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Characterization of Particulate From Fires Burning Silicone Fluids

The optical properties of particulate emitted from fires burning two distinct polydimethylsiloxane fluids (D_4 and M_2 or MM , where $D = (CH_3)_2SiO$ and $M = (CH_3)_3SiO_2$) were obtained using a transmission cell-reciprocal nephelometer in conjunction with gravimetric sampling. The specific absorption coefficient of particulate ash from fires burning D_4 and MM is significantly lower than that of particulate soot from an acetylene (hydrocarbon) flame. Scattering is the dominant part of extinction in fires burning the silicone fluids. This is very different from extinction by soot particles in hydrocarbon fires, where absorption is approximately five times greater than scattering. Temperatures and particulate volume fractions along the axis of a silicone fire (D_4) were measured using multi-wavelength absorption/emission spectroscopy. The structure of the D_4 flames is markedly different from hydrocarbon flames. The temperatures and particulate volume fractions very close to the burner surface are much higher than in comparably sized hydrocarbon flames. [DOI: 10.1115/1.1389057]

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Introduction

Fires fueled by silicones exhibit low heat release rates and fire severity [1,2]. The low heat release rates make them suitable for various industrial products such as transformer fluids, fire-barrier foam and thermal ablatives [3]. Also, silane and other silicon based fuels are being investigated for sub-micron particle synthesis [4,5]. In semiconductor industries, silane and its chlorides are used to deposit thin dielectric films on substrates using thermal or plasma dissociation of the vapor. A better understanding of the structure and properties of silicone fueled fires is needed for improved fire safety as well as to exploit their potential for particulate synthesis.

The burning velocity [6], chemical kinetics [7], and combustion hazards [8] associated with silicones have been studied in recent years. In addition, combustion models have been proposed for polydimethylsiloxanes [9]. The radiative emission fraction and ash composition of pool fires burning silicone fluids have been studied by Buch et al. [3]. The ash is composed of varying amounts of carbon depending on the initial fuel structure [3].

The measured radiative heat loss fraction to the surroundings for silicone pool fires with diameters from 0.1 m to 0.4 m is comparable to those of hydrocarbon flames [3]. In addition, longer chain length silicon fluids have lower radiative heat loss compared to the short chain silicones. The main reason for this behavior could be the lower silica ash volume fractions associated with the long-chain silicone fluids. However, there are very few studies that provide information on the ash volume fraction or the temperature distributions within siloxane flames.

In flames containing particles, obtaining temperature information using either conventional techniques such as thermocouples or laser based techniques such as Raman Scattering is not easily accomplished. Temperature and soot volume fraction information in hydrocarbon based fuels have been routinely obtained using intrusive emission/absorption pyrometry [10,11]. The crucial information required to obtain temperature and ash-volume fraction from siloxane flames is the specific absorption coefficients of the particulate.

Light scattering/extinction measurements in silane flames show that the particle diameters are rather large, varying from 50 nm to 200 nm [5]. Information, however, on the refractive indices, which are needed to obtain specific absorption coefficients are not available. In addition, Zachariah [5] performed measurements at one wavelength, while two wavelength pyrometry requires absorption coefficients at two wavelengths. Refractive indices and specific extinction coefficients in the infrared wavelengths (from 2 μm to 40 μm) for silica aerogel are available [12]. However, obtaining absorption coefficients from these measurements is not possible without knowing the scattering to extinction ratio. In addition, there is no information available in the literature on the absorption coefficients in the near infrared region of the ash formed during the burning of siloxane flames.

The objective of this study was to obtain the absorption coefficients of silicate ash in the near infrared region, and to utilize this information to obtain the temperature and ash volume fraction in a siloxane pool fire.

Experimental Methods

The specific absorption coefficients of the particulate volume fraction (referred to here as the ash volume fraction) were measured using a transmission cell reciprocal nephelometer in conjunction with gravimetric sampling. The schematic diagram of the instrument is shown in Fig. 1. This new measurement method was recently quantified and represents a unique capability for simultaneous measurements of the specific extinction and single scattering albedo with low uncertainty [13]. The nephelometer is similar to the one used by Patterson et al. [14] and Mulholland and Bryner [15]. The exhaust gas from a 5 cm diameter siloxane pool flame was mixed with diluting nitrogen and passed through a transmission cell. The specific absorption and extinction coefficients for particulate from fires burning two polydimethylsiloxane fluids (D_4 and MM) were obtained where $D_4 = ((CH_3)_2SiO)_4$ and $MM = ((CH_3)_3SiO_2)_2$. The optical cell was oriented vertically and no significant deposition of particulate onto the glass walls of the nephelometer occurred.

