band 370, 49-66, 1969.

bands of hot H_2SO_4 vapor. They are centered near 3610 cm^{-1} (2.77 μm), 1450 cm^{-1} (6.90 μm), 1224 cm^{-1} (8.17 μm), and 882 cm^{-1} (11.34 μm). The first band listed overlaps strong absorption by H_2O and CO_2. The second band listed , the one on which data are given in Reference 2, is also overlapped by an H_2O band as well as by SO_2 and SO_3 bands. The 8.17 μm band and the 11.34 μm band have been emphasized in the present study because they occur where there is much less absorption by gases likely to be present in samples to be investigated for H_2SO_4 vapor.

SECTION II

SUMMARY

A sample cell has been designed and built to contain hot H_2SO_4 vapor for spectroscopic analysis. The cell is heated to between 190° and 250°C by an electric furnace designed specifically for use with the sample cell. A small bulb connected to the main compartment of the sample cell contains liquid H_2SO_4. The partial pressure of the H_2SO_4 vapor is controlled by regulating the temperature of the liquid H_2SO_4, which is at lower temperature than any other part of the cell that is in contact with the vapor. Typical temperatures of the liquid H_2SO_4 and of the vapor being analyzed are approximately 180°C and 210°C, respectively.

The main body of the cell is made of glass, and the gasket material used to provide a leak-tight seal is resistant to chemical reaction with the H_2SO_4 vapor. Two silicon windows make it possible to transmit a monitoring beam of infrared radiant energy through the hot H_2SO_4 vapor. A gold-coated spherical mirror forms one end of the cell and reflects the monitoring beam so that the cell is double-passed with a total optical path length of 52 cm through the vapor.

A small grating spectrometer has been employed to scan spectral curves of transmittance for several samples over the 7 um to 13 μm spectral region. In addition to the H_2SO_4 vapor, some SO_3 vapor and H_2O are also present in the sample cell. Two strong H_2SO_4 bands occur in this region; wavelengths of maximum absorption are near 8.2 μm and 11.4 μm. Adjacent individual absorption lines within each band are probably separated by only approximately 0.3 cm^{-1} as they are in the 6.90 μm band (see Reference 2). These closely spaced individual lines were not resolvable by our spectrometer, which has a minimum practical spectral slitwidth of approximately 0.4 cm^{-1} in this portion of the spectrum.

The absorption by both the 8.2 μm band and the 11.4 μm band is quite strong. Approximately 250 ppm-meters of H_2SO_4 vapor produces an average absorptance of 10% over an interval of a few cm^{-1} near the strongest absorption in either of these bands.

SECTION III

CONCLUSIONS

Hot H_2SO_4 vapor has four relatively strong absorption bands between 2 μm and 12 μm. The two centered near 2.77 μm and 6.90 μm are probably of little value for monitoring the H_2SO_4 concentration because of the strong H_2O absorption in the same spectral regions. The other two bands, centered near 8.2 μm and 11.4 μm, are much more promising for monitoring purposes. Some interference by H_2O is to be expected in the 8.2 μm region; however, this interference can probably be accounted for if the H_2O concentration is known or is also monitored. Interference by hot CO_2 might be a problem for a monitor that makes use of the 11.4 μm band. This problem would probably be most serious when investigating the exhaust from a coal-burning power plant in which the CO_2 concentration may be as high as 10-20%. Most of the error due to interference by CO_2 can be accounted for by measuring the interference of the monitor to known concentrations of CO_2 and determining, by other methods, the concentration of this gas species in the gas being monitored.

Although the absorption by H_2SO_4 is quite strong, the absorption spectrum does not contain distinct, strong, well-separated features that make infrared detection and discrimination simple for some gases. From other experimental studies,[2] we find that the individual absorption lines have quite irregular spacings and that the average spacing between adjacent strong lines is usually less than 0.3 cm^{-1}. Thus, any dispersing instrument designed to take advantage of the fine spectral structure must have quite good spectral resolution, particularly for a portable field instrument. The most promising infrared monitor for H_2SO_4 would take advantage of the coarser, less-sharp, structure in the infrared spectrum. Such a monitor might involve a dispersing element or a series of narrow-bandpass filters. Because it employs the coarse structure in the spectrum, this type of instrument is more subject to interference by other gas species than is an instrument that makes use of several strong, sharp absorption features.

Gas-filter correlation techniques are commonly employed to exploit spectral structure that is not resolved by the instrument. These gas-filter correlation techniques could probably be employed successfully for H_2SO_4 vapor except for the difficult problem of confining a fixed and known amount of H_2SO_4 vapor in the gas-filter cell, the key component of such an instrument. The high reactivity of the vapor makes it difficult to maintain a vacuum tight seal and to prevent the disappearance of the vapor as it reacts with the walls of the cell and with the windows. Furthermore, the gas-filter cell would have to be heated in order to maintain an adequate pressure of the H_2SO_4 vapor.

Maintaining a constant gas-filter cell temperature in a field instrument is also a difficult problem. Because of these problems, a gas-filter correlation instrument does not seem to be practical for H_2SO_4 vapor

SECTION IV

RECOMMENDATIONS

For the near future, the most promising type of infrared, cross-stack monitor for H_2SO_4 vapor would contain a grating as a dispersing element and would make use of either the 8.2 μm band or the 11.4 μm band. Quite good sensitivity can be obtained by an instrument that compares the transmission in two or more narrow spectral intervals. If only two intervals are compared, one should be in a region of strong absorption by H_2SO_4 vapor, and the other should be in a nearby region where the absorption is much weaker. It may be desirable to choose two or more intervals of strong absorption and compare their combined transmission to that of two or more intervals of weak absorption.

The simplest, and probably the most dependable, method of performing the desired comparison involves using a small grating polychromator with multiple slits in the focal plane of the instrument. Each slit passes one of the selected narrow spectral intervals. A chopper located just back of the slits alternately allows energy to pass through the slits corresponding to the intervals of strong absorption then through those corresponding to weak absorption. A dispersive instrument with sufficiently high spectral resolution to make use of the fine structure due to lines separated by approximately 0.3 cm^{-1} does not appear to be practical for field monitoring. Therefore, it is necessary to use the coarser structure in the spectrum; there is probably little to be gained by making either spectral interval narrower than about 4 cm^{-1}. (This corresponds approximately to 0.03 μm near 8.2 μm and to 0.05 μm near 11.4 μm.)

The choice of the spectral intervals will depend as much on possible interfering gases as on the spectral structure of the H_2SO_4 vapor. Minimizing interference by H_2O is essential if the 8.2 μm band is employed. If the 11.4 μm band is employed, the interference by H_2O is much less, but there may be interference by CO_2 if this gas occurs in high concentrations, as it does, for example, in the exhaust from coal-burning power plants. Other gases that may be present in the sample and interfere with the measurements must also be considered. Ammonia, for example, absorbs strongly at many places within the 11.4 μm H_2SO_4 band and could interfere if it were present. Data shown in Section VI on the absorption by H_2O can be useful in selecting the proper intervals to reduce interference by this gas.

The instrument built previously by Aeronutronic Ford for EPA (Reference 1) can be adapted easily to monitor hot H_2SO_4 vapor by employing two or more narrow spectral intervals as just discussed. A grating assembly designed and built specifically for this purpose would fit into the space used for grating assemblies provided for other gases. As in the assemblies built previously, the grating

and all of the mirrors are fastened permanently into position. Unlike the previously built assemblies, a chopping mechanism alternately allows the passage of energy through the various slits in the retroreflector-grid assembly. The energy transmitted through the slits returns to the grating and is undispersed. After leaving the grating assembly, the energy passes on to the detector. The gas-filter cell and the rotating mirror chopper normally used are not employed with this assembly. The detector signal is processed by the same two-frequency electronics that have been provided with the instrument.

This instrument with a grid-chopper is recommended primarily as a research instrument to obtain approximate values for the concentration of hot H_2SO_4 vapor in the exhaust of various pollution sources and to investigate the problems involved in making accurate and reliable measurements on a routine basis. The instrument can be tested and calibrated in the laboratory by using a heated cell similar to the one described in Section V to contain the H_2SO_4 vapor. The same cell, but with the liquid H_2SO_4 removed, can contain hot CO_2, H_2O, or any other gas that might interfere with the measurement.

