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INFRARED ABSORPTION SPECTRA ATTRIBUTED TO
ION-NUCLEATED WATER CLUSTERS

by

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Research Division

September 1977



US ARMY ARMAMENT RESEARCH AND DEVELOPMENT COMMAND
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Emission/absorption	Atmospheric infrared absorption	Air
Absorption coefficient	Clusters	Bulk water phase
20. ABSTRACT (Continue on reverse side if necessary and identify by block number) (U) Until recently, it was assumed that atmospheric water was found in one of three phases: vapor (monomer), liquid droplets or ice crystals. Now it is known that an ion-nucleated, polymolecular cluster phase of water exists in the vapor phase, as well. These water clusters consist of numbers of water molecules gathered about small ionic nuclei, where the number of molecules per cluster is dependent upon relative humidity and other meteorological parameters. (Continued on Reverse Side)		

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20. ABSTRACT (Continued)

in real atmospheres, mean cluster sizes range from about 11 to about 14 molecules per cluster. Their infrared absorption is due to intermolecular hydrogen bonding, and the "continuum"-like absorption spectrum which they produce is shifted in wavelength by changes in mean cluster size. Water clusters are always present in the atmosphere and in the cleanest of laboratory experimental equipment. Except under supersaturations of about 420% relative humidity, clusters are not able, because of equilibrium considerations, to attain "critical" size and to grow to droplets. Water cluster theory and equations give excellent agreement with observed data for infrared "continuum" absorption.

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PREFACE

The work described in this report was authorized under In-House Laboratory Independent Research. This work was started in August 1976 and completed in June 1977.

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SUMMARY

The spectrum is presented of anomalous infrared absorption attributed to ion-nucleated water clusters in vapor-saturated air. The absorption is anomalous in the sense that it cannot be accounted for by water vapor or liquid water droplets. The spectrum was taken by emission, from a 3.05-m path length of cooling, saturated steam at 32.5°C. Relationships between mean cluster size, $(c)_{\text{mean}}$, saturation ratio, (s) , and wavelength shift of the spectrum are developed. The wavelength shift continues into the bulk liquid phase of water as well, where $(c)_{\text{mean}}$ is determined primarily by the purity of the liquid water sample. It is concluded that the anomalous infrared absorption in real atmospheres is attributable to ion-nucleated, polymolecular water clusters of size $11 \leq (c)_{\text{mean}} \leq 14$. Practical applications of these findings are discussed in this paper. A first composite of water cluster absorption spectra is constructed for the infrared wavelength region from 2 to 24 μm , for a variety of vapor-phase and liquid-phase conditions.

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INFRARED ABSORPTION SPECTRA ATTRIBUTED TO ION-NUCLEATED WATER CLUSTERS

I. INTRODUCTION.

In an earlier paper¹ we presented experimental infrared emission spectra of steam in the 7- to 13- μm wavelength region. These spectra showed anomalous emission which we attributed to tiny water droplets that were too small to produce noticeable optical scattering in the visible wavelengths. The spectra exhibited wavelength shifts with heating or cooling. Because instrumentation was not available, it was impossible to characterize the aerosols present in those trials.

As part of recent work in aerosol spectroscopy² we were able to perform similar experiments with condensing steam in which the aerosol was completely characterized. Operating under conditions of radiometric null at maximum radiometer gain settings, we have repeatedly observed a spectral feature in warm air saturated with water vapor which cannot be attributed either to water vapor or to water droplets. These recent experiments were designed so that a saturation ratio, $(s) = 1.0$ was maintained as residual steam clouds in a test chamber with a 3.05-m optical path were permitted to cool gradually and to dissipate. After careful corrections were made for spectral absorption/emission by water vapor or by residual droplets,³ the anomalous spectral feature remained in almost all trials. In thermal equilibrium, the observed anomalous emission had radiant emittance values three to four times higher than the combined emittances of water vapor and residual droplets present in the test chamber. In earlier papers, the radiometer¹ was described and the experimental test chamber arrangement and procedure² were discussed.

