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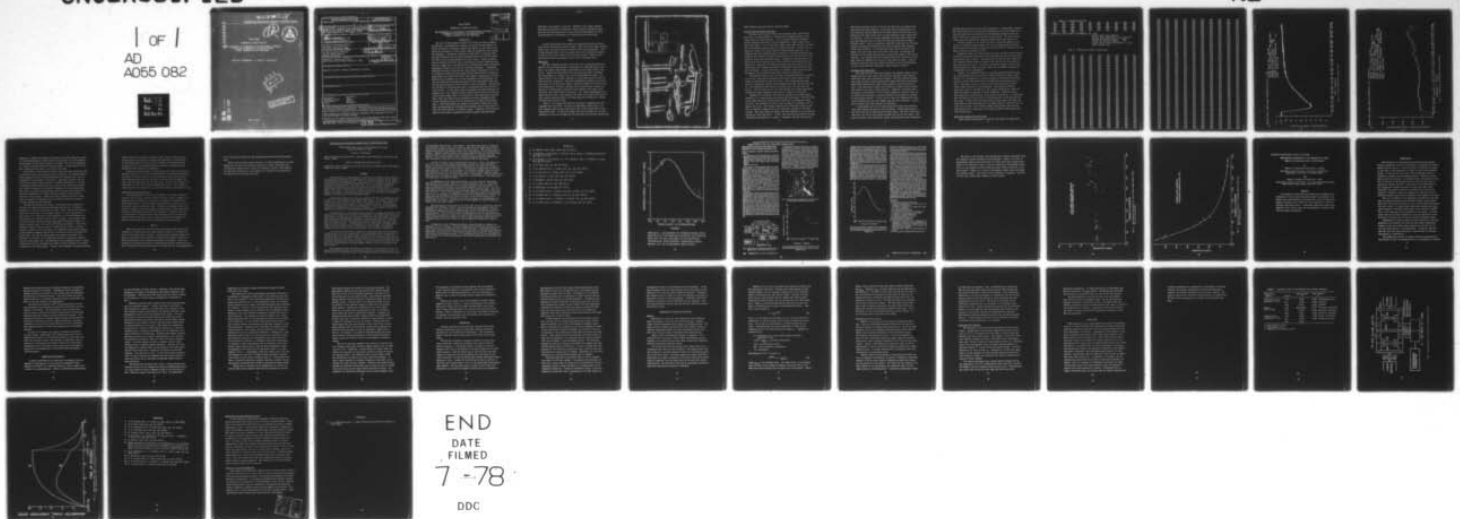
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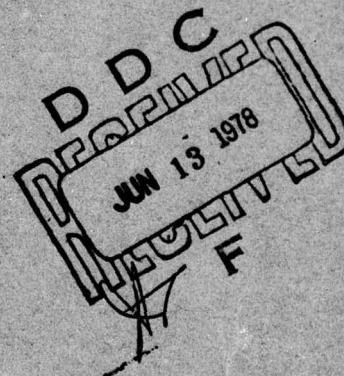


FINAL REPORT

CONTRACT NO. DAAD07-75-C-0070

DEVELOPMENT AND APPLICATION OF NEW EXPERIMENTAL LABORATORY
TECHNIQUES AND EQUIPMENTS FOR MEASURING LASER
ENERGY ATTENUATION IN THE ATMOSPHERE

PRINCIPAL INVESTIGATOR: Dr. Stuart A. Schleusener



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20. ABSTRACT (Continue on reverse side if necessary and identify by block number) An open walled White-cell system was constructed and tested for application to particulate attenuation measurements. Particle sizing and generating apparatus were included in the system as was a minicomputer based data acquisition system. Spectrophones were developed to measure atmospheric dust absorption using the KBr pellet method of sample handling. Results of these efforts were presented in one presentation and two open literature articles, reprints of which are included in this report.		

FINAL REPORT

CONTRACT NO. DAAD07-75-C-0070

THE DEVELOPMENT AND APPLICATION OF NEW EXPERIMENTAL LABORATORY TECHNIQUES AND EQUIPMENTS FOR MEASURING LASER ENERGY ATTENUATION IN THE ATMOSPHERE

INTRODUCTION

The use of electromagnetic energy by the Army for weapon guidance, surveillance, countermeasures, ranging, and remote sensing requires detailed information concerning molecular gas absorption and aerosol scattering and absorption of laser energy along the propagation path through the atmosphere. Substantial progress has been made in obtaining molecular gas absorption data by using folded-path White-type optical systems the last 8 years and spectrophones the last 3 years. However, measurements of aerosol extinction and in particular the aerosol absorption portion have lagged due to the difficulties of developing suitable laboratory measurement systems as was done for molecular absorption. In addition, molecular absorption research still needs further refinements due to lower absorption coefficient data needed and the large amounts of expensive gases needed for large volume White cell testing. Measurement of aerosol absorption and scattering in the open atmosphere are also desirable but have not been easily accomplished because of the long optical paths previously required and the lack of control of the environment. Nevertheless, the necessity of obtaining more data is increasing along with the use of laser devices. In some cases, aerosol absorption can assume dominant importance due to the unwanted thermal blooming effect which can be encountered by high energy lasers in an aerosol or other absorbing environment. Also, other real needs for more quantitative aerosol absorption coefficient information come from the varied nature of battlefield environments. These environments may contain dust, debris, smoke, soot, fogs, rain, and camouflage and countermeasure aerosols, any of which could inhibit the effectiveness of strategic devices. Therefore, while some progress has been made in measuring atmospheric gaseous and particulate

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absorption, much remains to be done. Therefore, this report presents some data and progress on White-cell usage in aerosol extinction tests and more data and conclusions on using a pellet-spectrophone for determining absorption coefficients for several particulate materials.

TASK I

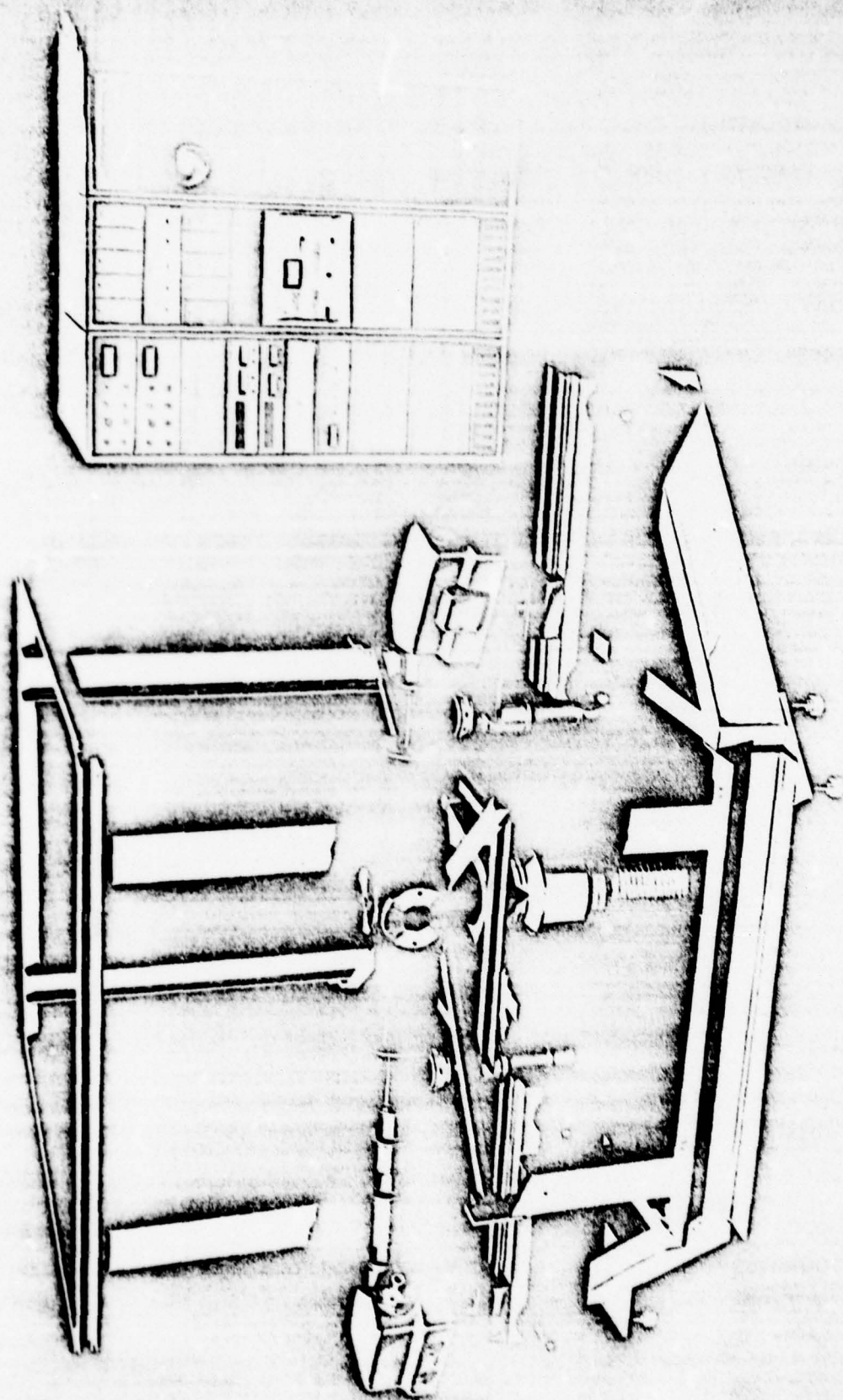
An open walled White-cell system was constructed and given initial tests during the previous contract period. Half of the just ended contract period was to have been spent on further testing and measurements. Part of this proposed effort was not completed due to an important development in Task II that took at least $3/4$ of the total effort. However, what was attempted and accomplished on Task I will be presented here.

Background

The main proposed approach to have been used in measuring laser propagation losses through aerosol environments in a White-type optics cell was to use layered sedimentation techniques for the larger sizes of particles. Layered settling starts with the particulate matter dispersed and concentrated in a layer at the top of a sedimentation volume. The layer thickness must be small compared to the distance below at which the amount settled is measured. This type of dispersion results in a minimum disturbance of the White-cell optical conditions between the mirrors, minimum contamination of mirror surfaces, and by using Stokes' settling law results in a determination of particle size present in the beam at any time after the initial layered dispersion. The layered sedimentation procedure has been used to help checkout the White-cell system itself and initial results were presented in the Final Report for Contract No. DAAD07-74-C-0091 dated March 6, 1975.