The extinction (I/I_0) of a He-Ne laser was measured using a detector. The light scattered by the particles along the optical path was also measured using a wide angle cosine sensor, whose responsivity varies as the cosine of the angle between the incident

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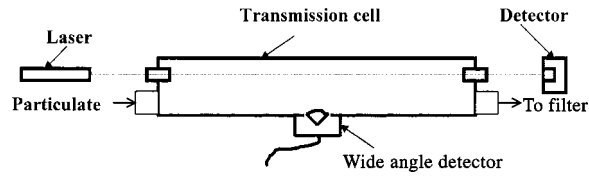


Fig. 1 Schematic diagram of the transmission cell reciprocal nephelometer

radiation and the normal. The mass flux of particles through the transmission cell was obtained by collecting and weighing the particulate on a filter.

The spectral mass specific extinction coefficient ($\kappa_{\lambda,m}$) of ash particles is given by

$$\kappa_{\lambda,m} = \frac{\ln(\tau_{\lambda})}{mL} = \frac{\ln(I_{\lambda}/I_{\lambda_0})}{mL}, \quad (1)$$

where τ_{λ} is the spectral transmittance, L is the length of the transmission cell, and m is the mass concentration of particulate. The extinction coefficient is composed of two parts, the absorption coefficient ($a_{\lambda,m}$) and total scattering coefficient ($\sigma_{\lambda,m}$). The total scattering coefficient is given by

$$\sigma_{\lambda,m} = \int_0^{2\pi} \int_0^{\pi} \sigma_{\lambda}(\theta, \phi) \sin \theta d\theta d\phi, \quad (2)$$

where θ is the scattering angle defined by the direction of propagation of the incident beam and direction of the scatter beam, ϕ is the azimuthal angle varying from 0 to 2π for a fixed θ , and $\sigma(\theta, \phi)$ is the scattering function. The total scattering coefficient was obtained by calibration with the non-absorbing aerosol dioctylphthalate (DOP) for which the scattering and extinction coefficients are equal. From the light extinction measurement, the calibration factors for the light scattering measurements was obtained [15]. A combined expanded measurement uncertainty of approximately 6 percent is expected for the absorption and scattering coefficients based on a detailed analysis using the transmission cell nephelometer [15,16]. All uncertainties reported in this paper represent the combined standard uncertainty with a coverage factor of 2 equal to two times the value of the standard deviation [17]. Uncertainty due to laser drift in the transmittance measurement was less than 1 percent.

The above procedure provides an estimate of the extinction coefficient ($\kappa_{\lambda,m}$) and the absorption coefficient ($a_{\lambda,m}$) at 632 nm for the silicate ash. The temperature (T) and the volume fraction of the ash particulate in a 10 cm diameter pool fire burning siloxane was measured using three-wavelength absorption/emission probe measurements, similar to the techniques described by Sivathanu et al. [10] and Choi et al. [18]. Utilizing the measured emission intensity at two wavelengths (700 nm and 800 nm), the temperature of the ash particulate can be obtained as

$$T = \frac{hc}{k} \left(\frac{1}{\lambda_1} - \frac{1}{\lambda_2} \right) / \ln \left(\frac{\epsilon_{\lambda_1} \lambda_2^5}{\epsilon_{\lambda_2} \lambda_1^5} \right) \left(\frac{I_{\lambda_2}}{\lambda_{\lambda_1}} \right), \quad (3)$$

where ϵ_{λ} is the specific emission coefficient, which for an arbitrary volume in thermodynamic equilibrium (and in the absence of self absorption within the volume) is equivalent to the absorption coefficient, a_{λ} [19]. I_{λ} is the measured spectral radiation intensity at wavelength, λ , h and k are the Planck and Boltzmann constants respectively, and c is the speed of light in vacuum. To obtain the temperature using Eq. (3) an estimate of the ratio $a_{\lambda_1}/a_{\lambda_2}$ (or equivalently, $\epsilon_{\lambda_1}/\epsilon_{\lambda_2}$) is needed. However, the transmission cell nephelometer provides an absorption coefficient only at one wavelength. Therefore, the ratio $a_{\lambda_1}/a_{\lambda_2}$ was obtained as described below.