II. EMISSION/ABSORPTION SPECTRUM ATTRIBUTED TO WATER CLUSTERS IN AIR.

Figure 1 shows the absorption spectrum of the anomalous feature as we observed it in the 8- to 13- μm wavelength region for the experimental conditions, namely, air temperature, $(\theta_k) = 305 \pm 5^\circ\text{K}$ and saturation ratio, $(s) = 1.0$. Each hollow point in figure 1, depending upon wavelength, averages 6 to 9 experimental data points. The vertical error bars for these representative data enclose the maximum excursions of any experimental data points at each wavelength. The horizontal error bars indicate the radiometer wavelength resolution, which was 2% of center wavelength. The values of the anomalous absorption coefficient, $(\alpha_c)_\lambda$, (m^2/gm) shown in figure 1 are used in equation 1:

$$\ln(1/T_\lambda) = (\alpha_c)_\lambda (C_c) (L_m) \quad (1)$$

where

C_c = the concentration of the anomalous species (gm/m^3)

L_m = the optical path length (3.05 m in the present experiments)

T_λ = the infrared transmittance at wavelength, λ , (μm)

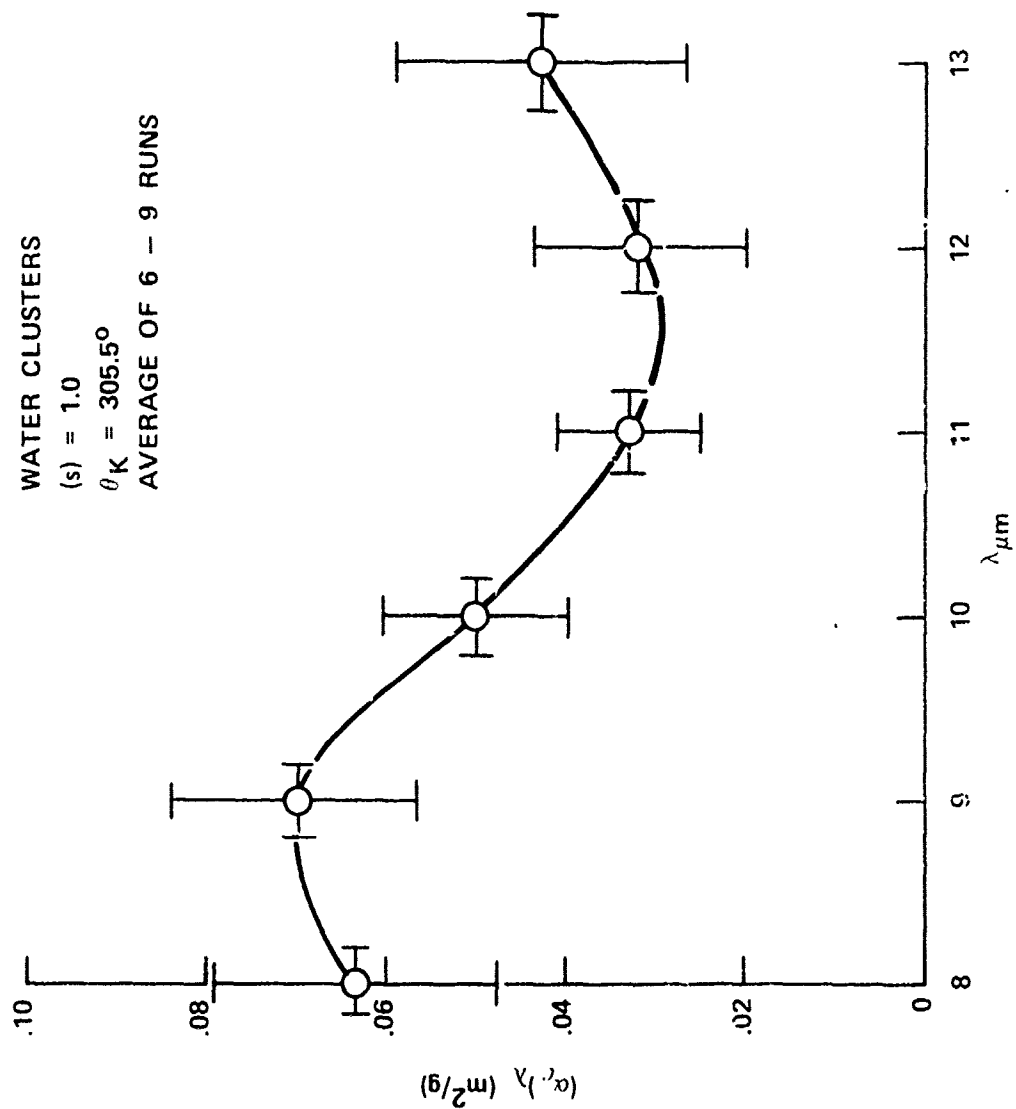


Figure 1. Infrared Absorption Spectrum Attributed to Water Cluster in Vapor-Saturated Air

Since, in the course of these experiments, we did not know the precise nature of the phase-transitional species present in our chamber (which accounted for the anomalous absorption), the determination of C_c was difficult because represented in this concentration were species which were neither vapor nor liquid. A simpler procedure would have been to combine $(\alpha_c)_\lambda$ and C_c into a single absorption coefficient with reciprocal length units. To do this however would have eliminated the opportunity to study changes in the spectral behavior of the species with experimental parameters, especially (s). Since we had already observed evidence of wavelength shifts in anomalous emission spectra of steam under varying saturation ratios, we decided to work with $(\alpha_c)_\lambda$ and C_c separately, as is our custom in aerosol spectroscopy.² The calculation of quantitative values of $(\alpha_c)_\lambda$ involved a determination of the water present in excess of that present as either vapor or liquid aerosol droplets larger than about 0.1μ . It will become apparent in the following discussion that the $(\alpha_c)_\lambda$ shown in figure 1 is in reasonably consistent agreement with observed data, but that the shape of the spectrum in figure 1 probably is more important than its absolute amplitude, at least for the present work.