Figure 1 is a reproduced sketch of the system configuration as contemplated for use by NASA. The frame is reasonably visible and is the most important part of the picture since that is the portion that is presently being used. In White cell usage, the mirrors replace the two monochromators on the end carriages and the multi-path test section replaces the

DYNAMIC SPECTROSCOPY LABORATORY



MSFC-72-S-E-AERO-104

Fig. 1. NASA frame.

NASA diffusion pump and aluminum cylinders shown.

General Photonics YAG 15-TWO Laser

Previous tests using the White cell has always used a He-Ne laser. Since the U.S. Army primarily is interested in neodymium lasers, a new laser source had been procured. It is a General Photonics YAG Model 15-TWO neodymium laser and it was incorporated as a laser source in the White cell system. It is CW and has a TEM₀₀ mode output of about 1.5 watts at 1.06 micrometers and as much as 5-6 watts multimode. It was first operated a-c while a d-c power supply was being designed and built. To propagate the laser output through the White cell involved design and construction of a support structure large enough to hold the YAG laser, He-Ne alignment laser, and associated optics. Alignment is accomplished by reflecting the He-Ne beam back onto itself from the cavity mirrors and neodymium rod. This type of alignment procedure normally results in a close coincidence of the transmitted portion of the He-Ne beam and the YAG output when lasing. A close coincidence is required to give visual aid when propagating the YAG beam back and forth through the White cell.

The cooling system for the YAG laser is somewhat complex and consists of a filter, pump, and an automobile transmission cooler radiator submerged in the laboratory sink for cooling. Some difficulty was experienced in keeping the coolant flow temperature down when the YAG laser was on. The circulating fluid used was deionized water for corrosive protection. The YAG laser had to be turned off when the coolant temperature exceeded 85 degrees Fahrenheit after about 10 minutes of operation.

Operation of the Nd:YAG laser from a-c results in an output ripple. Therefore, a d-c power supply was designed and constructed. Delivery times on the large capacitors exceeded three months and so substantial delays occurred. The completed d-c power supply is capable of supplying over 20 amps at 120 volts. The General Photonics YAG 15-TWO laser starts to lase with the applied voltage as low as 95 volts and a current of less than 15 amps. Even when using this supply, the beam stability is not as good as desired on a long term basis. In fact, this instability proved to be quite troublesome and difficult to improve. Increasing the size of the cavity

mode-control aperture helped to improve the stability, but output power variations were still annoying. By checking with the manufacturer, it was discovered that the particular YAG laser ordered and used has a configuration that lends itself more to higher multimode power outputs than to stability. Since the present requirements concern output stability more than anything else, it would have been more pertinent to order a different model. Such an opportunity presented itself when the YAG laser crystal was shattered during the first part of December when improper coolant flow occurred. It was determined that General Photonics could replace the broken rod with a rod curved on one end. By changing the curvature of the output coupling mirror, more stable output levels can be obtained. Such replacements were accomplished and the YAG laser is once again ready for operation at the end of this contract period. Again it should be stressed, much more effort was placed in the new developments in Task II and therefore substantial delays occurred in Task I.

YAG Laser White Cell Tests

More than 50 Nd:YAG laser path lengths were somewhat readily set up through the White cell system with good cell stability as had been observed for He-Ne laser transmission earlier. The first initial attenuation tests through a layered sedimentation glass bead medium encountered difficulties involving the laser output stability problem mentioned above. Good measurement results were difficult to obtain without further improvement in the laser output stability. Therefore, continued effort was spent on stabilization. Extra careful alignment, trial and error use of mode-control apertures, and other attempts at stabilization resulted in only partial success. At this point in time, the remaining YAG laser output instabilities were more nagging than serious as problems. At this point, further glass bead testing was undertaken.

A minicomputer was used for data taking as had been done for He-Ne laser tests during the previous contract period. Layered sedimentation was still used as the dispersion and sizing method. Two ADC's should have been used, one for an input signal reference and one for the output. The use of two ADC's would effectively negate the persistent source instability

problems that had been and were being encountered. Unfortunately, substantial difficulties were discovered in programming the two ADC method and so only the output ADC was used with the corresponding assumption that the input level didn't change much during the test period. Of course, laser output stability remained an important problem under these conditions.

The main purpose of the output ADC was to record the changes in the output in a digital format suitable for use in a larger computer system. The test data was obtained on paper tape from the H-P 2115 computer and then taken to the disc-operated H-P 2116.

During the tests, the largest particles reach the beam first and cause a fairly rapid output change due to the speed with which the concentration and particle sizes increase. The latter parts of the run have slower output variations because smaller particles are involved and their sedimenting rates are much slower. By using a particulate sample such as a normal distribution of known size glass beads, the attenuation (scattering) coefficient K can be determined.¹

Figure 2 is a computer printout of the ADC pretest calibration data and test measurements versus time for a 20-30 micron glass beads sample. Figure 3 is a photograph of a computer display of test signal normalized intensity versus time while Figure 4 contains the main desired test results. The source used was, of course, the General Photonics Nd:YAG 15-TWO laser emitting at 1.06 micrometers. Note that the plotted k values are closer to one than two. Two would be expected for large non-absorbing particles compared to source wavelength. Repeating these tests again gave results closer or even near a value of one. It would seem that the White cell optics could be refocussing some of the near diffracted energy. Future tests like these using the more stable YAG laser configuration and varying apertures at the output detector position should unravel this problem. If no further layered sedimentation tests are run, restrictive detector apertures will be used in order to avoid these possible difficulties.

White Cell Uniform-Dispersion Tests

While layered sedimentation is and has been useful for system test

MODE 11 CALIBRATION DATA

391	390	389	388	390	388	391	390	390	388
389	390	391	389	391	388	391	391	391	389
388	388	390	388	390	390	389	391	391	388
390	391	389	390	388	388	390	388	388	390
389	390	389	388	390	391	389	390	391	388
390	388	389	390						

WHITE CELL LASER BEAM TRANSMISSION VS. TIME

CALIB. DATA MEAN: 390.00
SAMPLE TIME INCREMENT: 100 MILLISEC.
NUMBER OF SAMPLES TAKEN: 1200
NUMBER OF DELAY SAMPLES: 10
CALIB. DATA STAND. DEVIATION: 1.24
MODE: SINGLE ADC

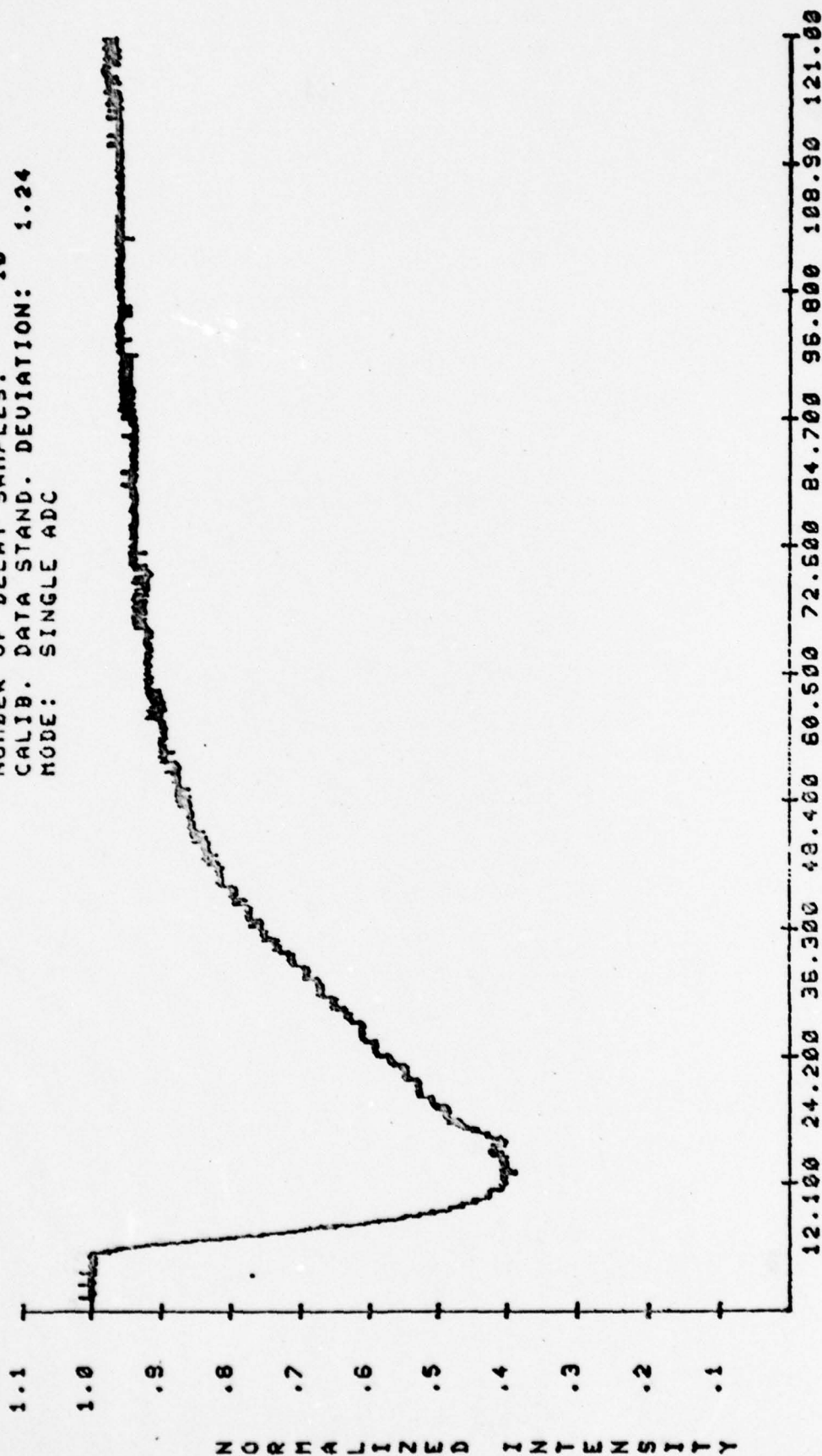
Fig. 2. Transmission printout versus time.