Multi-wavelength transmittance measurements were conducted above a burning (pre-vaporized) stream of D_4 exiting from a 5

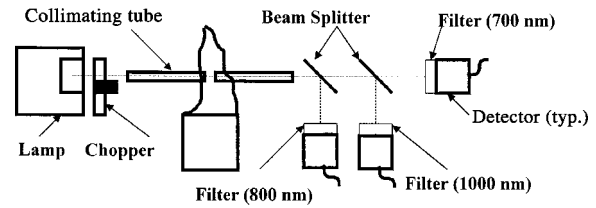


Fig. 2 Experimental arrangement used for multi-wavelength transmittance measurements

mm diameter heated tube. Figure 2 is a schematic drawing of the experimental configuration. The radiation emitted by a mercury arc lamp was collimated through a stainless steel tube, and frequency modulated using a mechanical chopper. The radiation traversed a 10 mm path in the flame, and was then split into three parts using two beam splitters. The parts were incident on three photo-detectors, which had narrow band pass filter (full-width half-maximum of 10 nm) in front of them centered at 700 nm, 800 nm, and 1000 nm. The detected signal was phase-locked using a lock-in amplifier to eliminate the emission due to radiation from the flame. The data was stored at 100 Hz using an A/D converter and a personal computer.

The conditional mean of extinction at 800 nm and 1000 nm, conditioned on the extinction at 700 nm was calculated from the measurements. The ratio of the natural logarithm of the extinction was used along with Eq. (1) to obtain the modeled spectral dependence of κ_{λ} as

$$\frac{\ln(\tau_{\lambda_2})}{\ln(\tau_{\lambda_1})} = \frac{\kappa_{\lambda_2}}{\kappa_{\lambda_1}} \cong \left(\frac{\lambda_1}{\lambda_2} \right)^m. \quad (4)$$

The ratio of specific absorption (emission) coefficients was assumed to have the same wavelength dependence as the extinction coefficient. This ratio was subsequently used in the two wavelength emission measurements along with Eq. (4) to obtain the local temperature [10]. The local ash volume fraction (f_v) in the D_4 pool was obtained from HeNe laser light extinction measurements

$$f_v = \frac{-\ln(\tau)}{\kappa_{\lambda,m} L \rho}, \quad (5)$$

where ρ is the density of particulate, and L is the length of the probe volume. The density of silica ash (taken to be equal to that of bulk amorphous silica) was taken to equal 2200 Kg/m³ based on the study by Zeng et al. [12].

Particulate was collected using iso-kinetic sampling for subsequent analysis using TEM. Samples were collected in the flame (~10 cm above the fuel surface on the central axis), above the flame in the plume (~20 cm above the fuel surface on the central axis), and in the liquid fuel bed from a 10 cm diameter steadily burning pool fire of D_4 . Samples collected in the fuel were dispersed in methanol and subjected to sonic treatment for 10 min. The dispersion was dropped onto a carbon coated Cu grid for TEM analysis. Samples collected in the flame were not subjected to a liquid medium or sonic treatment. Samples were also sent to testing laboratories for elemental analysis.

Results and Discussion

A sample TEM photograph of silica ash collected in the plume of a 10 cm diameter D_4 flame is shown in Fig. 3. The primary particles have fused together to form clusters of various sizes. Only a few primary particles are seen. Figure 3 shows that the primary particles vary in sizes ranging from 20 nm to 300 nm. Large primary particles were typical of particulate collected throughout the fire including within the flame, above the flame in the plume, and in the liquid fuel bed. The largest primary particle sizes were approximately 500 nm for samples collected from the



Fig. 3 TEM data obtained in the plume of a 10 cm diameter D_4 fire

liquid fuel. This is a much wider distribution of primary particle sizes than observed in soot particles. Clusters of primary particles of approximately 100 nm to 200 nm form the bulk of the particulate ash. The clusters are linked to form aggregates of sizes ranging from 1 μm to 3 μm . The cluster and aggregate sizes and shapes are similar to those observed in soot particles, although the primary particles are generally much larger.