With little additional effort, the grating assembly can be made so it is capable of using either the 8.2 μm band or the 11.4 μm band. A multiple-slit grid would be provided for each band, and the grid corresponding to the band not being used would be blocked while the other was left open. Not more than a few minutes would be required to change from one band to the other. Comparison of the results obtained with the two different bands would provide information on the reliability of the instrument and on the interfering gases. A similar set of slits could also be included near 8 μm to measure the concentration of H_2O. By previously measuring the interference by H_2O in the H_2SO_4 channel, the data on the H_2O concentration could be used to account for the H_2O interference. A similar set of slits that pass two or more intervals between 10 and 11 μm might be used to measure the concentration of CO_2 so its interference can be accounted for. A single chopping mechanism could serve for all of the suggested sets of slits that transmit between 8 μm and 12 μm. Only one set of slits would be used at a given time so that only one detector would be required.

On the basis of the data shown in Section VI, we estimate that the minimum detectable thickness of H_2SO_4 for an instrument like the one just described is between 5 ppm-meters and 30 ppm-meters. This value is limited by the uncertainty in the amount of interference by other gases and by the available methods of "zeroing" the instrument with no H_2SO_4 in the sampling path.

For the long term, the best possible infrared monitor for H_2SO_4 vapor would probably contain a tunable laser that could make use of the fine structure in the H_2SO_4 spectrum. As these lasers are further developed and become more convenient to use, they can probably be applied to H_2SO_4 measurements. Before the optimum wavelength interval for such a scanning laser can be determined, it is essential that the spectrum of heated H_2SO_4 vapor in samples at 1 atm total pressure be investigated with a spectral slitwidth less than 0.1 cm^{-1}. It would be better if the slitwidth was less than 0.05 cm^{-1} so that most of the structure could be observed. Such measurements require a high-quality spectrometer with good wavelength accuracy in addition to the very good resolution. A cell similar to the one described in Section V could be used to contain the samples.

A few minor changes should be made in the design of the cell used to contain hot H_2SO_4 for future studies. The most important purpose of these changes is to reduce as much as possible the amount of reactive material in contact with the hot H_2SO_4 vapor. By so doing, the samples can be made purer and there should be much less interfering absorption in the transmission curves. The continued usage of a glass cell body, gold-coated mirror, teflon gaskets and silicon windows is recommended.

Better seals between the endplates and the cell body could be obtained if the teflon gaskets could be compressed more firmly. In order to do this, the thickness of the glass wall of the cell should be increased to at least 5 mm to provide additional strength. Teflon gaskets, preferably O-rings, should be used between the silicon windows and the glass endplate. A mechanism should be designed to press the windows firmly against the gasket without breaking the windows. After the cell has been assembled, any small remaining leaks around the gaskets could be sealed with RTV, or some other pliable cement that is relatively inert and can withstand temperatures up to 250°C. Any small amount of RTV that contacts the H_2SO_4 vapor will probably not decompose to cause a leak or to produce a serious amount of impurity in the sample.

The thickness of the silicon windows should not exceed approximately 0.8 mm so as to minimize the absorption by the silicon. Because the thin windows may be weak, it is recommended that the diameters be 2.5 cm or less to reduce the force on the windows when the cell is evacuated. The surface of each window that contacts the H_2SO_4 vapor should not be coated with an anti-reflection coating. The coatings apparently react with the H_2SO_4 and form a substance that absorbs strongly in the 8 - 10 μm region. An anti-reflection coating on the outside surface of each window will improve the transmission and greatly reduce the fringes caused by optical interference between the two surfaces of each window. Wedging the windows slightly can also reduce, or even eliminate, the fringes.

A teflon valve should replace the glass valve with silicon grease that is used on the present cell in contact with the vapor. There is noticeable decomposition of the grease after a few hours use of the cell. The liquid H_2SO_4 reservoir connected as an appendage to the main part of the cell has worked satisfactorily and should be included on any future cell. The capability of heating the liquid independently of the main body of the cell is quite important and is recommended.

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SECTION V

EXPERIMENTAL APPARATUS AND METHODS

The optical arrangement employed to obtain the spectral data on H_2SO_4 vapor is shown in Figure 1. Radiant energy from a Nernst source is chopped at 450 Hz and is directed through the sample section to a grating monochromator. Mirror M2 forms an image of the source near mirror M3, which is adjacent to the entrance window of the sample cell. Spherical mirror M4 forms one end of the sample cell and re-images the source near mirror M5. Spherical mirror M7 forms another image of the source on the entrance slit of the grating monochromator.

The sample cell is enclosed within a custom-made furnace that heats the sample cell to between approximately 190°C and 250°C. The furnace, which is not shown in Figure 1, surrounds the sample cell and is placed inside a plywood box. The box contains insulation that surrounds the furnace. The radiant energy beam passes through two pieces of glass tubing that hold the insulation away from the monitoring beam. A portion of the furnace not shown in Figure 1 holds the insulation away from the monitoring beam in the region near mirrors M2 and M5.

The monochromator is a modified version of a Perkin-Elmer Model 99 Prism Monochromator. The original prism has been removed and replaced by a grating with 75 lines per mm that is used in the first order. A longpass interference filter placed near the entrance slit eliminates overlapping orders of shorter wavelengths that are also passed by the grating. The detector has a copper-doped germanium element and is cooled by liquid helium. The detector signal is amplified and demodulated by a synchronous demodulator, the dc output of which is recorded on a strip-chart recorder. The spectral slit-width varied from approximately 0.5 to 4 cm^{-1}.

The construction of the sample cell is illustrated by the two views shown in Figure 2. The cell is designed to have a minimum of contact between the hot H_2SO_4 vapor and any material with which it might react. It is necessary to heat the sample cell in order to obtain a sufficiently high pressure of the H_2SO_4 vapor for its spectrum to be obtained. Attached to the main body of the cell by a piece of glass tubing is a small glass reservoir that contains an azeotropic mixture (98.3%) of liquid H_2SO_4 in water. The pressure of the vapor in the sample cell is controlled by regulating the temperature of the liquid H_2SO_4. The liquid H_2SO_4 is maintained a few degrees C cooler than any other part of the cell with which the vapor comes in contact. This avoids condensation on the inside of the sample cell. It is particularly important that condensation be avoided on the cell windows or on the gold-coated mirror. The vapor in the sample cell contains H_2SO_4, SO_3, and H_2O. The relative abundances of the vapors of these three gas species depend upon the temperature