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214	212	205	205	196	199	191	185	188	188
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372	374	373	375	375	374	372	373	375	373
375	372	374	372	373	372	375	373	372	375
372	373	373	375	375	375	373	372	374	373
375	372	374	374	372	374	373	374	372	375

WHITE CELL LASER BEAM TRANSMISSION VS. TIME

CALIB. DATA MEAN: 390.00
 SAMPLE TIME INCREMENT: 100 MILLISEC.
 NUMBER OF SAMPLES TAKEN: 1200
 NUMBER OF DELAY SAMPLES: 10
 CALIB. DATA STAND. DEVIATION: 1.24
 MODE: SINGLE ADC



TIME - SECONDS

Fig. 3. Source transmission versus time.

WHITE CELL LASER BEAM ATTENUATION VS. DIAMETER

CALIB. DATA MEAN: 390.00
 SAMPLE TIME INCREMENT: 100 MILLISEC.
 NUMBER OF SAMPLES TAKEN: 1200
 CALIB. DATA STAND. DEVIATION: 1.24
 STAND. DEVIATION OF DIAMETER: 10.0 MICRON
 TOTAL NUMBER OF PARTICLE PER CUBIC CM. 541
 MEAN OF PARTICLE DIAMETER: 25.0 MICRON
 MODE: SINGLE ADC

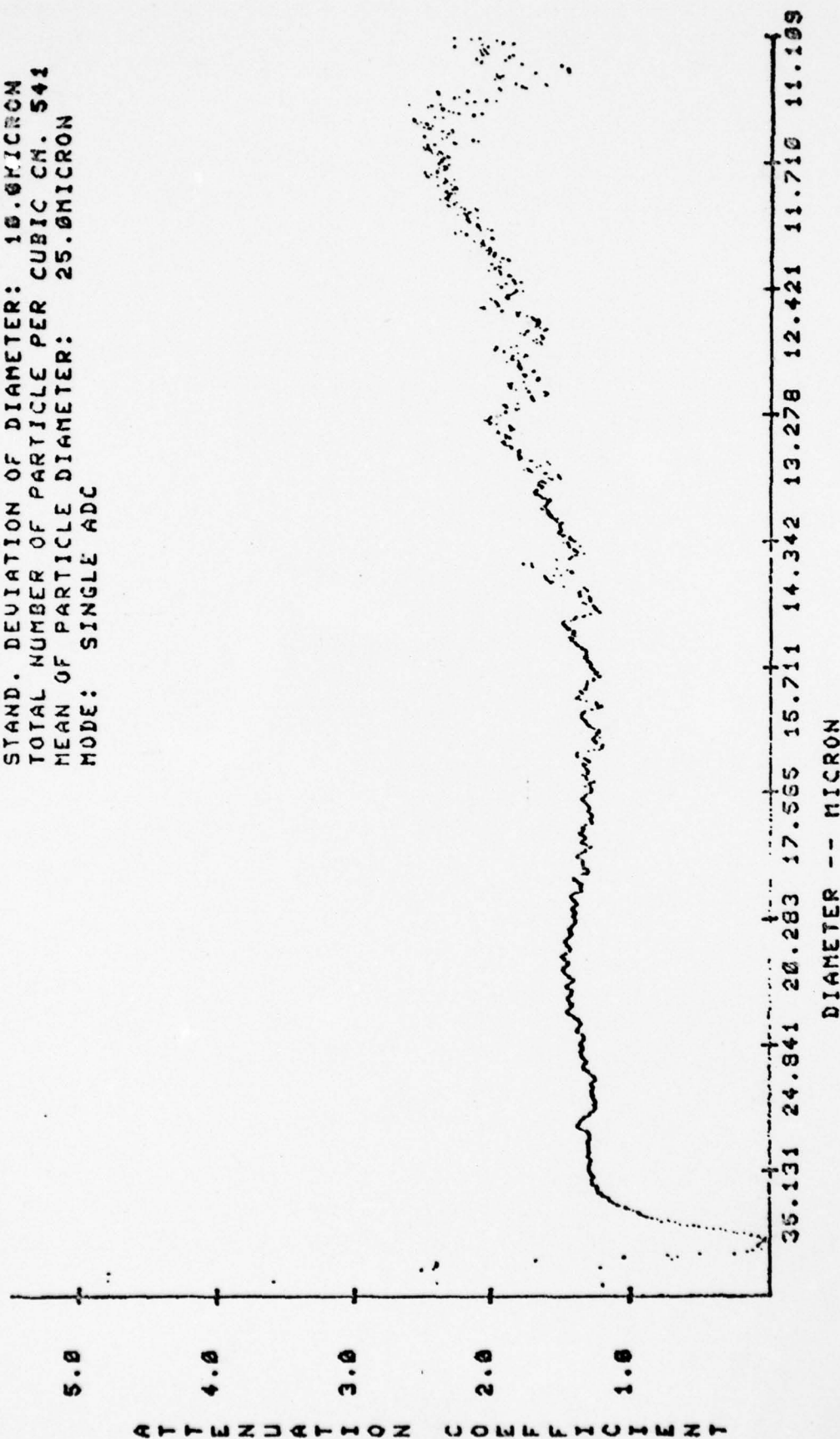


Fig. 4. Attenuation coefficient K versus diameter.

purposes, it appears that maximum usefulness can be best achieved by going to a uniformly-dispersed smaller-particle test medium. This has required a modification of the center section of the White cell and the acquisition of a state-of-the-art particle sizer and counter. A major portion of the Task I effort has been made in the uniform test area.

Modification of the White cell center section has been relatively easy to accomplish since only a masked volume is required. The layered sedimentation apparatus with the top dispersal area was removed first. Next, a rectangular box was placed in the center. Apertures were cut to let the laser beams through as had been done for layered sedimentation. However, a masking system was added which can be used to shut off the test volume from outside turbulence while particle dispersion is being accomplished. When ready, the masks are dropped away from the apertures allowing an attenuation measurement to be made as fast as possible, after which the masks are reset in place. During this time a particle sizer and counter must be operating to characterize the test medium wherein the laser attenuation measurement has been made.

For particle counting and sizing, a Knollenberg Model ASASP-300 instrument was ordered from Particle Measuring Systems, Inc. in Boulder, Colorado at a cost of \$7,500 from E.E. Dept. funds. The unit was received during the latter half of the contract period. It has a minimum detectable size of 0.1 micron and a size resolution of 0.1 micron through four size ranges up to a maximum size of about 5 microns. The instrument came in with an output both as an analog signal and a BCD digital format suitable for direct input to an in-lab H-P 5406B data processor. Much of the graduate student's time was spent in interfacing the sizer to the mini-computer. However, this will result in an automated sizing system superior to the complete commercial \$13,000 unit, of which we could only afford the \$7,000 head. His cost has been minimal with respect to hardware, but time-wise, he has had to put substantial effort into layout and construction of circuit cards, etc. The magnitude of this interfacing work is large enough that it was not completed by 24 March 1976 and therefore has delayed initiation of the testing phase. However, once completed, the data processing

system should be an important test facet since tests of this type are only as good as the ability to measure and record what is being tested. In the future, White cell tests will be completed and reported as soon as they can fit in the research schedule depending on possible future funding and other different commitments.

Another work task related to uniform-dispersion tests was the future emphasis expected to be given to countermeasure smoke attenuation problems. In order to initiate such tests, the fume hood was moved into the basement laboratory and a decision was made to reroute the roof exhaust outlet to the fume hood and White cell test area. This has been largely accomplished on a first try basis for feasibility tests. A more sophisticated system will probably have to be used for possible toxic smoke testing.

Conclusions and Recommendations

All of the proposed tests were not completed for several important reasons. First of all, extensive particle sizing, particle counting, and data processing interfacing work has had to be done for connection to a minicomputer system. This has delayed initial testing but will prove necessary for future experimental usage. Secondly, an important development in Task II made it necessary to apply extra attention to other experiments because of immediate applicability and importance. Therefore, only now is it possible to run quantitative uniform-dispersion tests as had been proposed for this contract period. With or without future funding for Task I, some of these tests will be completed in the near future.

TASK II

White cells are useful in molecular gas absorption work but are very expensive to install and require large amounts of gases to be tested because of the large volumes involved. Spectrophones, with much smaller volumes, are currently being tested for measuring small amounts of laser radiation absorption both in gaseous and aerosol environments. Due to the importance of measuring atmospheric particulate absorption coefficients and since a modified spectrophone seems to be a useful tool for such measurements,

all of the Task II effort has been directed towards particulate absorption work.

Most of the following report material is a direct reproduction of three papers, all of which were developed during this contract period and two of which have already been presented and appeared in print. Some material has been added inbetween the three papers for continuity and presentation of results not yet formally published.

Spectrophone Measurements of the Absorption of Laser
Energy by Atmospheric Dust

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Atmospheric Sciences Laboratory, US Army Electronics Command, White Sands Missile
Range, New Mexico 88002

SUMMARY

The absorption of electromagnetic radiation, in particular laser energy, by the particulate matter in the atmosphere is an important variable in the study of atmospheric propagation effects. Therefore, the investigation of the complex refractive index of atmospheric particulate matter is a research area which is becoming increasingly more active (Ref 1-5). One of the major problems in experimental programs is to separate scattering and absorption effects. A spectrophone offers possibilities in this area since it responds only to absorbed energy (Ref 6-10). A new method to measure particulate absorption, using spectrophones, is proposed. The technique has been applied to atmospheric particulate samples in the CO₂ laser 10.6- μ m spectral region.

Several spectrophone geometries are possible with the new method, but common to them all is the handling of the particulate matter. The sample is deposited on the surface of an optical flat which may be interior to the spectrophone or serve as the front or rear window of the spectrophone chamber. The energy absorbed by the particulate matter is transferred to the gas in the spectrophone, resulting in a signal from the pressure transducer. These signals can then be related to the amount of energy absorbed by the sample.

The operation of a spectrophone system requires a chopped, reasonably strong radiant source (tenths of a watt or more depending on the absorption strength of the sample). In order to test the new technique, an absorption region was desired which had wavelength-dependent absorption. Previous work (Ref 11-12) indicated that atmospheric dust has wavelength-dependent absorption behavior in the CO₂ laser wavelength region. A tunable CO₂ laser was chosen with a variable speed chopper in the beam path. In addition to the above equipment, the experimental setup included a single chamber spectrophone, a power meter, a capacitance manometer system with a 0-1 torr sensing head, and associated electronics.