Water clusters are the phase-transitional species between water vapor and liquid water droplets in the absence of "foreign" condensation nuclei. However, as C. T. R. Wilson^{4,5} showed in his classical cloud chamber work, later lucidly summarized by J. G. Wilson,⁶ water clusters in real atmospheres are not able to grow large enough to reach stable size except in the presence of vapor supersaturations of at least about (s) = 4.2 (i.e., 420% RH), for standard starting conditions of temperature and pressure. As stated by J. G. Wilson, the critical diameter for droplet formation corresponds to a cluster size or count (c = water molecules/cluster) of about 30. Thus, in real atmospheres we are given a picture of water clusters of statistical size distributions with "tails" extending from (c) = 2 to (c) = 30 but with statistical mean values of (c) somewhere in between, co-existing with a bewildering array of "foreign" condensation nuclei, and with the whole lot dependent for average cluster or droplet size primarily upon meteorological parameters, especially the saturation ratio, (s).

We know also that water clusters can be nucleated in two ways: on ions, or by homogeneous nucleation. McDonald's papers^{8,9} on homogeneous nucleation of water vapor are classics, and Abraham¹⁰ has given an excellent presentation of this subject. But in applying their data⁸⁻¹⁰ to the real atmosphere case which we were considering, we found that homogeneous nucleation of water clusters did not satisfy our observed results for two reasons:

1. The expected mean value of cluster count (c) for homogeneously nucleated water clusters is zero, not somewhere between 2 and 30 depending primarily upon the value of (s) as will be seen to be necessary in the present discussion.
2. The numbers and mass fractions of water clusters predicted by homogeneous nucleation theory are not nearly large enough to account for the observed infrared spectral effects.