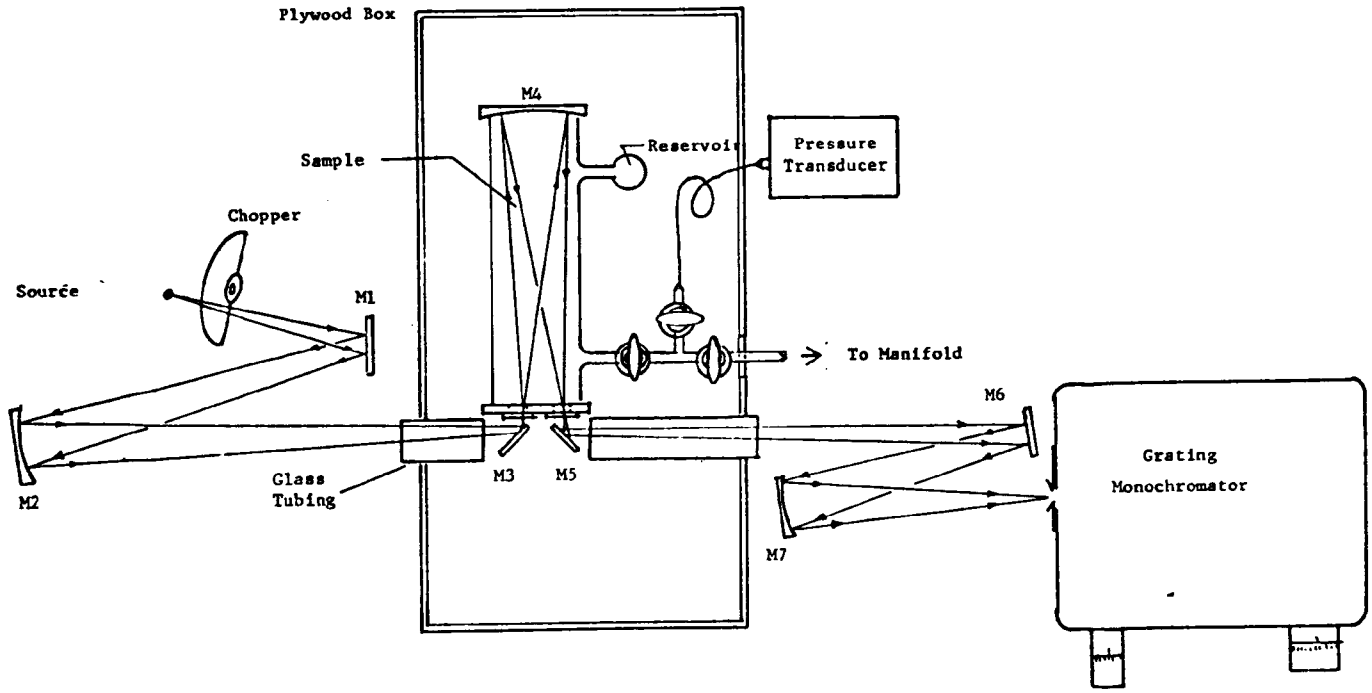
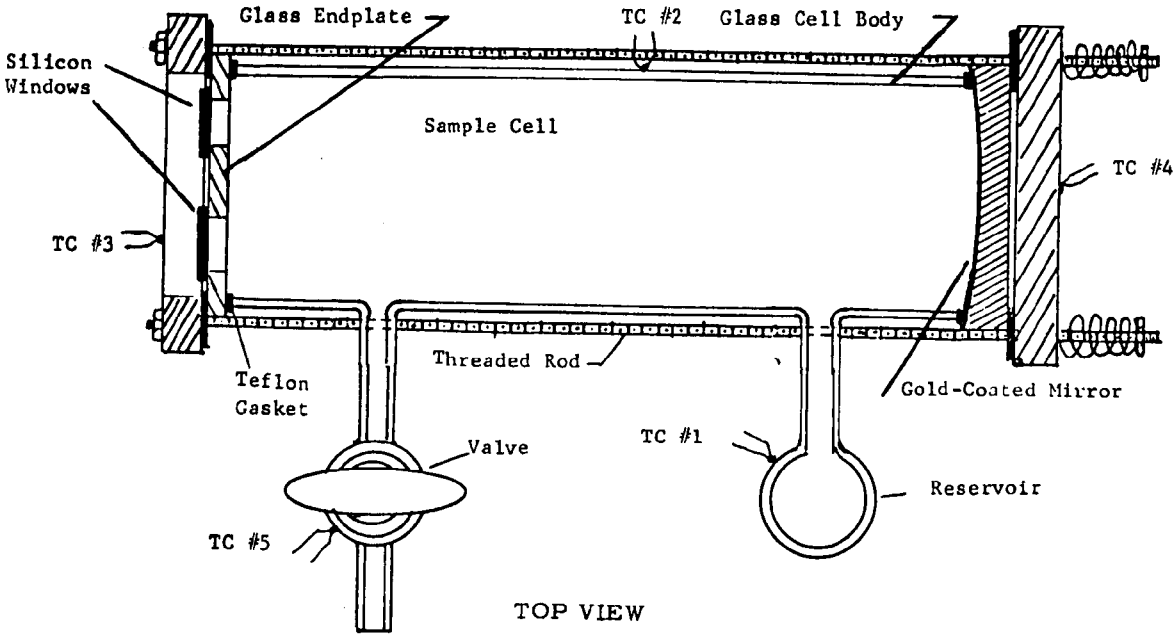


Figure 1. Optical diagram of the apparatus used to study hot H_2SO_4 vapor.



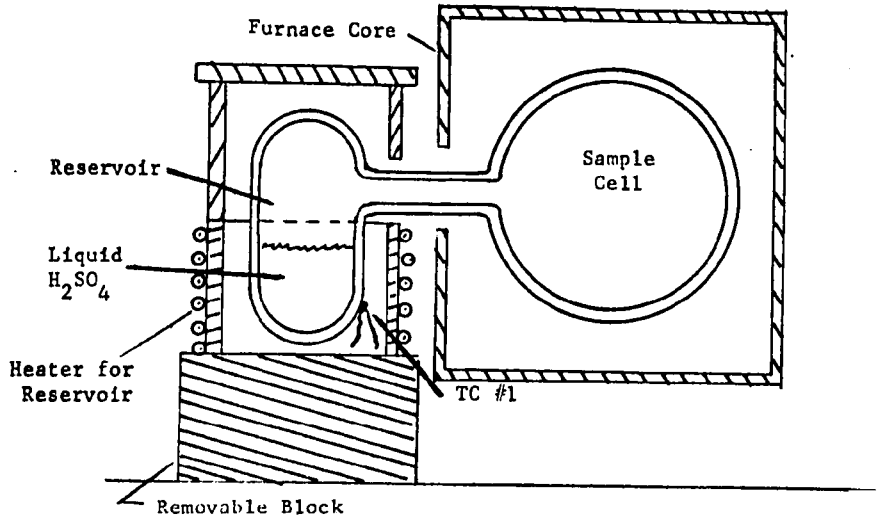


Figure 2. Two views of the sample cell.

and upon the amount of H_2O in the system.

One end of the sample cell is formed by gold-coated mirror M4. The center of curvature of this mirror is near a point midway between the centers of the two silicon windows. Thus, radiant energy coming from an image near one of the windows is refocused to form an image near the other window, as illustrated in Figure 1. A flat teflon gasket forms the seal between the mirror and the cylindrical glass tubing that makes up the main body of the sample cell. A similar teflon gasket forms a seal between the other end of the cylindrical tubing and a glass plate that forms the opposite end of the cell. The glass endplate and the mirror are held in place by two aluminum endplates as illustrated in the upper portion of Figure 2. Coiled springs on each of four pieces of threaded rod are compressed to hold the ends firmly against the cylindrical body of the cell. A flat ring of teflon placed between the aluminum endplate and the mirror serves as a spacer and places the force against the backside of the mirror approximately opposite the gasket that forms the seal. Use of this ring makes the shear on the mirror much less than it would be if the aluminum endplate made contact with the center portion of the mirror. A similar arrangement is used between the glass endplate and the corresponding aluminum endplate on the opposite end of the sample cell.

We experienced problems in maintaining a reliable vacuum seal with the teflon gaskets used as illustrated in Figure 2. This problem was solved by applying a ring of RTV sealing compound around the gasket after it had been installed. A continuous ring of RTV completely covered the portion of the teflon gasket exposed to the outside of the cell. The RTV was bonded to the glass body of the cell and to the mirror on one end and to the cell body and glass endplate on the opposite end. The RTV sealed the leaks around the teflon gaskets with only a very small amount of it in contact with the hot H_2SO_4 vapor. After operating the cell for a few days, we found no evidence of degradation of the RTV used around the teflon gaskets. More important, there was spectral evidence of only a small amount of an impurity in the sample cell that might have resulted from a chemical interaction between the H_2SO_4 vapor and the RTV.

We originally intended to employ teflon O-rings to provide the vacuum seal between the silicon windows and the glass endplate. Because of problems encountered in obtaining a sufficiently tight seal, we found a solution that made use of the RTV in the manner illustrated in Figure 3. Each 25 mm diameter silicon window was placed over a circular hole in the glass endplate. The window was held firmly against the glass plate and the RTV seal was applied around the outer edge of the window as indicated. No RTV was applied to the flat portion of the window that would be in contact with the hot H_2SO_4 vapor. Although a small amount of RTV is necessarily in contact with the H_2SO_4 vapor, we found that the impurities in the sample that result from an interaction between the vapor and the RTV was not serious when the windows were installed carefully by the method illustrated. In one instance, one of the windows was not sealed properly. The RTV was not completely cured when the sample cell was evacuated, and a small amount of RTV was forced by the higher outside pressure through the gap between the silicon window and the glass into the interior of the sample cell. When the cell was heated, the hot H_2SO_4 reacted with the RTV and apparently formed an impurity with a spectrum that interferes with the spectrum of the H_2SO_4. When the cell was dismantled for cleaning, it was apparent that the H_2SO_4 had reacted strongly with the RTV that it had contacted.