In the work reported herein, samples of atmospheric dust were distributed on the surface of a zinc selenide flat. The particulate matter covered (geometrically) about 1% of the total surface area of the window that was exposed to the laser beam. The individual particle sizes were mostly below 5 μ m in diameter with a few between 5 and 15 μ m. The particle diameters were determined by using an optical microscope to scan the surface. The zinc selenide flat with dust was used as the front window of the spectrophone chamber; the rear window was also zinc selenide.

The tunable CO₂ laser radiation was chopped at rates between 0.1 and 1 Hz, thereby alternately heating the particulates and then allowing them to cool. As the particles are irradiated, part of the absorbed energy is transferred directly

to the chamber gas and part to the window. The window part must be considered for atmospheric dust, since these particles tend not to be spherical in shape and may have substantial thermal contact with the window. Thus, some of the absorbed energy may be transferred through the window and out of the spectrophone chamber. Another problem involves the energy scattered from the particles which may be absorbed by the chamber walls and hence lead to a pressure increase. However, only a small portion of the scattered energy hitting the chamber wall will be absorbed, with most of it being reflected out of the system.

In this work three sets of measurements were made. First, the absorption due to the zinc selenide window was measured; second, with the particulate matter on the window, measurements were obtained with the sample on the inside of the spectrophone chamber; and third, the window was reversed and the measurement made with the sample on the outside. The signals obtained from the capacitance manometer with only the absorption from the zinc selenide window present were about 50 millivolts, and were independent of wavelength. With the particulate sample present, the addition signal ranged up to 150 millivolts. The laser power for these tests was about 0.4 watts.

A combination of the above measurements, to account for all the energy absorbed by the particles, results in the data plotted in Fig. 1. These data are, in principle, proportional to the energy absorbed by the dust sample. In order to qualitatively verify the wavelength dependence shown in Fig. 1, a conventional KBr pellet extinction spectrum of the same sample was made. This spectrum, containing both absorption and scattering losses, is included as the solid line in Fig. 1. It has been normalized to the spectrophone data at a wavelength of 9.6 μm . By reversing the sample from inside the spectrophone to outside, the energy transferred through the window and lost to the chamber signal can be partially accounted for. Analysis of the data in regions of low absorption indicates that the effect of scattering in the spectrophone is small and does not exceed 10% of the total signal.

The spectrophone system used in the above test has been calibrated with both nichrome wire and P(20), 10.59- μm , CO_2 line absorption measurements. The two measurements agreed fairly well. However, both of these calibration procedures are based on absorption along the spectrophone chamber axis and not just at a window surface. More tests are needed to determine if there are any differences. Spectrophone measurements similar to those described above were made for materials such as powdered quartz and amorphous carbon, whose absorption coefficients are reasonably well known (Ref 13-14), so that an empirical calibration could be made.

In addition to the calibration problem, additional work is required to optimize this new technique. The spectrophone system with which the data in Fig. 1 was obtained was designed for gaseous absorption work. Other geometries are under study, including depositing the aerosol particle on a transmitting substrate completely contained within a spectrophone chamber and the use of a differential (two chamber) spectrophone system. It is anticipated that further development of this new method of measuring atmospheric particulate absorption will produce much needed data.

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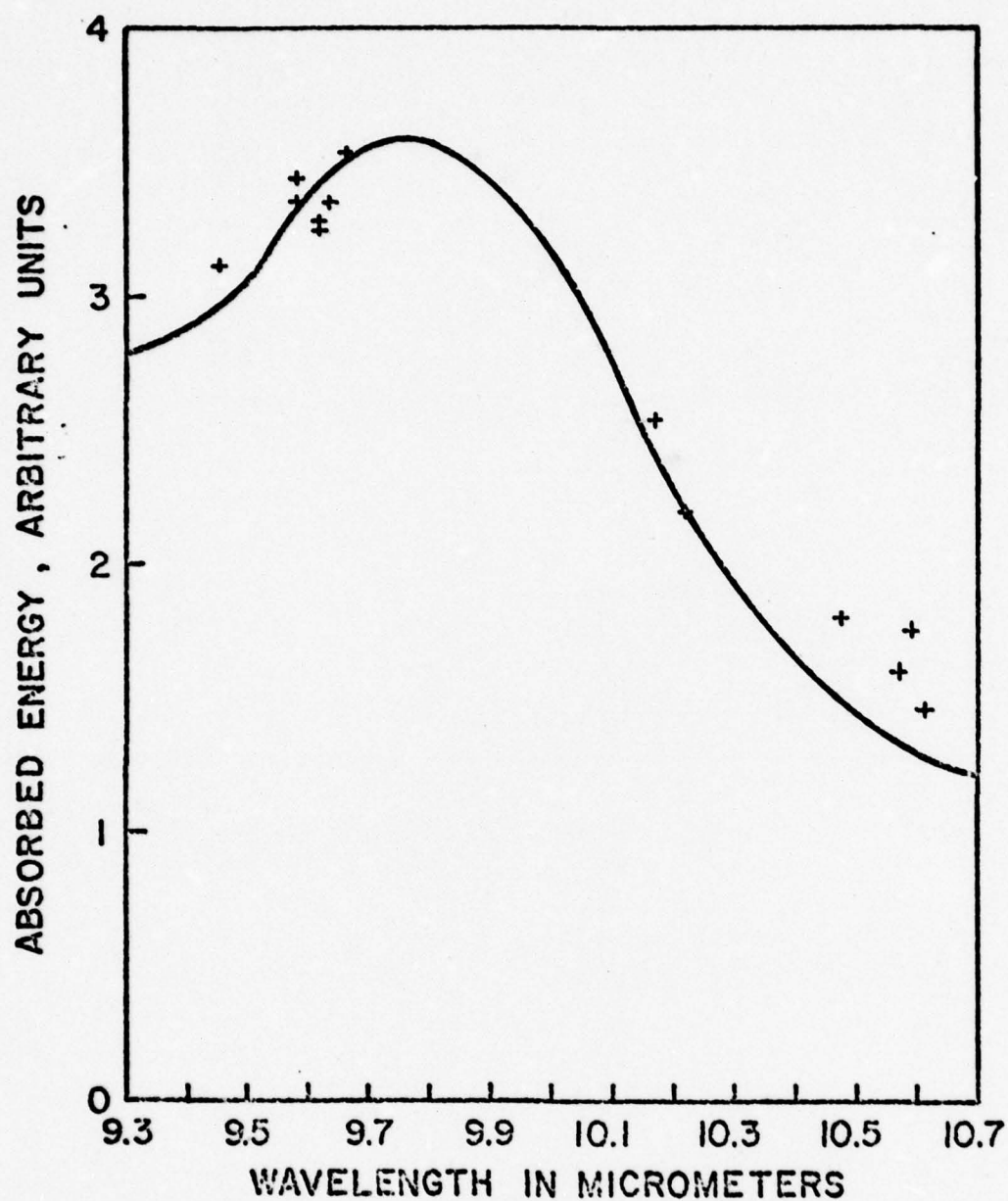


FIGURE 1

WAVELENGTH DEPENDENCE OF ABSORBED ENERGY FROM SPECTROPHONE MEASUREMENTS(SHOWN BY THE CROSSES) COMPARED TO THAT OBTAINED BY CONVENTIONAL TRANSMISSION SPECTROSCOPIC TECHNIQUES (SOLID CURVE) FOR AN ATMOSPHERIC DUST SAMPLE.

Differential spectrophone measurements of the absorption of laser energy by atmospheric dust

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The absorption of electromagnetic radiation, in particular laser energy, by the particulate matter in the atmosphere is an important variable in the study of atmospheric propagation. Therefore, the investigation of the complex refractive index of atmospheric particulate matter has become an active research area.¹⁻⁵ One of the major problems in experimental programs is to separate scattering and absorption effects. Spectrophones show promise in this area since they respond to absorbed energy.⁶⁻¹⁰ A new method of measuring atmospheric particulate absorption using a differential spectrophone system has been tested, and initial results have been obtained in the 9- μ m and 10- μ m wavelength regions.¹¹ The differential spectrophone reduces window signals and is used to obtain increased sensitivity. A tunable CO₂ laser was used as the source shown in the diagram of the test system configuration in Fig. 1.

The tunable CO₂ laser source is chopped at about 0.5 Hz. The beam passes through both chambers to a power meter. Both chambers contain nonabsorbing N₂ (at 660 Torr pressure) and 2.5-cm diam NaCl flats. The flat in the first chamber balances the absorption of the particulate substrate flat in the second chamber. Absorption by the windows, flats, and particulate sample results in energy transfers to the N₂ gas. This raises the temperature and pressure of the gas, resulting in pressure changes measurable with the capacitance manometer differential sensing head. The window and flat absorption effects approximately cancel, leaving the particulate absorption signal by itself as the only observed result. The leak valve is set (nearly closed) so it isolates both chambers from each other at the chopping rate and yet maintains steady-state pressure equilibrium.

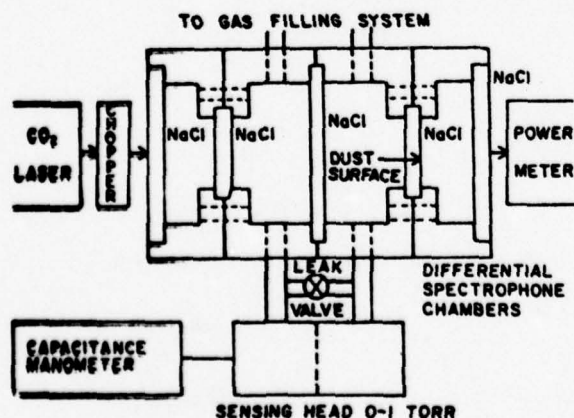


Fig. 1. Block diagram of differential spectrophone system. Atmospheric dust is on the indicated substrate surface.

The capacitance manometer head in Fig. 1 senses pressure differences between the two chambers by using the changing capacitance of a thin stressed metallic membrane as part of an electrode system. The total range of the head is 0-1 Torr. The associated electronics has multiple ranges with the most sensitive being 0.001 Torr full scale. Figure 2 is a photomicrograph of an atmospheric dust sample as it appeared when on a substrate flat. The dust was brushed onto the surface from a sample collected by forcing air through a membrane filter. Note the small amount of surface coverage, even though the signal resulting from the absorption was nearly 100 mV. Signals obtained with no particulates on the substrate flat in the second chamber are less than 5 mV. Therefore, even more dilute samples can be used in those cases where collection is a problem.



Fig. 2. Photomicrograph of atmospheric dust on NaCl flat used in second chamber of the differential spectrophone.