Thus, in the balance of this paper we shall attempt to show that figure 1 depicts the infrared spectrum of water clusters in air, of cluster sizes (c) between 2 and 30, distributed about some mean value of (c), and corresponding to the case where (s) = 1.0 and $(\theta_k) = 305.5^\circ\text{K}$. Furthermore, we shall attempt to show that these water clusters are formed on *ionic nuclei*.

III. RELATIONSHIP BETWEEN (c), (s), AND $\lambda_{\max}$.

The literature contains many references pertaining to the growth of water clusters on ionic nuclei in equilibrium with various saturation ratios, (s). The work of Castleman and Tang¹¹ is perhaps best known. The lower curve of figure 2 shows results obtained by these workers relating mean molecule counts, (c), of water clusters formed on lead ion, (Pb+), for various values of (s). J. G. Wilson⁷ indicated that the sign of the ion will affect (c):(s) equilibria, with negative ions being favored in the case of water. In addition, results of other workers^{12,*} indicated that when ionically nucleated in typical vapor-saturated air (s) = 1.0 and for $273^\circ \leq \theta_K \leq 373^\circ \text{K}$, mean water cluster sizes in air of between (c) = 11 and (c) = 17 were found. Thus, in figure 2 an upper curve is drawn, nearly parallel to the lower curve of Castleman and Tang's data,¹¹ but for substantially larger values of cluster size, (c), corresponding to given (s) values. The left-hand end of the upper curve in figure 2 is drawn through a point which can be determined from the absorption spectrum of water vapor [i.e., monomer, (c) = 1] by a lengthy calculation. (This will not be presented here.) In this discussion, the precise relationship between the mean size of water cluster distributions found in real atmospheres and saturation ratio is relatively unimportant at this point. Since the upper curve of figure 2 is probably representative, we shall use its equations in the subsequent discussion.

$$(c) = 1.2 \ln(s) + 14.2 \quad (2)$$

$$(s) = \exp [0.833(c) - 11.8] \quad (3)$$

It was mentioned earlier that a wavelength shift occurs in the spectrum of figure 1 with changes in (s). The maximum of $(\alpha_c)_\lambda$ at $\lambda_{\max} = 9.0 \mu\text{m}$ moves to the left (i.e., toward higher frequencies) at lower (s) values, and the magnitude of $(\alpha_c)_\lambda$ diminishes very rapidly as well. In our chamber measurements, this seemed to give an abrupt loss of emission signal when vapor saturation was lost, usually a few minutes after evaporation of the last visible liquid water droplets. Evidence of this wavelength shift also is found in atmospheric or laboratory infrared spectra of the 8- to 13- μm wavelength region taken under conditions where (s) is known or can be calculated. In practice, it is found that $\lambda_{\max}$, the wavelength at which the peak value of $(\alpha_c)_\lambda$ is found (as in figure 1), is approximated by

$$\lambda_{\max} = 2.41 \sqrt{c} \quad (4)$$

and since (c) can be calculated from (s) if their relationship is known, as for example from equation 2, it is possible to relate $\lambda_{\max}$ to both mean cluster size (c) and to (s) with which (c) is in equilibrium. For example, at (s) = 0.1 (10% RH), $(\alpha_c)_\lambda$ will have shifted its peak to $\lambda_{\max} = 8.15 \mu\text{m}$ from $9.0 \mu\text{m}$ at (s) = 1.0.

* Harden, C. S. Chemical Systems Laboratory, Aberdeen Proving Ground, Maryland 21010; private discussions on charged water cluster size distributions as functions of saturation ratio based upon quadrupole mass spectrophotometric measurements, July 1976.