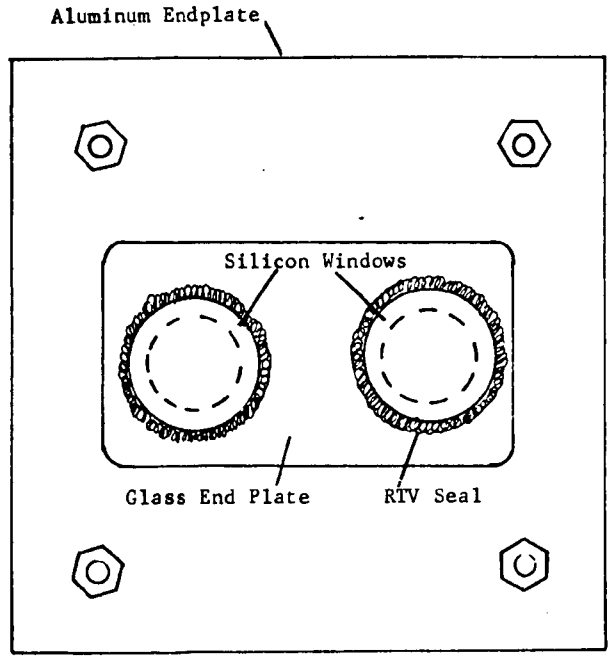


Figure 3. End view showing the location of the silicon windows and the method of sealing them.

The silicon windows that were first intended for use are 2 mm thick and contain an anti-reflection coating on each surface to maximize transmission near 10 μm. Although this coating is optimized for 10 μm, it reduces reflection throughout the entire 7-12 μm region investigated. Absorption by the silicon was bothersome, but it did not seriously affect the quality of the transmission data. Fortunately, the most prominent silicon absorption feature is at approximately 9 μm where it does not interfere significantly with either of the two strong H_2SO_4 absorption bands investigated. A thinner pair of windows, each 0.8 mm thick, were available in the laboratory and were used in order to reduce the absorption by the silicon. However, these thinner windows did not contain an anti-reflection coating, and a problem was created by the interference between the radiant energy reflected from the two parallel surfaces of each window. This interference produced "fringes" in the spectrum of the transmitted energy; the fringes were separated by approximately 2 cm^{-1} and caused more than 20% difference between the maximum and the minimum transmittance. After these tests were completed, the thin windows were removed, and the original 2 mm thick windows with anti-reflection coating were replaced on the cell. We then found that the H_2SO_4 vapor reacted with the anti-reflection coating and produced a film on the windows that absorbed strongly between approximately 8 μm and 10 μm. The best data were obtained with the thin windows and with the monochromator slits wide enough to smooth-out the fringes produced by the windows. A rectangular hole is cut in the aluminum endplate opposite the windows to allow passage of the radiant energy.

The method employed to heat the body of the sample cell and the liquid reservoir is illustrated in the lower panel of Figure 2 and in Figure 4. The stationary core that makes up the main part of the furnace surrounds the body of the sample cell and is made from aluminum square-stock approximately 3 mm thick and 100 mm on each side. Insulated electrical heating wire is wrapped around this aluminum as indicated in the figures. The glass tubing that connects the

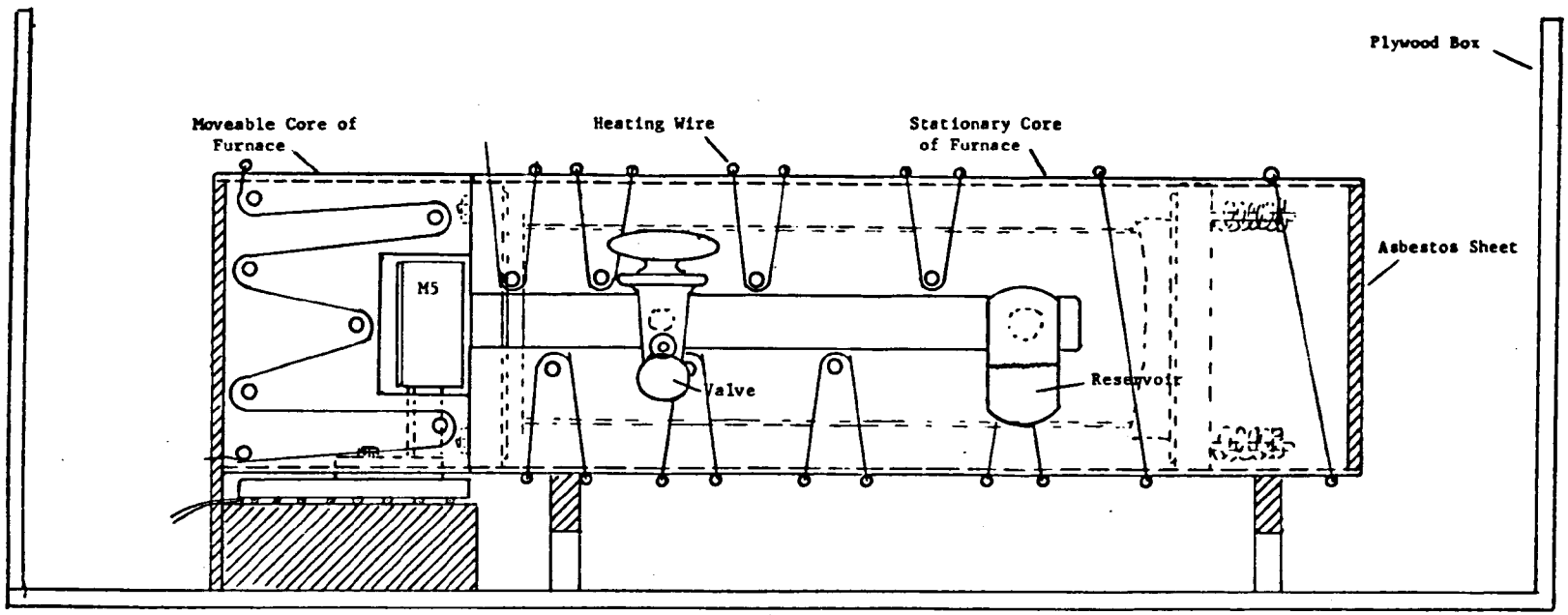


Figure 4. Side view of the furnace with the sample cell in place.

main body of the sample cell to the liquid H_2SO_4 reservoir protrudes through a slot in the side of the stationary aluminum core. This allows the temperature of the reservoir to be regulated almost independently of the temperature of the main body of the cell. The reservoir is enclosed within two cylindrical pieces of ceramic tubing with a ceramic cap over the top as indicated in the lower portion of Figure 2. Heat is supplied to the reservoir by electrical heating wire wrapped around the lower piece of ceramic. Additional heating wire, not shown in Figure 2, is wrapped around the glass tubing that connects the reservoir to the main body of the cell. The heating wire for the core of the furnace is illustrated in Figure 4, but not in Figure 2.

A moveable core of the furnace was employed as indicated in Figure 4 to provide extra heat in the region near the silicon windows. This short, moveable core is made of aluminum square-stock similar to that of the stationary core. After the cell has been put in place inside of the stationary core of the furnace, an optical assembly that contains mirrors M3 and M5 is fastened in place and the moveable core is installed. Holes cut in the two sides of the moveable core allow the light to pass through to mirror M3 and out from mirror M5. The bottom side of the moveable core is also notched out so that it can slide over the mount for the mirrors M3 and M5.

Pieces of flat asbestos sheet, approximately 5 mm thick, are fastened to the four sides of both the stationary core and the moveable core of the furnace. In addition to providing some insulation, these asbestos plates hold the coils of heating wire firmly against the aluminum square-stock that forms the cores of the furnace. After the sample cell and the furnace have been installed and aligned, the plywood box that contains the sample cell and furnace is filled with loose insulation. The two pieces of glass tubing shown in Figure 1 keep the insulation from interrupting the monitoring beam as it enters and exits from the sample cell and furnace assembly. When the reservoir is to be maintained at a low temperature in order to reduce the amount of H_2SO_4 vapor in the cell, the insulation is removed from around the reservoir. With the insulation removed from around the reservoir and the remainder of the sample heated to approximately 200°C, the liquid H_2SO_4 in the reservoir is typically at approximately 45°C.

The heating wires are connected in five separate circuits, and each is controlled independently in order to maintain the temperature of the different components as desired. One heater circuit includes the wire around the stationary core, one includes the wire around the moveable core, and a third is connected to the moveable block that supports the mounts for mirrors M3 and M5. The fourth heater circuit is shown in the lower panel of Figure 2 and provides heat to the liquid H_2SO_4 reservoir. The fifth heater circuit is not shown in any of the figures; it includes a few coils of wire around the glass tubing that connects the reservoir to the sample cell and a few coils around the three glass valves and the tubing that connects them. The power in each circuit is manually controlled by a variable transformer. Approximately 130 watts are required to maintain the sample cell at 210°C.