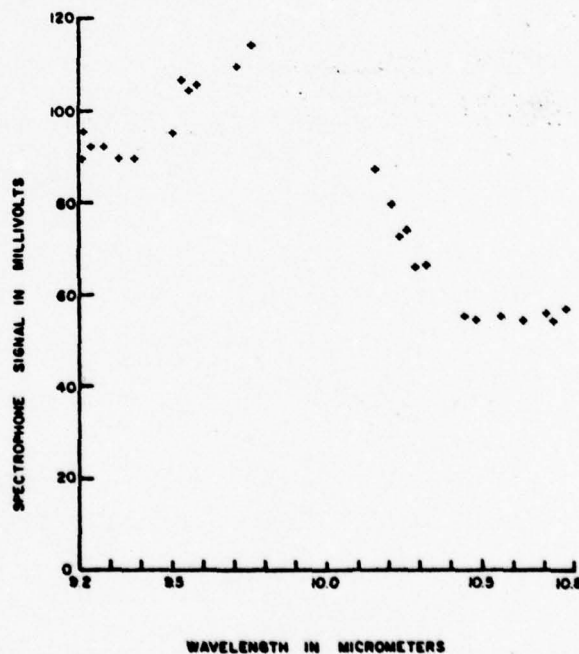


Fig. 3. Spectrophone experimental results. The crosses (+) are the signals obtained at specific CO₂ laser wavelengths for the sample shown in Fig. 2.

Figure 3 shows the results of multiwavelength CO₂ laser absorption tests on the atmospheric dust sample in Fig. 2. The extinction curve (scattering plus absorption) in Fig. 4 was obtained using part of the same parent dust sample with the potassium bromide pellet technique and an ir spectrophotometer (Perkin-Elmer model 521). This curve is similar in shape to that obtained by others^{12,13} using different dust samples. The qualitative agreement between Figs. 3 and 4 is evident. The need for a calibration process for the spectrophone-generated results is obvious, since comparison with different measurement procedures is impossible without it.

Calibration of the differential spectrophone system is possible using the known absorption of the CO₂ laser P(20) line at 10.591 μ m in CO₂ gas. Such a test in a mixture of N₂ and CO₂ in the second chamber and nonabsorbing N₂ only in the first chamber results in a signal voltage level proportional with the amount of CO₂ gas used. The known absorption coefficient is then used to calculate the absorbed power (which is normally less than can be directly measured using conventional methods). These calculations result in a sensitivity of about 1.75 mV per μ W ab-

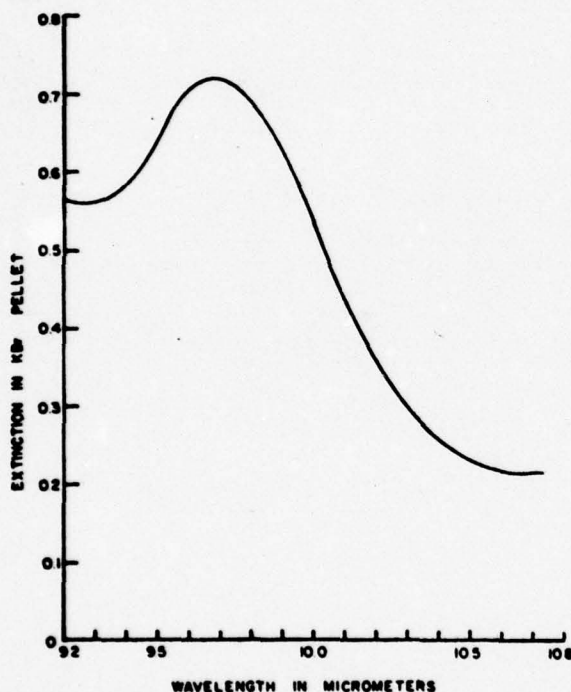


Fig. 4. Extinction curve of atmospheric dust vs wavelength obtained using the KBr pellet technique and an ir spectrophotometer. The same parent dust sample was used to obtain the results of Figs. 3 and 4.

sorbed for the differential system in Fig. 1. This number can be used to obtain the absorption coefficient of a substrate particulate sample if particle size and number density (or total mass) are known.

Measuring absorption characteristics of particulate samples on optical substrates in a differential spectrophone has several important advantages. Two of these are (1) extremely high sensitivity useful for dilute samples and low absorption coefficients and (2) the ability to separate absorption losses from scattering losses. One disadvantage concerns the heat transfer path between the particles and substrate. Substrate and window absorption equilibrium response times are as much as 20–30 sec in the system in Fig. 1. Therefore, any absorbed energy that flows from the particles to the substrate will not show up as a contribution to the signal when chopping at about 0.5 Hz. However, if the substrate heats up more than the particles due to its own absorption, any energy flow between the substrate and the particles will be from the substrate. Under these conditions the particulate energy absorption should all show up as a signal due to the much faster response times.

Another problem also surfaced when making initial tests of the system in Fig. 1. None of the NaCl components were wedges or antireflection coated, thus giving rise to varying wavelength transmission and reflection effects. These showed up during the multiwavelength testing as approximately $\pm 10\%$ absorption signal errors due to reillumination of the particles. The data in Fig. 3 represent averages of repeated tests. The above errors can be avoided by using antireflection-coated or wedge-shaped flats.

It is felt that the advantages of the above described aerosol absorption measurement method far outweigh the encountered disadvantages. However, much work remains to develop and analyze the system more fully.

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Not shown in the November 1975 Applied Optics article reproduced above are substrate results obtained on other material. Figure 5 is a plot of spectrophone signal versus wavelength for 30-40 micron glass beads distributed on the substrate surface. Note the variation in the measured points. This is a good example of the troublesome etalon effects mentioned in the above papers. Figure 6 is a plot of spectrophone signal versus wavelength for powdered quartz. Less point-to-point variation is apparent. The form of this curve matches that obtained by other methods.

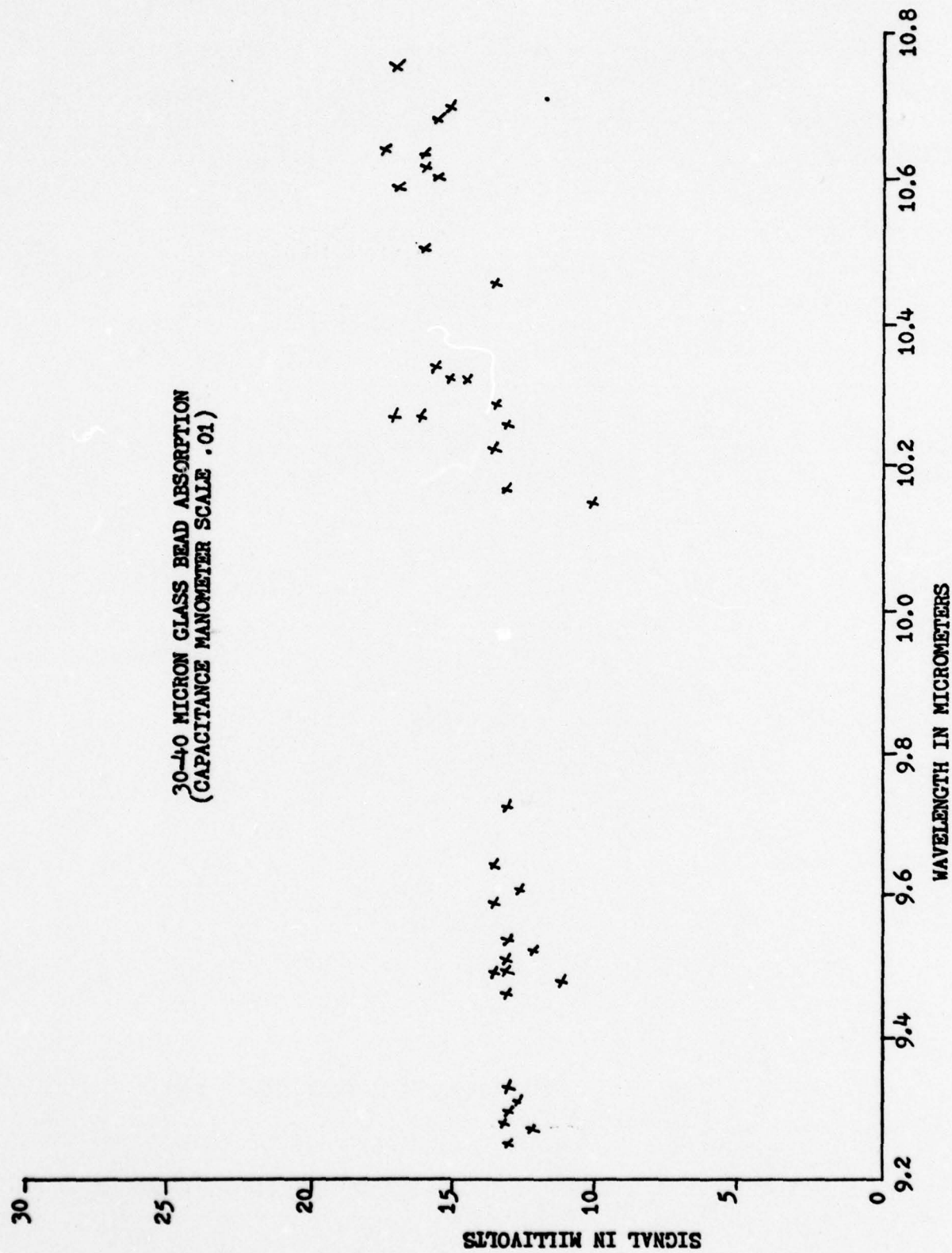


Fig. 5. Spectrophone absorption of 30-40 micron glass beads versus CO₂ laser wavelengths.