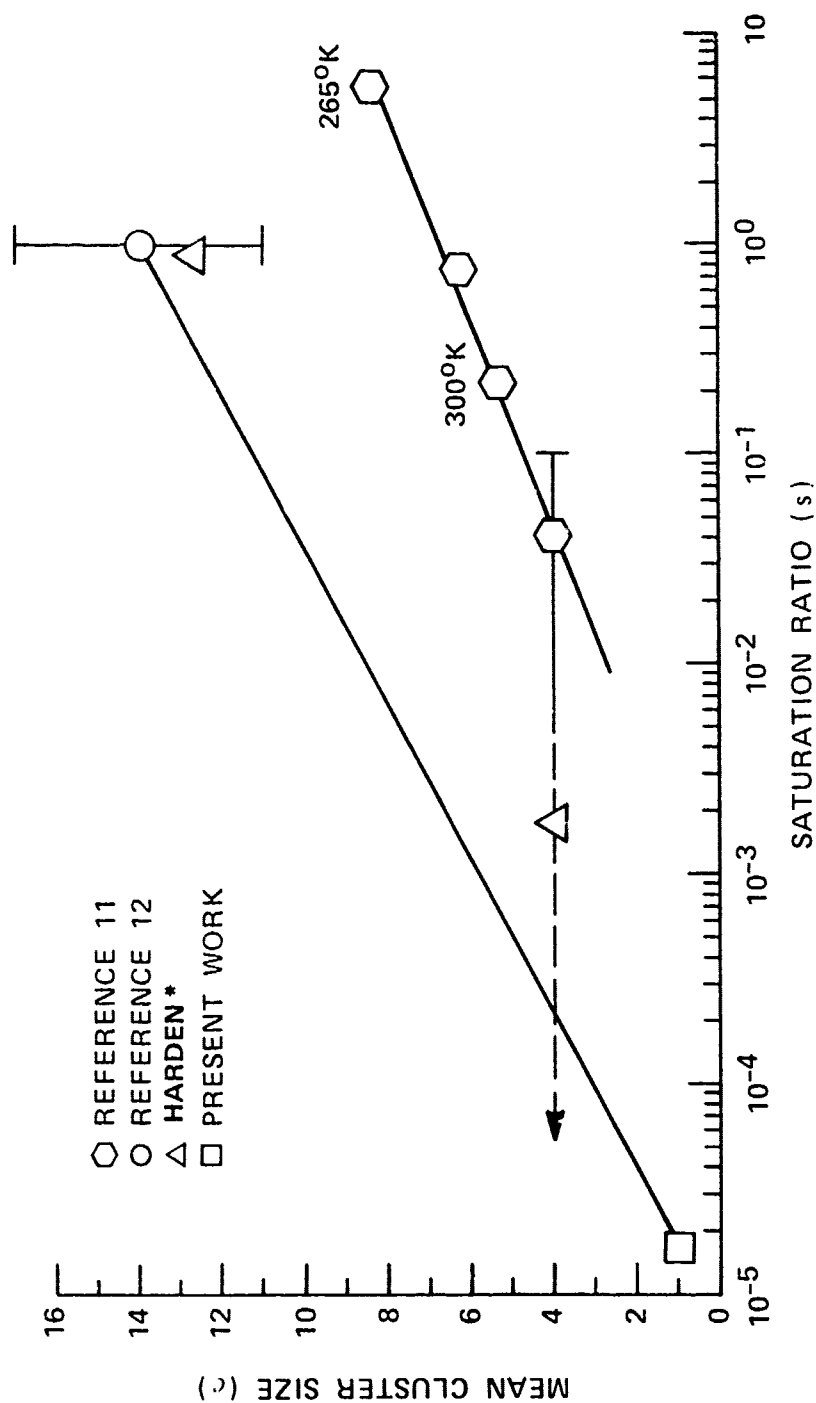


Figure 2. Relationship Between Mean Cluster Size and Saturation Ratio for Water Clusters in Air

Upper curve — data taken from papers of Potter and Hoffman,^{1,2} from Harden,* and from present work.

Lower curve — data taken from paper of Castleman and Tang,^{1,1} for (Pb⁺) ions.