Figure 2 shows the location of five thermocouples attached to the sample cell. By carefully adjusting the power to each of the individual heaters, it is possible to maintain the temperatures indicated by thermocouples 2, 3 and 4 to within 2°C. Thermocouple No. 1 indicates the temperature of the liquid H_2SO_4

in the reservoir, which can be controlled almost independently of the other parts of the cell.

In a typical experiment the sample cell is heated to approximately 210°C with all of the insulation in place except for that around the reservoir so that its temperature remains near ambient. Under this condition, the vapor pressure of the H_2SO_4 is so low that no absorption by the vapor can be observed with the spectrometer. Therefore, a spectral curve scanned with the cell in this condition can serve as a 100% transmittance curve to be compared with the spectral curves obtained with vapor in the cell. When the heater for the reservoir and the insulation have been put in place, the liquid H_2SO_4 can be heated and stabilized in approximately 30 minutes.

After a series of spectral curves have been obtained at different vapor pressures, the insulation from around the reservoir and the heater for the reservoir are removed to allow the liquid to cool. As the liquid cools, the vapor in the cell condenses in the reservoir. The decrease in the absorption is monitored by recording the output from the synchronous amplifier with the grating monochromator adjusted to pass a wavelength of strong absorption by the vapor.

The total pressures of a few of the samples were measured by using the pressure transducer shown in Figure 1. The line to the pressure transducer and the volume bounded by the three glass valves was filled with N_2 to a pressure slightly higher than the estimated pressure in the sample cell. The valve to the cell was then opened for a few seconds to allow a small amount of N_2 to flow into the cell until the pressure in the cell was the same as that in the transducer. A high impedance recorder measured the output of the pressure-transducer, which had been calibrated previously. The volume filled with N_2 before it is let into the cell is approximately 3% of the volume of the cell; therefore, the N_2 that flows into the cell, and thus adds to the pressure, is easily accounted for. By following the method described above, the N_2 in the line served as a buffer and prevented the flow of H_2SO_4 vapor into the room-temperature pressure transducer where it would condense.

SECTION VI

RESULTS OF TRANSMISSION MEASUREMENTS

Figures 5 and 6 show representative spectral curves of transmission obtained for hot H_2SO_4 samples at different pressures. Background curves obtained with no H_2SO_4 in the sample cell contained some coarse structure due to absorption by the silicon windows, spectral variations in the transmission of the interference filter, and spectral variations in the efficiency of the grating. This structure also appeared in the original curves for the H_2SO_4 vapor. The curves in Figures 5 and 6 have been replotted on a percent transmittance scale to eliminate the structure due to variations in the background. The physical slitwidth of the spectrometer was set at 1.2 mm, which produces a spectral slitwidth that varies from approximately 2 cm^{-1} at 12 μm to 4 cm^{-1} at 8 μm. The relatively wide slits were used to smooth out the fringes produced by the 0.8 mm thick silicon windows. The windows used at the time these data were obtained did not contain antireflective coatings; thus, the optical interference between the two surfaces produced fringes that were spaced approximately 2 cm^{-1} apart.

In order to check for possible spectral structure that was not resolved by the 2 cm^{-1} - 4 cm^{-1} resolution, a few curves were scanned with spectral slitwidths that varied from approximately 0.5 cm^{-1} at 12 μm to 1 cm^{-1} at 8 μm. These data were obtained while using thicker (2 mm), anti-reflection coated windows to avoid the fringes produced by the thinner windows. Somewhat narrower slits would have been employed for these data, but the Ge:Cu detector was noisier than usual at the time so that the detector noise would have been excessive if narrower slits had been used. The spectral curves scanned with the narrow, 0.5 - 1 cm^{-1}, spectral slits appeared essentially the same as those shown in Figures 5 and 6, which were obtained with wider slits. There was only a small, expected change in the curves near the Q-branch at approximately 8.2 μm and at a few other similar places where the slope of the curve changes abruptly. The H_2SO_4 curve in Figure 7 contains the 8.2 μm Q-branch and was scanned with a spectral slitwidth of approximately 1 cm^{-1}. No new structure was observed with the narrow slits that was not also observed with the wider slits. It is probable that adjacent lines in much of the spectrum are separated by approximately 0.3 cm^{-1}; thus the structure due to individual lines could not be observed with spectral slitwidths as wide as 0.5 cm^{-1}. Known positions of absorption lines of CH_4 and NH_3 were used for wavelength calibration of the monochromator.

Scans of the spectra were continued beyond the region represented in Figures 5 and 6 to approximately 7 μm. The region between 7 μm and 7.8 μm has not been included in the figures because the curves were difficult to interpret quantitatively. The background curve (100% transmittance) sloped steeply downward from 7 μm to 7.8 μm because of the decreasing efficiency of the grating toward shorter wavelengths. In addition, the 7 - 7.8 μm spectrum shows much structure due to H_2O in the optical path. The extra effort that would have been required to obtain quantitative data in the 7 - 7.8 μm was not considered worthwhile in the present study because the strong H_2O absorption renders this region worthless for monitoring H_2SO_4.

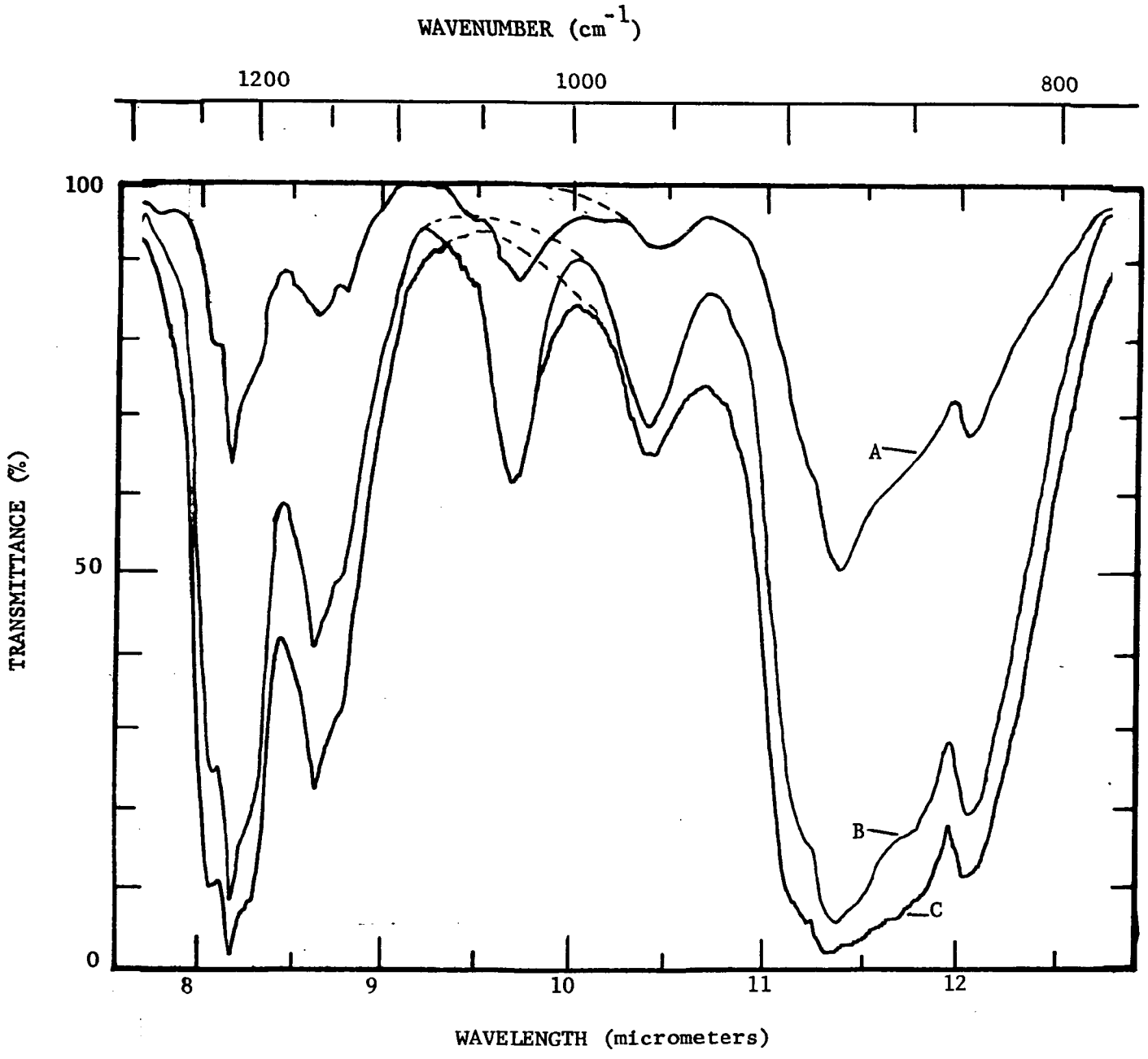


Figure 5. Spectral curves of transmittance for three samples of H_2SO_4. Path length = 52 cm. Vapor temperature = 235°C. The spectral slitwidth varies from approximately 4 cm^{-1} at 8 μm to 2 cm^{-1} at 12 μm. Approximate pressures of the H_2SO_4 vapor are: A, 3 torr; B, 15 torr; and C, 28 torr.