Table I shows the specific extinction and absorption coefficients and the uncertainty obtained from measurements using the transmission cell nephelometer for D_4 , MM , and Acetylene. The specific extinction coefficient ($\kappa_{\lambda,m}$) of the silicate ash from D_4 and MM is approximately 5 and 2.5 times smaller than that of soot particles from acetylene flames. In addition, the scattering albedo (the ratio of the scattering to extinction coefficient, $\sigma_\lambda/\kappa_\lambda$) of the silicate ash is also shown in Table 1 and is approximately four times larger than that of the hydrocarbon soot particles. This may be due to the small imaginary component of the refractive index of silica compared to that of soot. Therefore, scattering has a major role in the radiative transfer in silicone fluid fires compared

Table 1 Specific extinction, absorption, and scattering coefficients and their uncertainties obtained from the transmission cell nephelometer

Fuel	$\kappa_{\lambda,m}$ (m^2/g)	$a_{\lambda,m}$ (m^2/g)	$\sigma_{\lambda,m}$ (m^2/g)	$\sigma_\lambda/\kappa_\lambda$
D_4	$1.7 \pm 8\%$	$0.18 \pm 6\%$	$1.52 \pm 6\%$	$0.90 \pm 10\%$
MM	$3.0 \pm 8\%$	$0.89 \pm 6\%$	$2.11 \pm 6\%$	$0.71 \pm 10\%$
Acetylene	$7.6 \pm 4\%$	$5.89 \pm 4\%$	$1.71 \pm 4\%$	$0.22 \pm 6\%$

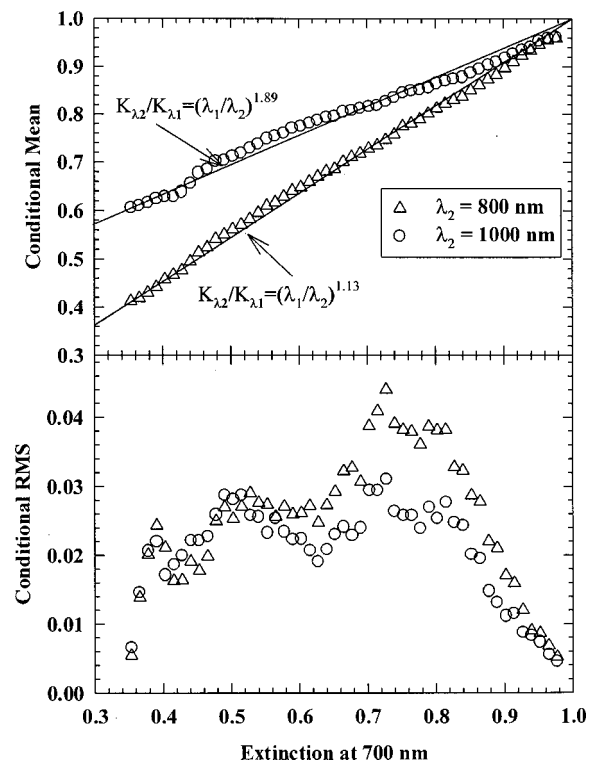


Fig. 4 Conditional mean and RMS of spectral extinction

to their relatively minor role in hydrocarbon fires. For hydrocarbon fires, there is some controversy on the variation of refractive indices with the C/H ratio of the fuel [20,21], however, the maximum variation for the extinction or the absorption coefficient is approximately 30 percent. For the fires burning silicone fluids, the variation of extinction and absorption coefficients with C/Si ratio is very high. The specific absorption coefficient ($a_{\lambda,m}$) of MM , which has an initial C/Si ratio (by mass) of 1.29 is 0.18 (± 6 percent) m^2/g , whereas that of D_4 (initial C/Si ratio of 0.86) is 0.89 (± 6 percent) m^2/g . Elemental analysis of actual particulate sample collected from the D_4 plume and from the D_4 fuel bed yielded a C/Si mass ratio of 0.24 (± 26 percent) and 0.23 (± 19 percent), respectively [22,23]. The fact that the value of the C/Si ratio for samples taken above the flame (in the plume) and samples from the fuel bed were nearly identical suggests that the C/Si ratio does not vary significantly throughout the flame. Particulate samples from the MM flame would presumably yield somewhat higher values of the C/Si ratio. Therefore, to model the radiative transfer in flames burning silicone fluids, the specific absorption and extinction coefficients have to be measured for each individual fuel. The specific extinction coefficient of 7.6 (± 4 percent) m^2/g for the acetylene soot is much higher than the value obtained by Dalzell and Sarofim [24], but is within 2 percent of the value reported recently by Zhu et al. [16].