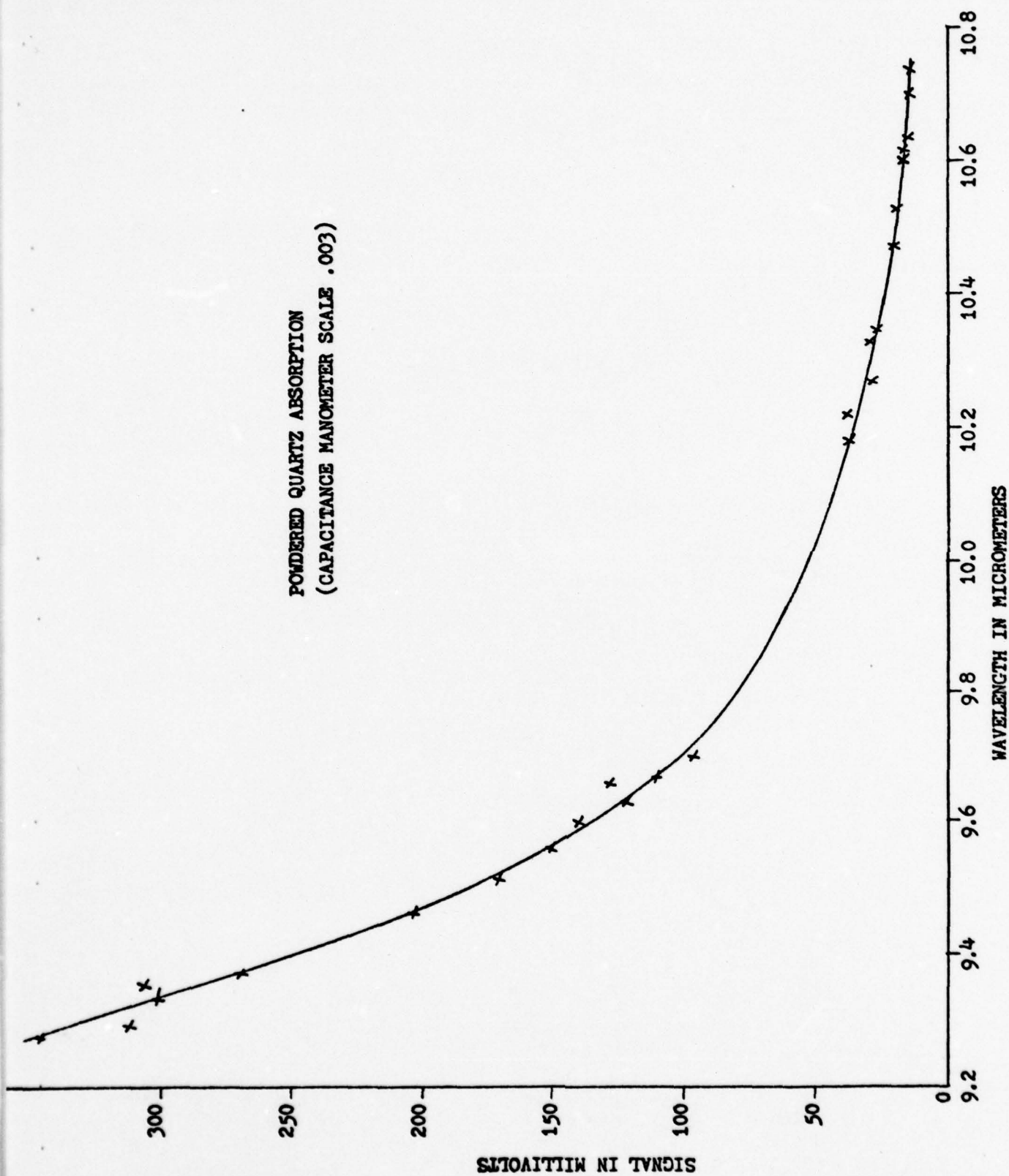


Fig. 6. Spectrophone absorption of powdered quartz versus CO_2 laser wavelengths.

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**SPECTROPHONE MEASUREMENTS OF THE ABSORPTION OF LASER
ENERGY BY ATMOSPHERIC DUST IN THE INFRARED**

by

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ABSTRACT

A new method of quantitatively measuring the absorption of atmospheric dust or other particulate matter is described. The system uses a differential-spectrophone KBr pellet technique and should ultimately prove useful from the ultraviolet to the far infrared wavelength region. Preliminary measurement data on atmospheric dust, quartz, and soot are presented at 1.06, 9.6, and 10.6 micrometer wavelengths.

INTRODUCTION

The absorption of electromagnetic radiation by particulate matter suspended in the earth's atmosphere is a quantity of considerable current interest. It must be known to calculate the effect of such dust on the propagation of laser or other electromagnetic energy. At the present time, very little quantitative data is available throughout most wavelength regions. Qualitative information about the absorption of infrared energy by atmospheric dust can be obtained by the KBr pellet method, and this has been exploited by several workers.^{1,2} Some quantitative measurements in the visible and infrared spectrum have been made using other methods,³⁻⁶ and recently Fischer has extended his work into the infrared.⁷ Therefore, while some progress has been made in measuring atmospheric particulate absorption, much remains to be done.

This is in contrast to the substantial progress that has been made in obtaining molecular gas absorption data by using folded-path White-type optical systems the last 10 years and spectrophones the last 4 years. It appears that spectrophone systems can also prove valuable in obtaining particulate matter absorption data.^{8,9} This is the technique that is presented in this paper. It should prove useful and provide quantitative imaginary refractive index data from the ultraviolet to the far infrared wavelength region.

Measurements of aerosol absorption and scattering in the open atmosphere are desirable, but have not been easily accomplished because of the long optical paths required and the lack of control and measurability of the environment. Therefore, most present day tests are being conducted in varying forms of simulated environments in laboratories.

The fundamental problem in making laboratory measurements of the refractive index of atmospheric dust is in designing a technique

whereby the scattered energy and absorbed energy can be separately and quantitatively determined. Spectrophones can be designed to respond primarily to absorbed energy, thereby enabling separation of scattered and absorbed energies. Our first system has a measured absorption to scattering sensitivity ratio of about 200, and this is more than adequate for most studies.

Our previously reported work^{8,9} using spectrophones to measure atmospheric dust absorption lacked both quantitative and calibration capabilities. The quantitative aspect has been overcome by using a measurable dilution mixing process involving high accuracy analytical balances and KBr host pellet material. The calibration problem is presently being minimized through infrared extinction measurements, nichrome wire dissipation, and CO₂ gas absorption tests at the CO₂ laser P(20) line at 10.6 micrometers. The remaining question is what final accuracy can be reasonably attained.

In view of the above, this paper is intended to basically do three things: demonstrate that a spectrophone system can be selectively sensitive to the absorbed energy in a dust sample rather than scattered energy, show how such a particulate spectrophone may be calibrated, and finally, present some preliminary measurement data on atmospheric dust at CO₂ laser wavelengths of 9.6 and 10.6 micrometers and at the Nd:YAG laser wavelength of 1.06 micrometers.

PARTICULATE SPECTROPHONE

A recent comprehensive and theoretical treatment of gaseous absorption spectrophones has been given by Rosengren¹⁰ while Bennett and Forman¹¹ have completed studies on heat transfer from a solid to a nonabsorbing gas. However, differential spectrophones

are not mentioned in either article. Therefore, some of the basic principles as related to differential spectrophone operation will be presented. Both CO_2 and Nd:YAG lasers have been used as sources in the diagram of the test system configuration illustrated in Fig. 1.

Unlike our previous tests, neither laser source has been chopped in a repetitive manner. Instead, the source is allowed to pass through both chambers to a power meter for a period of time. Both chambers contain nonabsorbing N_2 (at 660 Torr pressure, local atmospheric pressure) and 13-mm diameter KBr pellets. The pure KBr pellet in the first chamber balances the absorption of the particulate-holding KBr-mixture host pellet in the second chamber. Absorption by the windows, pellets, and particulate sample result in energy transfers to the N_2 gas. This raises the temperature and pressure of the gas in each chamber, resulting in a net particulate sample contribution measurable with the capacitance manometer differential sensing head. Through proper design, unwanted absorption effects can be made to approximately cancel, leaving the particulate absorption signal by itself as the only observed result. An offset signal can occur in systems without antireflection coated or Brewster-angle components. However, such offset signals are normally small and are caused by less power reaching the back chamber due to losses at all earlier air-to-solid surfaces. The leak-valve in Fig. 1 is set (very nearly closed) so that it nearly isolates both chambers from each other during the test time period and yet maintains steadystate pressure equilibrium otherwise.

The capacitance manometer head in Fig. 1 senses pressure differences between the two chambers by using the changing capacitance of a thin stressed metallic membrane as part of an electrode system. The total range of the head is 0-1 Torr. The associated

electronics has multiple ranges with the most sensitive being 0.001 Torr full scale.

The pressed KBr pellet spectroscopic extinction technique is a qualitative measurement method that is well known and most useful in the infrared.^{1,2} The transmission spectrum of the disk containing atmospheric dust or other particulate sample is an attenuation spectrum of the sample, since both absorption and scattering are involved. However, in the infrared where the radiation wavelength is large compared to the particle size of atmospheric dust, absorption can be the dominant attenuation mechanism that varies with wavelength. The spectrum can then be thought of as a qualitative representation of the wavelength dependence of the absorption coefficient. In the visible, the pressed pellet method is not as useful because the particles are not small compared to visible wavelengths. Scattering can then become the dominant attenuating mechanism in the pellet, and the spectrum is no longer a representation of the absorption coefficient. Additionally, somewhat dense samples have to be used in both the visible and infrared wavelength regions since appreciable losses must take place to be accurately measured in a straight transmission measurement. In comparison, since spectrophones are so sensitive, very dilute pellet sample mixtures can be used to measure absorption losses of atmospheric dust mixed into the KBr pellet shown in the back chamber in Fig. 1. Experimental tests have proven this to be true with samples as small as 10^{-5} grams of dust resulting in absorption signal-to-noise ratios of greater than 10. This requires the use of a sensitive analytical balance; otherwise, standard mixing and pressing techniques are followed in forming the sample pellet.

Energy that is absorbed by the atmospheric dust mixture inside the KBr pellet transfers from the dust particles to the KBr

host pellet material and thence to the N_2 gaseous medium. The spectrophone absorption test equilibrium time constant (source beam on) is from 15 to 60 seconds depending on the size of the KBr pellets and the leak-valve setting. Any unwanted scattered energy interacts with the system in a different manner than the absorbed energy. First of all, it is desired to allow as much as possible of the scattered energy to escape from the spectrophone chambers, thereby not contributing to the test signal. Therefore, the particulate spectrophone in Fig. 1 is constructed as short and wide as conveniently possible. Secondly, any scattered energy that strikes a highly reflecting spectrophone chamber wall will be primarily reflected through perhaps at worst a number of reflections out of the system. Thirdly, any scattered energy that does get absorbed in the walls of the spectrophone chambers does not efficiently re-enter the N_2 gaseous medium and therefore does not make an appreciable signal contribution. The results of a number of scattered energy tests show that scattered energy contribution to a developed spectrophone signal is down by a factor of 200 or more compared to absorbed energy.