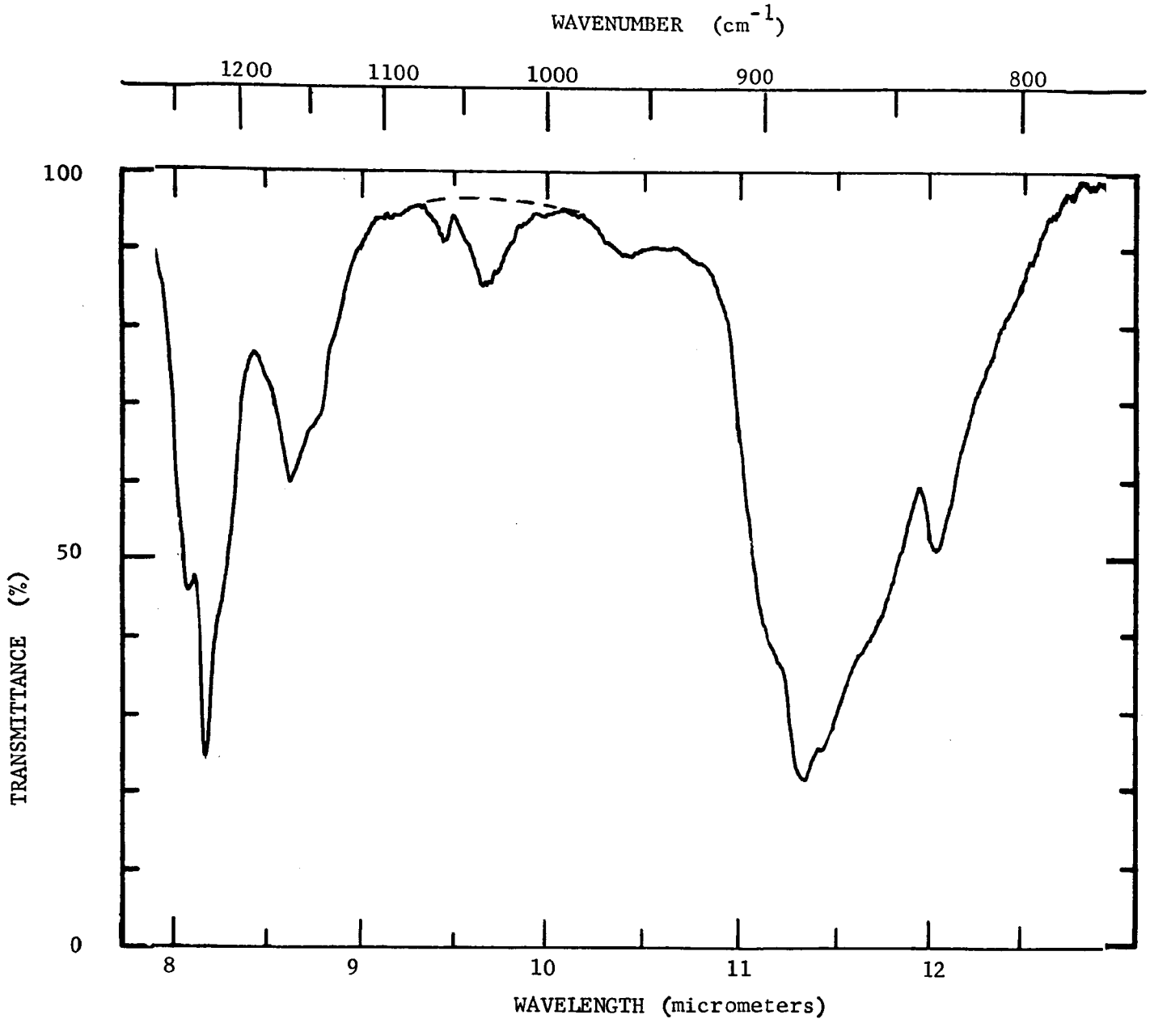


Figure 6. Spectrum of H_2SO_4 vapor. Path length = 52 cm. Vapor temperature = 214°C. H_2SO_4 vapor pressure ≅ 7 torr. Spectral slitwidth varies from approximately 4 cm^{-1} at 8 μm to 2 cm^{-1} at 12 μm.

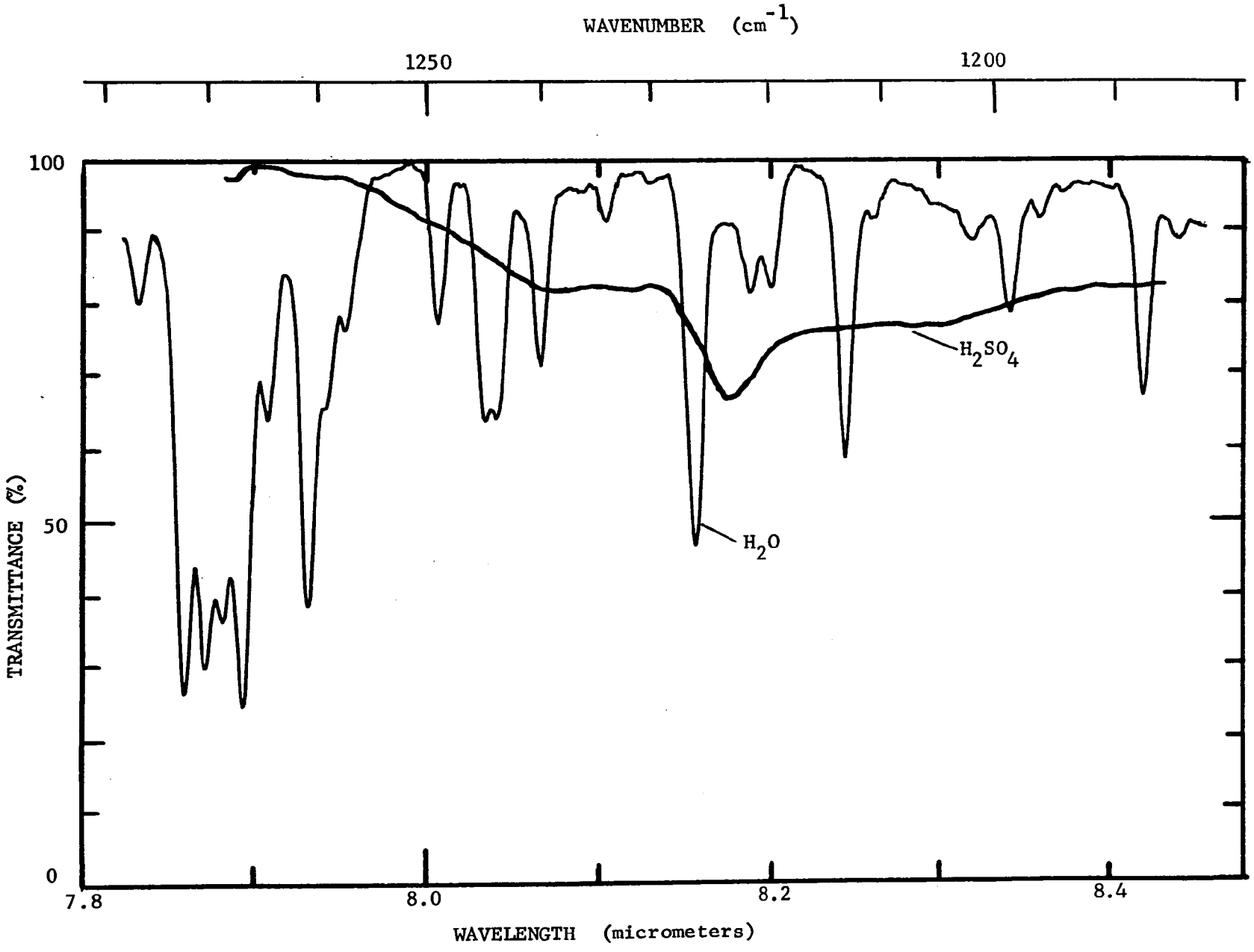


Figure 7. Spectra of H_2SO_4 and H_2O between 7.85 μm and 8.45 μm. The spectral slitwidth is approximately 1 cm^{-1}. The optical path length is 52 cm. The approximate pressures of the gases are: H_2SO_4, 2 torr; and H_2O, 600 torr.