The second set of measurements conducted for this study involved multi-wavelength extinction measurements performed on a 100 mm diameter pool fire burning D_4 . Simultaneous extinction measurements at 700 nm, 800 nm, and 1000 nm were conducted using the experimental arrangement shown in Fig. 2. The radiative fraction from this pool fire is approximately 30 percent [3]. The measurements were obtained at a height of 5 cm above the burner.

The conditional mean and the conditional RMS of extinction at 800 nm and 1000 nm, conditioned on the extinction at 700 nm are shown in the top and bottom panel of Fig. 4. The mean extinction at 800 nm and at 1000 nm increases linearly with the extinction at 700 nm. The conditional RMS of extinction at 800 nm and 1000 nm is less than 7 percent of the mean. Therefore, the measure-

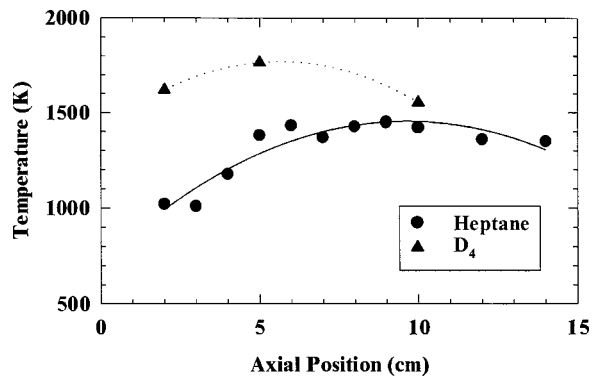


Fig. 5 Mean temperatures along the centerline as a function of distance above a 10 cm diameter D_4 pool fire. Measurements from a 10 cm heptane pool fire are also shown [18].

ments can be used to obtain a reasonable estimate of the spectral dependence of the extinction coefficients, using Eq. (4). Results obtained using a linear regression fit to the data and Eq. (4) are shown in Fig. 4. The spectral extinction coefficient varies inversely to the 1.13 power of the ratio of wavelengths between 700 nm and 800 nm. This information was used in Eq. (3) to obtain the local temperatures in the D_4 pool fire.

The mean temperatures along the axis of a D_4 pool fire are shown in Fig. 5. The measurements were obtained at three axial locations. For comparison, the mean temperatures along the axis of a heptane pool fire [18] are also shown in Fig. 5. The pool diameter in both cases was 100 mm. The mean temperatures at an axial location of 2 cm are approximately 1620 K (± 100 K) and 1020 K (± 50 K) for the D_4 and heptane pool fires respectively. This difference is in contrast to the similarity in the calculated adiabatic flame temperature determined using the NASA Chemical Equilibrium Code [25] for the D_4 and heptane flames, which equals 2370 K and 2290 K, respectively. The heptane pool fire has a fuel rich zone close to the burner, leading to lower temperatures. The structure of the heptane pool fire is similar to a laminar diffusion flame. On the other hand, the mean temperature close to the surface of the D_4 pool fire is much higher, and the temperature profile resembles that of a partially premixed flame. This effect could be due the fuel-bound oxygen present in D_4 participating in chemical reactions close to the fuel surface. Visual photographs show that the heptane pool fire bulges outwards, while the D_4 pool fire necks inwards. This structure is similar to alcohol pool

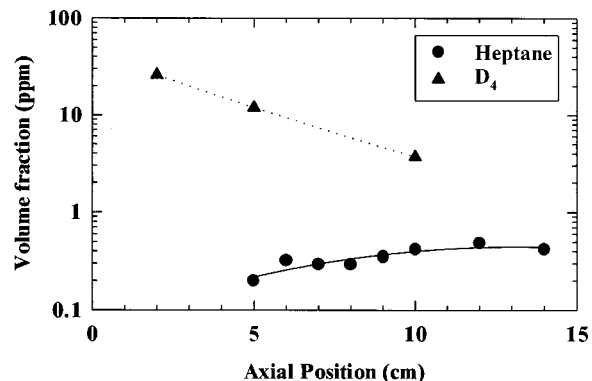


Fig. 6 Mean particulate volume fractions along the centerline as a function of distance above a 10 cm diameter D_4 pool fire. Measurements from a 10 cm heptane pool fire are also shown [18].