* Harden, C. S. Chemical Systems Laboratory, Aberdeen Proving Ground, Maryland 21017; private discussions on charged water cluster size distributions as functions of saturation ratio based upon quadrupole mass spectrophotometric measurements, July 1976.

IV. THE WATER DIMER.

The few attempts which have been made to relate atmospheric infrared absorption to water clusters have involved the water dimer, i.e., the cluster of size $(c) = 2$. Varanasi, *et al.*¹³ reported unusually high, or anomalous, water "vapor" absorption coefficients near 10- μ m wavelength with inverse temperature dependency which was considered by Bignell¹⁴ and others,¹⁵ possibly to be explained by the presence of the water dimer. Presumably, the equilibrium considered in moist air was



where the equilibrium constant is very small and the expected value is near zero. Ionic nucleation is not considered in equation 5. Rather, the "nucleation," if it can be so called, is of the homogeneous type. We have shown earlier that homogeneous nucleation almost certainly is not the mechanism of water clustering responsible for infrared activity observed in the open atmosphere. As for ionic nucleation, $(c) = 2$ is the expected value only for extremely low values of (s) . Thus, in the context of the present discussion, the dimer is but a small contributor to infrared absorption on the low- (c) "tail" of a statistical distribution of absorbing clusters centered on some mean value of (c) determined primarily by (s) . In real atmospheres, from the equations presented here, a reasonable approximation is:

$$11 \leq (c)_{\text{mean}} \leq 14.$$

V. A COMPOSITE PICTURE OF WATER CLUSTER INFRARED ACTIVITY.

We hypothesize at the outset of this section that the physical phase in which water clusters are found seems to have little effect upon their infrared spectra. The relationships between broad, continuum-like infrared absorption band wavelength shifts and solute ion concentrations in liquid water solutions are well documented¹⁶ if not fully understood. Clearly, these ion concentrations affect complex refractive index measurements even of "pure" water, especially the imaginary index component.* Using equation 4, it was possible to predict, with reasonable agreement, the location of λ_{max} for the absorption peak due to water clusters of larger mean size which are found in the liquid, as compared to the vapor phase. λ_{max} is found near 14 μ m in infrared spectra of reasonably pure liquid water films (hopefully uncontaminated by ions from salts used to fabricate sample cell windows), corresponding to mean cluster sizes of about $(c) = 34$ molecules per average water cluster formed on available ionic nuclei at any given instant in bulk liquid water samples. Other lesser cluster absorption maxima are found near 6.9- μ m and 4.7- μ m wavelengths in "pure" water film spectra. They, too, change or shift wavelength with solute ion concentration and, hence, with mean cluster size. Their behavior can be described by equations that have the form of equation 4, but with suitably adjusted coefficients.

* Querry, M. R. University of Missouri, Kansas City, Missouri; private discussions of refractive index measurements of water-containing liquid solutions, in the infrared. March 1977.

From all available information we were able to prepare a tentative, first composite picture of infrared absorption activity by water clusters in air or in liquid water solution. This composite is shown in figure 3. We cannot overemphasize the fact that this figure is intended only as a starting point for subsequent work. The approximations used in its preparation were of first order only, and it would be most fortuitous if subsequent work confirmed any degree of precision in these primarily calculated curves.