The 7 - 7.8 μm region contains a very strong absorption band of SO_3 centered near 7.4 μm in addition to a portion of the 6.90 μm H_2SO_4 band discussed in Reference 2. Strong absorption by SO_2 also occurs between 7.2 μm and 7.5 μm. Although this region is difficult to interpret accurately, it does provide information on the maximun amount of SO_3 in the samples. For example, sample C, which is represented in Figure 5, produced approximately 80% absorptance near 7.3 μm. From unpublished SO_3 data obtained previously in our laboratory, we estimated that less than 1 torr of SO_3 would be required to produce this absorptance. Some of this absorption is undoubtedly due to H_2SO_4, and some may be due to SO_2. Therefore, we conclude that the partial pressure of SO_3 was much lower than the H_2SO_4 partial pressure in the samples studied.

The H_2O in the approximately 4-meter optical path outside of the sample cell produced enough absorption in the background curve that the concentration of H_2O in the sample cell could not be determined from the spectra. Little or no difference in the H_2O absorption was observed between the sample spectra and the corresponding background curves.

Using data from the JANNAF Thermochemical Tables[(5)] on the equilibrium constants for the gases involved, we estimated the partial pressures of H_2O, SO_3, and H_2SO_4 vapors over 98.3% H_2SO_4 liquid at the experimental temperatures. The calculated total pressures were typically 2-4 times as high as those measured with the pressure transducer. Although the large discrepancy between the measured and calculated pressures has not been completely resolved, we believe that most of it is due to erroneously high temperature measurements of the coolest place in the cell, which determines the vapor pressure. It is possible that the thermocouple on the liquid H_2SO_4 reservoir was not in good thermal contact with the liquid and was maintained hotter than the liquid because of heat from reservoir heater. The top portion of the reservoir may also have been even cooler than the liquid. The comparison between the calculated and measured pressures suggest that the temperature indicated by the thermocouple attached to the liquid reservoir may have been as much as 20°C too high.

We assumed a liquid H_2SO_4 temperature that corresponded to a calculated total vapor pressure equal to that measured with the pressure transducer. The measured temperature of the main body of the sample cell was then used to calculate the partial pressures of each of the three gas species for this total pressure. At the sample cell pressures used, H_2SO_4 accounted for approximately 50% of the total pressure. Thus, the H_2SO_4 pressures listed for the samples represented in Figures 5 and 6 are equal to half of the measured total pressure. The calculated pressure of SO_3 was only a few percent of the total pressure, a result that agrees with the spectroscopic measurements near 7.3 μm.

The primary objective has been to obtain spectral data on the absorption by an amount of H_2SO_4 vapor that is reasonably well known. Therefore, little effort has been devoted to investigating the equilibrium conditions. Insufficient time was available to investigate fully the discrepancy between the measured and calculated pressures. Impurities, including excess H_2O, in the sample cell may have

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[5] JANNAF Thermochemical Tables, Dow Chemical Co. 1967, plus Addenda to date.

contributed slightly to the discrepancy by shifting the SO_3-H_2SO_4 equilibrium. The thermal data given in Reference 5 are believed to be reliable.

The build-up and decay of impurities in the sample cell was monitored by observing the infrared absorption. The impurities appeared in two different forms. Some of the impurities appeared as a deposit on the windows and on the gold-coated mirror and remained when the cell was evacuated. Other impurities were mixed with the H_2SO_4 vapor and disappeared with the vapor when the cell was evacuated. The spectra of the impurities varied in shape from day to day, indicating that the same species of gases were not present all of the time. Certain identifiable spectral features were present on some days but not on others. After the sample cell had been cleaned, reassembled, and heated to approximately 210 - 220°C, there was little or no spectral evidence of impurities until the liquid H_2SO_4 reservoir was heated to produce H_2SO_4 vapor. Most of the impurities that absorbed significantly were apparently formed by a reaction with the hot H_2SO_4 vapor.

On several occasions just after spectra of the H_2SO_4 vapor had been scanned, the liquid H_2SO_4 reservoir was cooled so that the vapor would condense. Spectra scanned after the vapor had condensed showed little, or no, absorption that would be attributed to the vapor. Some of the absorption attributed to gaseous impurities remained after the cooling, but this disappeared when the cell was evacuated. Part of the absorption attributed to impurities also disappeared when the reservoir was cooled, indicating that some of the impurities condensed or that their presence was dependent on the presence of H_2SO_4 vapor.

The absorption feature apparent in Figures 5 and 6 that occurs between approximately 9.4 μm and 10.1 μm is due primarily to a gaseous impurity that was present in even the cleanest samples. The amount of this absorption varied relative to the amount of H_2SO_4 vapor from one sample to another. For example, curves B and C of Figure 5 both show approximately the same amount of absorption at 9.7 μm although there is much more H_2SO_4 vapor in sample C than in sample B. The shape of the curve in Figure 6 is also seen to be different from the curves in Figure 5 between 9.4 μm and 10.1 μm. Thus, the impurities producing this absorption were apparently different on the two different days when the spectra were scanned. The broken lines in Figures 5 and 6 indicate the estimated spectrum for H_2SO_4 vapor with no impurities.

The feature with the transmission minimum near 10.4 μm is probably due to H_2SO_4 and to some impurity. The absorption in this region appeared to not be completely correlated with the H_2SO_4 pressure, but more so than the absorption near 9.7 μm. Little special effort was directed toward isolating and identifying the weak absorption by impurities. The emphasis was on the investigation of strong H_2SO_4 absorption in spectral regions that might be useful for monitoring the H_2SO_4 concentration.

The hot H_2SO_4 vapor apparently reacted with the anti-reflective coatings and produced a substance that absorbs strongly between approximately 8 μm and 10 μm. This absorbing substance could not be wiped or washed from the windows, and the original anti-reflective coatings appeared to be decomposed. After only a few minutes of operation with hot H_2SO_4 vapor in the cell, the absorption between

8 μm and 10 μm could be seen to increase. This changing absorption made it impractical to obtain quantitative absorption data for the 8.2 μm band with the anti-reflection-coated windows in place. The substance deposited on the windows also absorbed slightly in the region of the 11.4 μm band, but much less than in the region of the shorter wavelength band. This substance accounts for some of the apparent absorption by H_2SO_4 illustrated by the curve in Figure 7. The contribution by this substance is slight for wavelengths shorter than 8.1 μm, but it increases for longer wavelengths.

Figure 7 contains a spectrum of hot H_2SO_4 vapor superimposed on a spectrum of H_2O vapor and illustrates the problem of H_2O interference if the 8.2 μm band of H_2SO_4 is used. Good sensitivity to H_2SO_4 could be obtained by an instrument that compares the transmission in two 4 cm^{-1} wide intervals, one centered near 1223 cm-1 and the other centered near 1233 cm^{-1}. The lower wavenumber interval contains strong absorption and is within the Q-branch. The 1233 cm^{-1} interval contains weaker absorption, yet it is close enough to the other interval that the instrument zero-setting would be relatively insensitive to changes in source temperature, spectral responsivity of the detector, etc. Unfortunately, strong absorption by H_2O coincides with the Q-branch of H_2SO_4. Therefore, an instrument that compares these two intervals would also be sensitive to H_2O vapor because the H_2O absorption is much stronger in one interval than in the other.