Due to the long heat transfer time constants of the KBr pellet, chopping rates using rotating choppers proved to be too high, even at 0.5 Hz. Therefore, a simple off-on-off manual blade rotation test procedure is used with an on-time of about 20 seconds for 250 mg pellets. The leak-valve must be very nearly closed for satisfactory operation with such a long time constant. In fact, system operation is feasible without any leak-valve at all between the front and back chambers. However, much longer waiting periods are required between tests and the test on-time is increased to the several minute region before equilibrium occurs. The presence of a leak-valve materially aids the test procedure since

the pressures in the front and back chambers are then automatically equalized before test initiation, even in the presence of small leaks or outgassing conditions. Once the leak-valve is properly set, it can't be changed between sample and calibration tests.

Typical test result curves of capacitance manometer output signals are shown in Fig. 2. Curve A corresponds to the desired leak-valve setting with the signal strength represented by the level change occurring between the off and final equilibrium conditions. Curve B corresponds to a more open leak-valve setting and is somewhat less desirable since the maximum level change is dependent upon heat transfer rates.

CALIBRATION

Initial calibration tests have been performed using three different approaches; CO_2 gas absorption, infrared extinction measurements, and nichrome wire dissipation. Test calibration results using the three methods agree within $\pm 10\%$ at this early stage.

The CO_2 gas calibration procedure consists of using a CO_2 laser source and a known CO_2 absorption line such as P(20) at 10.59 micrometers. A CO_2 - N_2 gas mixture is placed in the back chamber while only N_2 is inserted in the front chamber at the same total pressure. Pure KBr pellets are in both the front and back chambers to keep both volume and balance test conditions as similar as possible to those encountered when making pellet-particulate absorption tests. Between 150 and 200 Torr of CO_2 is used due to the short path length (about 1.5 cm) in the particulate spectrophone back chamber. The CO_2 laser source is tuned to the P(20) 10.59 micrometer line center and chopped at about 0.25 Hz as compared to

the approximate 0.05 Hz rate used for pellet-particulate tests. This faster on-off rate is possible due to the shorter CO₂ gas absorption energy transfer rates. The presence of the 150-200 Torr CO₂ pressure makes it necessary to include self-broadening effects along with the N₂ foreign-broadening. In addition, the comparative thermal properties of CO₂ and N₂ must also be taken into consideration because of their widely different magnitudes. A calibration absorption sensitivity figure of about 2100 ±200 volts per watt absorbed was obtained for the .03 capacitance manometer scale and a leak-valve setting resulting in a 20 second test period for a 250 mg pellet.

A second approach to calibrating the differential spectrophone involves use of a highly absorbing sample pellet mixture such as acetylene soot in the 10 micrometer wavelength region. Since scattering can be approximated as being negligible compared to soot absorption at these wavelengths, an extinction test can be compared to the spectrophone signal obtained by using the same pellet. After pellet air-to-solid boundary reflection losses of about 8% are subtracted, the remaining loss due mostly to absorption can be equated to the spectrophone signal. The resulting absorption sensitivity figure of 2000 ±200 V/W compares well with the CO₂ derived figure of 2100 V/W. It should be noted that the geometry of the calibration absorption region conditions are somewhat different between the two methods. Therefore, it is not surprising to see some difference between the two numbers.

The third approach to calibration uses a coil of nichrome wire placed in the back chamber. By measuring the resistance of the coil, a known amount of energy can be dissipated and the corresponding pressure change converted to a voltage signal by the capacitance manometer. During the calibration tests, it was discovered that where the coil is positioned in the back chamber is

an important factor in the calibration results obtained. It was also determined that the time constant of the wire itself is substantially less than the pellet-chamber interaction. Therefore, the nichrome wire was wound in a flat coil geometry and placed in the pellet position in the back chamber. Absorption sensitivity calibration numbers of about 1900 ± 200 V/W were obtained. This is in good agreement with the CO_2 and infrared extinction calibration method results.

EXPERIMENTAL PROCEDURE AND RESULTS

General

Preliminary results have been obtained at three different laser wavelengths; 1.06, 9.6, and 10.6 micrometers. Samples tested include atmospheric dust, quartz, and acetylene soot. The quartz and soot samples were chosen for their widely varying absorption properties between the three wavelengths and the fact that some absorption data is available using other methods.

Table I lists the absorption indexes as measured. It is estimated that the results contain a $\pm 25\%$ uncertainty which is expected to be improved using newer laser sources and a thermally shielded capacitance manometer system.

All of the tests were run using a laser power input to the calibrated power meter of 200 milliwatts. The sample mixture pellet is placed in the back chamber so that it is as close as possible to the power meter. The meter readings are then corrected to correspond as closely as possible to the power level at the pellet which is separated from the power meter by the back NaCl window. Alternatively, the back NaCl window can be temporarily removed for more power measurement accuracy if necessary.

Lambert's law is used to calculate the absorption numbers from the experimental test data. Particle size (with respect to wavelength) appears to be an important parameter due to its possible effect on absorption per unit mass of sample. However, in these early tests we have assumed that all samples have been properly prepared such that particle size is less than the test wavelengths used. Later on, the pellet-spectrophone method may be very valuable in determining the extent of size-absorption effects. Lambert's law is given by

$$I = I_0 e^{-\alpha \rho x} \quad (1)$$

where x is the distance in centimeters, α is the mass absorption coefficient in units of cm^2/g , and ρ is the particulate mass density. It is used in calculating α or $k = \alpha \rho$ from the test results. Absorption data can also be expressed in terms of the imaginary part of the complex refractive index, n' . Appropriate parameters are:

n' = imaginary part of the complex refractive index.
= absorption index.

$k = \alpha \rho = \frac{4\pi n'}{\lambda} = \text{absorption coefficient.}$

$n'/\rho = \text{mass absorption index.}$

$k/\rho = \text{mass absorption coefficient.}$

$\lambda = \text{wavelength.}$

Rearrangement of Eq. 1 results in

$$e^{-\alpha \rho x} = 1 - W_{\text{abs}}/I_0 \quad (2)$$

where W_{abs} is the absorbed power. For these tests, I_0 is obtained from the power meter reading by adding on the power lost at the NaCl back window surfaces and the power lost in the pellet. Pellet

Pellet losses include both KBr host pellet surface reflections and W_{abs} . Normally, W_{abs} is quite small and can be neglected in determining I_0 . However, it still must be known for use in Eq. 2 and is determined by dividing the test signal in volts by the system absorption sensitivity parameter of 2000 V/W on the .03 capacitance manometer scale. The 2000 V/W figure is an average of the three calibration methods. The equivalent sample thickness x can be found by calculating the particulate volume per pellet and dividing by the pellet cross-sectional area. If ρ is known, k or n' can be determined from the test results. If ρ is not known, k/ρ or n'/ρ or α can be found.

Scattering Extinction Tests

Equation 1 as stated and used concerns absorption losses only. However, scattering extinction effects can be important and even dominant in shorter wavelength regions. One of the main purposes of the 1.06 micrometer wavelength quartz test listed in Table I was to attempt verification of spectrophone separation of scattering and absorption losses. It was not expected that much, if any, spectrophone absorption signal would be observed, since quartz has a negligible absorption coefficient at this wavelength. However, some scattering is known to occur and if the spectrophone is at all sensitive to scattered energy, some signal should develop. No signal was detected, verifying at least some insensitivity to scattering losses.

Further efforts at determining a scattering sensitivity factor, as compared to absorption, consisted of applying a controlled density of 15-25 micron diameter nonabsorbing glass bead spheres on one surface of a KBr pellet. The pellet was then placed in the back chamber of the spectrophone and irradiated with a Nd:YAG laser source at 1.06 micrometers. A developed spectrophone signal

of 0.96 volts was measured. This was followed by an extinction test with the back NaCl window removed and the bead covered pellet placed alternately in and out of the beam. After normal KBr reflection losses were subtracted, an attenuation loss of 105 milliwatts remained under the same power level conditions that the spectrophone test was run at. The ratio of 0.96 volts to 105 milliwatts scattering loss is equivalent to a scattering sensitivity of 9.1 V/W. Since the absorption sensitivity, as discussed earlier, is about 2000 V/W, the spectrophone system ratio of absorption to scattering response is somewhat greater than 200. This ensures practical operation down into the shorter wavelength regions. It also appears that this ratio of 200 may be improved by further design modifications if necessary.

Absorption Test Results

The atmospheric dust test sample was collected in Tel Aviv, Israel. Spectrophone sample pellets weighing 250 mg and containing 2.9×10^{-5} g dust/pellet were made from a mixture containing 8.39372 g KBr and 0.00097 g dust. Test signals of from 2-12 volts were obtained depending on which of the three source wavelengths were used. We have compared our spectrophone results at 1.06 micrometers with that obtained using previously reported diffuse reflectance methods^{3,4} on part of the same parent sample. Some difference can be noted in Table I. Agreement at 9.6 and 10.6 micrometers is good compared with data recently published by Fischer⁷, even though his sample was collected at an entirely different location in Mainz, Germany.

For quartz absorption tests, sample pellets weighing 250 mg and containing 3.6×10^{-5} g quartz/pellet were made from a mixture of 6.86896 g KBr and 0.00100 g finely ground quartz. Measured signal levels were in the 2-10 volt range for the 9.6 and 10.6

micrometer wavelengths. As stated previously, no absorption signal was observed at 1.06 micrometers. Reasonably good agreement with Peterson and Weinman¹² exists at 9.6 and 10.6 micrometers.

The third material tested at the three laser wavelengths was a mixture of KBr and acetylene soot. Due to its strong absorption properties, a beginning mixture of 12.07537 g KBr and 0.00163 g acetylene soot was further diluted until only 3.3×10^{-6} g of soot/pellet was obtained. A comparison of the data in Table I with that of Foster and Howarth¹³ reveals good agreement at 1.06 and 9.6 micrometers.