Nevertheless, figure 3 does contain a great substantial amount of "hard" data. To begin with, steam turns out to be a nearly ideal medium for the study of infrared absorption effects of water because both the vapor and liquid phases are present under vapor saturation conditions and at high vapor pressures. Initially, recondensation of steam droplets produces rather pure liquid water droplets, and thus the shift of $\lambda_{\max}$ (equation 4) of the continuum absorption is accomplished smoothly between physical phases. Potter and Hoffman¹² found not only anomalous infrared emission in this phase transition at 100°C, but apparent luminescence (i.e., nonthermal behavior) as well, which they attributed to water clusters. Good infrared absorption coefficient, $(\alpha)_{\lambda}$, spectra of water vapor (monomer) and liquid water films also are available from many sources, including our own measurements. Wavelength shifts with specific solute concentrations in the liquid phase are known (e.g., for HCl.¹⁶ used as data points in figure 3). A portion of the curve passing through the "phase change" point of figure 3 is the spectrum of figure 1. Shapes of the curves in the vapor phase are known from atmospheric and laboratory infrared spectra of anomalous water "continuum" absorption, e.g., as summarized by Roberts, *et al.*¹⁷ The wavelength shift in the vapor phase can be deduced from water vapor spectra where (s) is known.¹⁸ While specific points in figure 3 corresponding to various (s) and (c) values in the vapor phase can only be estimated from figure 2 and equations 2 and 3, the limiting values of $(\alpha_c)_{\lambda}$ for the monomer (vapor), and its wavelength spectrum, are known.

The development presented in this paper can be extended to include equations relating water cluster absorption to anomalous atmospheric infrared "continuum" absorption which has proven to be very troublesome to modelers of atmospheric transmission and its prediction, especially in the 7- to 13- μ m atmospheric window region. Bignell¹⁴ and others have call this "e-type" absorption. Infrared continuum absorption by atmospheric water will be discussed in a forthcoming paper which will be devoted specifically to that subject.

VI. CONCLUSIONS.

We conclude that:

1. Figure 1 is very probably the infrared absorption spectrum of the statistical distribution water clusters found under atmospheric pressure at saturation humidity (s) = 1.0 and $\theta_k = 305.5^\circ\text{K}$. The probable mean cluster size or molecular count at any instant in time is on the order of (c) = 14 (equations 2 and 3). In real atmospheres, the range of mean water cluster sizes is on the order of $11 \leq (c)_{\text{mean}} \leq 14$.