For best results, the H_2SO_4 absorption should be quite different in the two intervals being compared, and the H_2O absorption should be weak and approximately the same in both intervals. If the interval centered near 1233 cm^{-1} were to be used, the amount of H_2O interference could be reduced by selecting the other interval such that the H_2O absorption is nearly the same in both intervals. A 4 cm^{-1} wide interval centered near 1253 cm^{-1} might satisfy this requirement for the interval of weak H_2SO_4 absorption. The accuracy with which H_2O interference can be accounted for in this manner is limited because the relative amounts of absorption in the two intervals are usually dependent on the amount of H_2O and on the gas temperature. Thus, the intervals may be chosen so that the H_2O absorption is matched for one condition, but it may not be matched for other temperatures or other amounts of H_2O vapor. If an H_2SO_4 monitor is to be designed for gases in which the H_2O vapor concentration is quite high, it would probably be best to choose two intervals containing weak H_2O absorption, even if the difference in the absorption by H_2SO_4 in the two intervals is somewhat less than in other pairs of intervals that contain strong H_2O absorption.

Of course, the maximum sensitivity can be achieved by one interval where the H_2SO_4 absorption is the strongest and the other outside of the band where the absorption is negligible. Such a wide separation of the two intervals generally increases interference by other gases, by dirt on windows, or by particulate matter in the sample. The transmittance of particulate matter suspended in the gas or dirt that accumulates on windows usually changes slowly with changing wavelength. Therefore, interference by either of these two types of matter is usually minimized by employing two closely-spaced intervals.

A few preliminary data on CO_2 absorption were obtained in the spectral region between 10 μm and 12 μm. Absorption by CO_2 is negligible near the 8.2 μm band. The sample cell used for H_2SO_4 was filled to 1 atm with pure CO_2 while the cell was heated to 205°C. At the absorption minimum near 11.3 μm, this CO_2 sample absorbed very slightly, but less than 0.5%. Near 11.0 μm and 11.9 μm on either side of the minimum, the absorption had increased to approximately 2% and continued to increase toward both longer and shorter wavelengths. These data on CO_2 absorption have not been included because the absorption in the region of primary interest by the largest sample is too small to illustrate the potential interference. In order to show the nature of the CO_2 absorption, we have included in Figure 8 a spectrum from a report previously published by us.[6] Superimposed on the CO_2 spectrum for comparison is a portion of the H_2SO_4 curve A from Figure 5. The CO_2 sample represented in Figure 8 was at room temperature and consisted of 0.30 atm of pure CO_2 with an optical path length of 1185 meters. This amount of CO_2 is much greater than would probably ever be encountered in the effluent from a stationary source. However, the absorption in this spectral region by a given amount of CO_2 increases rapidly with increasing temperature. As a result, a few atm meters of CO_2 in a 200°C stack might produce enough absorption to interfere with an H_2SO_4 measurement that made use of the 11.4 μm band. By measuring the gas temperature and the CO_2 concentration, the interference could probably be accounted for.

Other gases that might interfere with an instrument using the 11.4 μm band, depending on the type of pollution source being monitored, are ammonia and ethylene. The absorption by H_2SO_4 does not change rapidly with wavelength at any place in the 11.4 μm band. Therefore, any two narrow intervals within the band that are compared to measure the H_2SO_4 concentration must be well-separated and be subject to the above mentioned disadvantages of instability and interference.

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6. Burch, D. E. "Semi-Annual Technical Report Investigation of the Absorption of Infrared Radiation by Atmospheric Gases." Prepared by Philco-Ford Corporation, Report No. U-4784, for ARPA, Air Force Cambridge Research Laboratories under Contract No. F19628-69-C-0263, 31 January 1970.

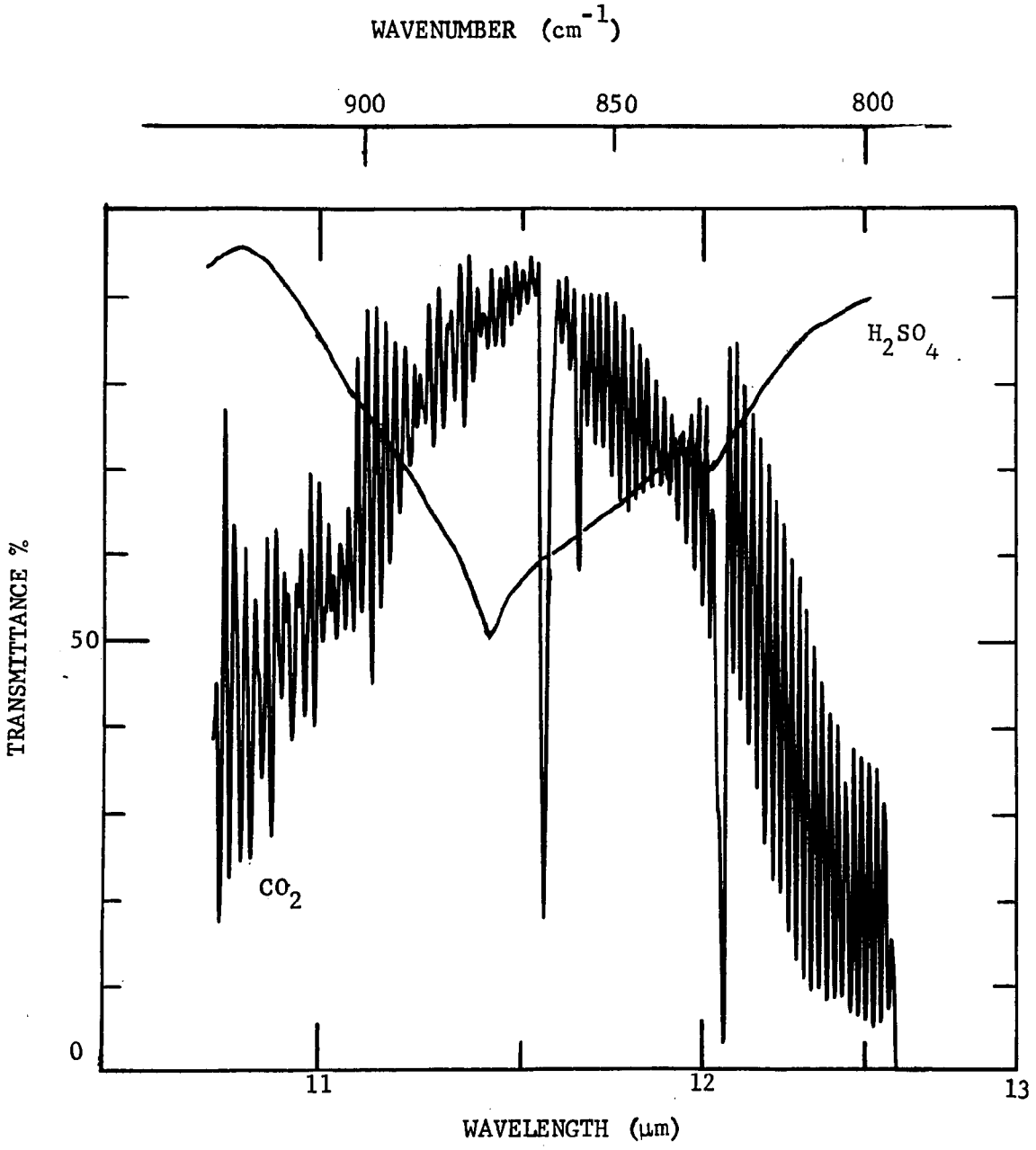

Figure 8. Spectra of H_2SO_4 and CO_2 between 11 μm and 12.5 μm.

TECHNICAL REPORT DATA
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16. ABSTRACT

A sample cell has been designed and built to contain hot H_2SO_4 vapor for spectroscopic analysis. The cell is heated to between 190° and 250°C by an electric furnace designed specifically for use with the sample cell. A small bulb connected to the main compartment of the sample cell contains liquid H_2SO_4. The partial pressure of the H_2SO_4 vapor is controlled by regulating the temperature of the liquid H_2SO_4, which is at lower temperature than any other part of the cell that is in contact with the vapor. Transmission curves have been scanned for a variety of samples over the 7.8 μm - 12.8 μm region with spectral slitwidths between 0.5 cm^{-1} and 4 cm^{-1}. At wavelengths of maximum absorption near 8.2 μm and 11.4 μm, the absorption coefficient is approximately 0.0004 (ppm - meters)$^{-1}$.

17. KEY WORDS AND DOCUMENT ANALYSIS

a. DESCRIPTORS	b. IDENTIFIERS/OPEN ENDED TERMS	c. COSATI Field/Group
*Air pollution		13B
*Sulfuric acid		07B
*Sulfur trioxide		20C
*Sulfur dioxide		14B
*Electromagnetic absorption		
*Infrared spectroscopy		

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