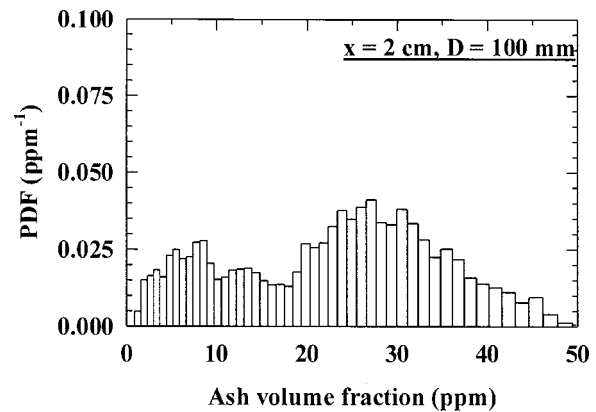


Fig. 7 PDF of ash volume fraction at an axial location 2 cm above the 10 cm diameter pool fire burning D_4

fires, which also have fuel bound oxygen. Radial profiles in the siloxane flame are expected to show steep gradients only near the flame edge.

The time-averaged particulate volume fractions as a function of location on the axis of the D_4 pool fire are shown in Fig. 6. For comparison, measurements from a 10 cm heptane pool fire are also shown [18]. At an axial location 2 cm above the burner, the mean soot volume fraction is less than 0.1 ppm (or $\mu\text{L/L}$) for the heptane pool fire. On the other hand, the mean ash volume fraction at 2 cm above the burner is 26 ppm for the D_4 pool fire. The mean soot volume fraction increases from a value less than 0.1 ppm (± 10 percent) at an axial location of 2 cm to approximately 0.5 ppm (± 10 percent) at an axial location of 12 cm for the heptane pool fire. This is the expected soot structure for a diffusion flame. However, the ash volume fraction decreases from approximately 26 ppm at 2 cm, to 15 ppm at 10 cm for the D_4 pool fire. The existence of high levels of particulate volume fractions and high levels of temperatures close to the surface explains the similarity in the radiative heat loss fractions of silicone and hydrocarbon based pool fires, despite the very low emissivity of silicate ash in comparison to soot. Both the temperature and soot volume fraction profiles confirm that a flame burning siloxane has a very different structure as compared to a flame burning a hydrocarbon fuel.

The probability density function (PDF) of ash volume fraction at a height of 2 cm above the burner for the D_4 pool fire is shown in Fig. 7. The ash volume fractions at 2 cm above the burner range from 2 ppm to 50 ppm, with a mean value of 22 ppm, and a RMS value of 11 ppm. This indicates that the flow is turbulent very close to the burner surface. This behavior is different from hydrocarbon pool fires, where the PDF of soot volume fraction close to the burner surface typically shows a lognormal distribution [26].

Conclusions

The major conclusions of the present study are:

- 1 The specific absorption coefficient of ash generated from flames burning D_4 is 30 times smaller than that of soot generated from flames burning acetylene.
- 2 The specific absorption and extinction coefficients of the ash from fires burning silicone fluids increase significantly with increasing C/Si ratio of the fuel.
- 3 Scattering in fires burning silicone fluids is the dominant mechanism for light extinction.
- 4 The structure of fires burning silicone fluids are different from those burning hydrocarbons in that higher temperatures and particulate concentrations are observed very close to the burner surface. The higher particulate concentrations and higher tempera-

tures in fires burning silicone fluids lead to the same radiative heat loss fractions (as hydrocarbon flames) despite the lower emission coefficient of silicate ash.

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Nomenclature

a_{λ}, m = mass specific spectral absorption coefficient
 c = speed of light
 h = Planck's constant
 I_{λ} = spectral radiation intensity
 k = Boltzmann's constant
 T = temperature
 L = radiation path length
 m = mass concentration of particulate

Greek

ε_{λ} = spectral emissivity
 κ_{λ}, m = mass specific spectral extinction coefficient
 λ = wavelength
 σ_{λ}, m = mass specific spectral scattering coefficient
 θ = polar angle
 ϕ = azimuthal angle
 ρ = particulate density
 τ_{λ} = spectral transmittance

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