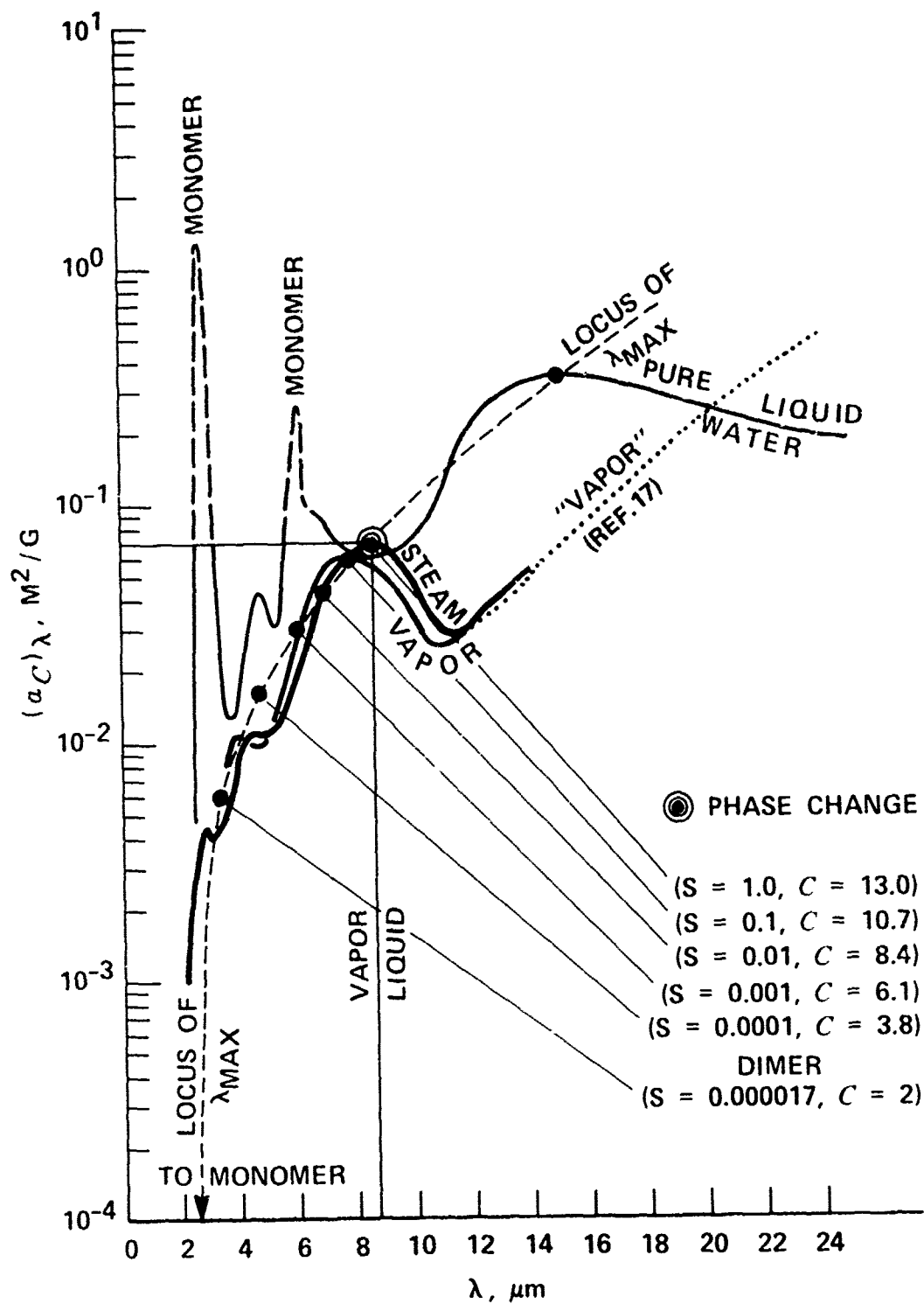


Figure 3. Composite Picture of Water Cluster Infrared Absorption Activity in the Liquid and Vapor Phases, Based Upon Best Current Data

2. The wavelength of the maximum value of the absorption coefficient, $(\alpha_c)_\lambda$, which is $\lambda_{\max} = 9.0 \mu\text{m}$ in figure 1, shifts toward shorter wavelengths (higher frequencies) with decreasing saturation ratio, (s), or with other changes that tend to reduce (s), e.g., increasing air temperature. This wavelength shift can be approximated by equation 4.

3. The water dimer equilibrium expressed by equation 5 makes only a vanishingly small contribution to anomalous infrared absorption by atmospheric water.

4. Ions in the atmosphere or in liquid water solution are the nuclei for infrared-active water clusters. The finding of Spencer, *et al.*¹⁹ and others that stable droplet formation and growth can be enhanced by coronal discharges is consistent with this idea. In water solutions, clusters produce continuum-like infrared absorption in liquid water films, especially at longer wavelengths.

5. A first composite of water cluster absorption coefficient spectra (figure 3) can be constructed showing the response of $(\alpha_c)_\lambda$ to many parameters. This composite can serve as a point of departure for investigations into specific manifestations of water cluster absorption in the infrared, e.g., "c-type"¹⁴ infrared "continuum" absorption by atmospheric water.

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GLOSSARY

- $(\alpha_c)_\lambda$ = absorption coefficient of anomalous infrared absorption attributed to water clusters, m^2/gm .
- $(c), (c)_{mean}$ = mean number of water molecules comprising water cluster sample in a given instant of time, or cluster size of a given cluster at a given instant, water molecules/cluster.
- C_c = concentration of anomalous species considered as water clusters in sample, gm/m^3
- L_m = optical path length, m.
- λ_{max} = wavelength of peak absorption attributed to clusters, μm .
- (s) = saturation ratio (fractional relative humidity) of water in air.
- T_λ = infrared transmittance through sample at wavelength, λ .
- θ_k = absolute temperature, $^\circ K$.

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