CONCLUSIONS

This work shows that a spectrophone device can be constructed which is at least 200 times more sensitive to absorbed energy than scattered energy in measurement of the imaginary refractive index of an atmospheric dust sample. Application of this device to samples of powdered quartz shows that it gives good agreement with known values of the imaginary index. This occurs both in the 10 micrometer region where quartz is a strong absorber and at 1.06 micrometers where quartz does not absorb significantly, even though attenuation in powdered quartz at 1.06 is high due to scattering. The spectrophone method, when applied to atmospheric dust samples, also agrees reasonably well with measurements made by two other methods. Measurements made on carbon particles are also in agreement with other researchers. In addition, it is shown that the spectrophone measurement can be made on very small quantities of sample. This is a feature of considerable importance for study of atmospheric dust where long collection times are required to acquire even small quantities of sample. Considering the inherent flexibility given by the fact that the signal obtained from

a pellet spectrophone is independent of wavelength, we now have a device that permits measurements at different ultraviolet, visible, or infrared wavelengths to be directly compared. Because of these rather desirable characteristics, it appears that the KBr pellet spectrophone is a device worth developing further.

TABLE I. Absorption Index of Atmospheric Dust, Quartz, and Soot

Particulate Matter	$\lambda (\mu m)$	Spectrophone ^a n'	Other Method ^b n'
Atmospheric Dust (Assumed $\rho = 2.4$)	1.06	0.036	0.023 Unpublished data ^{3,4}
	9.6	0.290	0.286 Fischer ⁷
	10.6	0.135	0.145 Fischer ⁷
Quartz ($\rho = 2.66$)	1.06	Negligible	$< 10^{-7}$ Peterson & Weinman ¹²
	9.6	0.325	0.283 Peterson & Weinman ¹²
	10.6	0.051	0.034 Peterson & Weinman ¹²
Acetylene Soot (Assumed $\rho = 1.7$)	1.06	0.586	0.65 ^c Foster & Howarth ¹³
	9.6	0.733	0.90 ^c Foster & Howarth ¹³
	10.6	0.732	

a Mean values of tests.

b Interpolated data.

c Compared with natural gas soot.

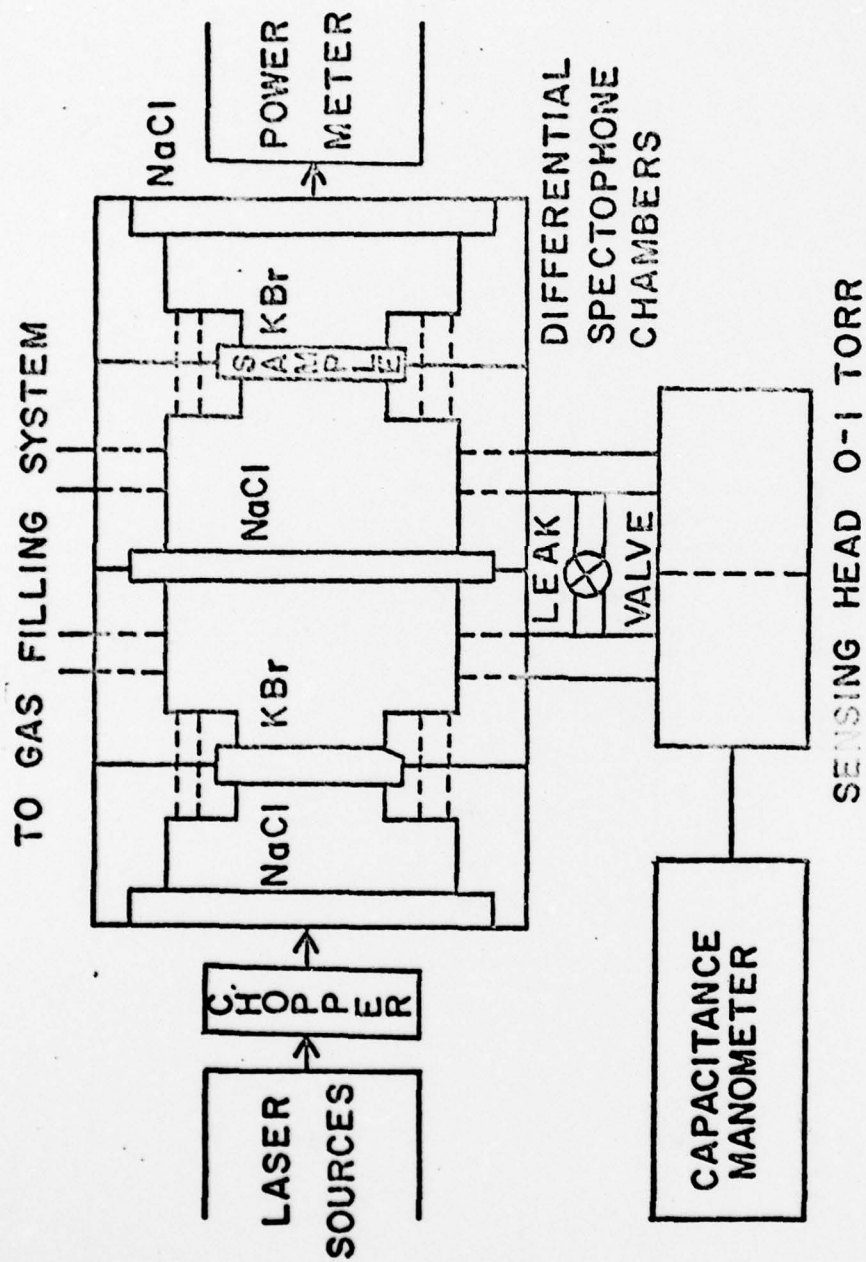


Fig. 1. Block diagram of differential spectrophone system.

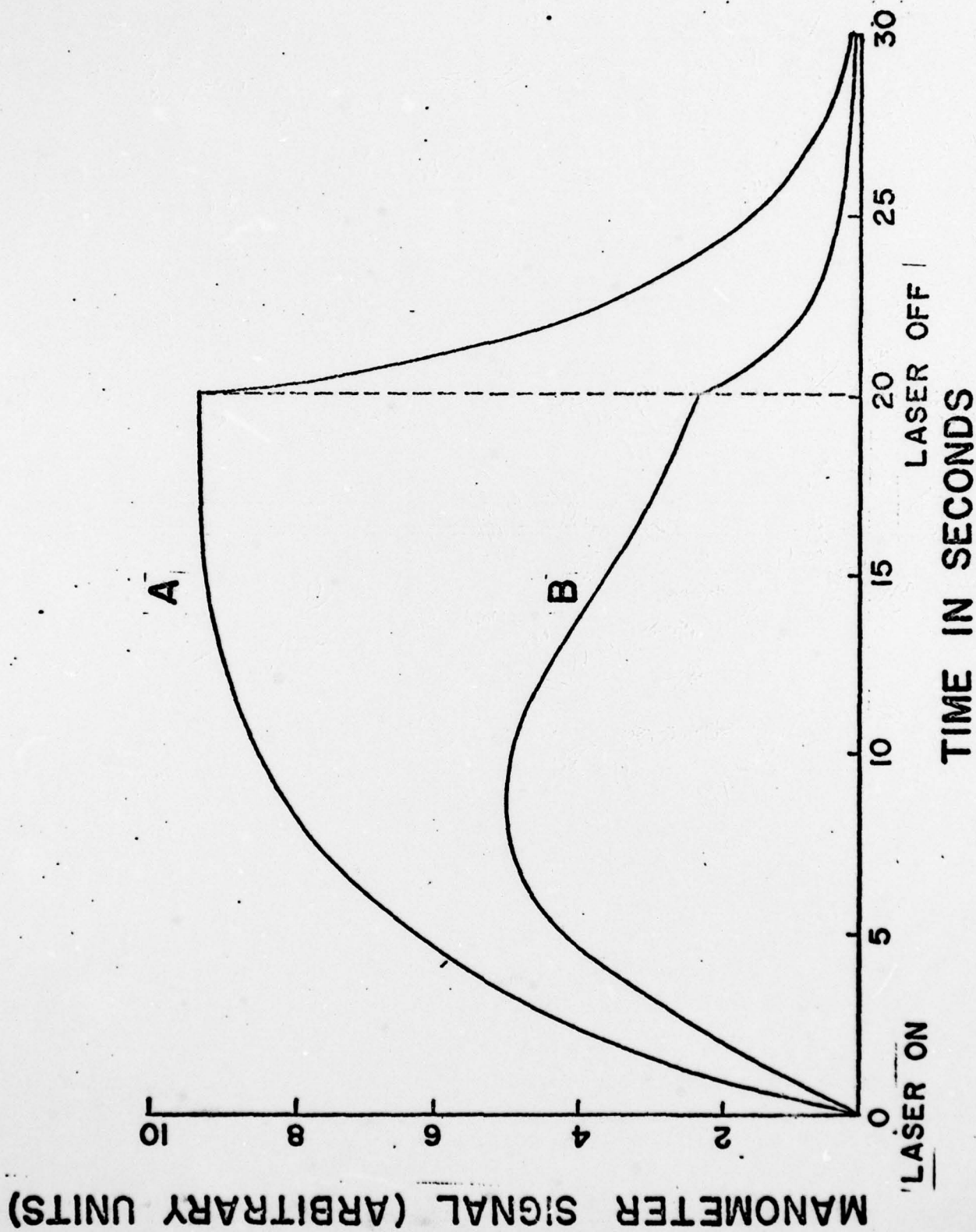


Fig. 2. Spectrophone output signal. Curve A corresponds to the desired leak-valve setting while Curve B corresponds to a too open position.

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Flowing Spectrophone Feasibility Tests

In-situ atmospheric particulate absorption coefficient tests are really the desired end result of past and future research efforts. Therefore, first feasibility tests were run in an attempt to develop a flowing spectrophone system. A thin-walled copper tube was used as a barrier between the absorption medium and the capacitance manometer measuring system. This effort was made to determine if there was any possibility of using an indirect approach wherein a part of the absorption energy could be transferred to the capacitance manometer through a pressure change barrier. The first results were extremely encouraging until it was discovered that a small leak existed through the copper wall and that this leak was responsible for much of the signal first observed. However, later tests have shown that a signal can be transmitted, although a heat isolation section is needed in series with the leak valve. A better approach would appear to be a method using membrane filters in the sensing head lines. This would protect the head from contamination and still provide direct pressure change capability. This approach will be given further effort whenever possible in the future.

Conclusions and Recommendations

Even though extra effort was placed in Task II at the expense of Task I, much more work remains to be done. This is very much due to the importance of the pellet-spectrophone approach to determining atmospheric particulate absorption coefficients. It is highly recommended that extensive efforts be placed in the continuation of the development of this device. Specific items needing further work are calibration, minimization of scattering effects, geometry, ultimate sensitivity, and particle size effects. Fortunately, none of these areas appear to be a major limiting factor. Other spectrophone system modifications should also be investigated.

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