

Temperature of Multibubble Sonoluminescence in Water

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Sonoluminescence (SL) spectra were collected from water doped with several organic liquids at low concentrations. Most of the organic substances studied show emission from C₂ and an overall decrease in the intensity relative to SL from pure water. This decrease is due to the consumption by the organic substrates of hydroxyl radicals and other incipient emitting species produced during sonolysis. Small concentrations of carbon disulfide do not lead to emission from C₂ but do cause an increase in SL intensity across the spectral window, most likely due to its own fluorescence. Carbon tetrachloride does not change the intensity of water sonoluminescence but does exhibit C₂ emission. This indicates that the dissociation of carbon tetrachloride inside the cavitation bubble is independent of the products of water sonolysis. Benzene shows the strongest C₂ emission and was studied in the greatest detail. The emission of excited-state C₂ arising from the sonication of benzene/water mixtures at 20 kHz was used to determine an effective emission temperature during cavitation in water. Interband analysis of the two C₂ bands observed during irradiation of water/benzene mixtures at 278 K under Ar indicates an emission temperature of 4300 ± 200 K.

High-intensity ultrasonic vibrations in liquids are accompanied by cavitation: the formation of vapor/gas filled bubbles that pulsate in a highly nonlinear manner. The energy stored during the growth of the bubble in the rarefaction phase of the acoustic field is released when the bubble violently collapses in the positive phase of the acoustic field. This is manifested as acoustic noise, shock waves, chemical reactions, and the emission of light (sonoluminescence, SL).¹ This violent collapse is predicted to generate a hot spot of thousands of Kelvin within the bubble,^{1–5} but there have to date been only a *limited* number of experimental measurements of the temperature of this hot spot.^{6–11}

Although the sonoluminescence of water has been studied for more than 50 years,^{12,13} reliable measurements of the effective temperature during aqueous cavitation remain unresolved. Given the importance of aqueous cavitation to numerous issues (sonography and bioeffects of ultrasound, sonochemical remediation of aqueous pollutants, synthetic applications of sonochemistry, etc.¹⁴), we decided to apply our previous spectroscopic analysis of sonoluminescence from nonaqueous liquids to aqueous solutions doped with small amounts of hydrocarbons. MBSL (multibubble sonoluminescence, light emission from cavitation clouds) spectra from dilute mixtures of organic liquids in water were collected. We have analyzed the emission from excited states of C₂ and find that cavitation in benzene/water solutions at 20 kHz has an effective emission temperature of 4300 ± 200 K.

Sonoluminescence from water was discovered in the early 1930s when Marinnesco and Trillat¹² found that a photographic plate could be fogged in the presence of a cavitation field, and both Frenzel and Schultes¹³ and Zimakov¹³ subsequently observed this emission using the unaided eye. Subsequent

research has been aimed at deciphering the mechanism by which the sonoluminescence occurs. In addition, MBSL has proved to be an important probe of the conditions reached within a cavitating bubble (see reviews¹). Gunter et al.¹⁵ and Srinivasan and Holroyd¹⁶ collected the first low-resolution MBSL spectra from water saturated with different gases. These spectra were reported to consist of only a featureless continuum, leading the authors to suggest that the emission was blackbody radiation at 6000–11 000 K. The work by Taylor and Jarman¹⁷ and Sehgal et al.¹⁸ showed that the MBSL spectra of water in fact contained emission from excited hydroxyl radical in addition to continuum emission, and they suggested that sonoluminescence is due to chemiluminescence as well as incandescence of the bubble contents. Didenko et al.¹⁹ have more recently conducted a series of experiments exploring the parametric dependence of water MBSL and have argued that the emission is due to excited small molecules formed during bubble collapse. This interpretation is not universally accepted, however, and others have proposed that the continuum arises from a plasma created during bubble collapse.

This uncertainty is due, in part, to the lack of definitive measurements of the temperature of cavitation in water. We have collected and analyzed excited-state C₂ MBSL spectra from mixtures of organic liquids in water in order to determine both the mechanism by which the light is generated, and the temperature inside an imploding cavitation bubble. Previous work by Sehgal et al.²⁰ has shown that the intensity of MBSL from water is sensitive to the presence of organic liquids. This report did not include spectra, however, and thus no conclusions could be made as to the identity of the emitting species or the mechanism of sonoluminescence.

Methods

The experimental apparatus (Figure 1) consists of a temperature-jacketed stainless steel cell (15 cm long by 4 cm inner

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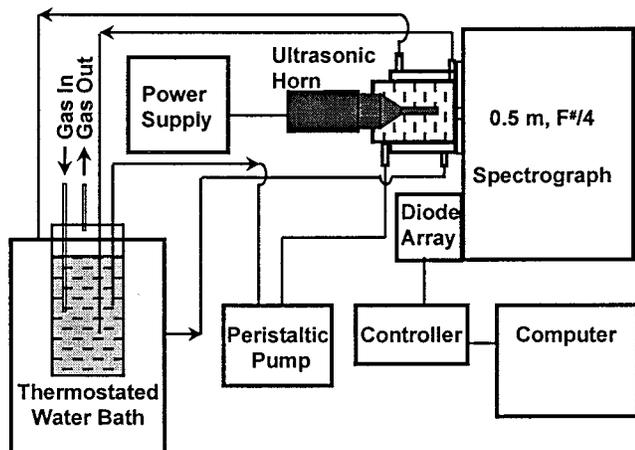


Figure 1. Schematic of experimental apparatus.

diameter) into which a 0.5 in. diameter ultrasonic horn (Heat system model 375, 20 kHz) is inserted. The O-ring at the top of the cell provides a seal between the horn and the cell, and light is collected through a quartz window at the bottom of the cell. The acoustic intensity is 50 W/cm^2 at the horn surface, as determined calorimetrically. The cavitation field was concentrated between the tip of the horn and the quartz window. The liquid was kept in a 1 L glass reservoir placed in a thermostated bath and was continuously pumped through the sonication cell. The thermostated jacket of the cell combined with continuous circulation of the cooled liquid through the cell kept the solution temperature constant at 5°C . Nanopure water (500 mL, 18 S-cm) was saturated with argon for 1 h before addition of the organic substrate. Measured quantities of organic substrates were added to the water in the reservoir, and the mixture was continuously pumped through the cell for another 30 min to allow for the dissolution of the additives. The argon sparge was continued throughout. Liquids used in experiments were either 99% pure and used as received or were purified by standard techniques.

An Acton Research 505F spectrograph with a resolution of 5 nm was used for low-resolution studies of sonoluminescence. An image-intensified, UV-enhanced, 512 element diode array (Princeton Instruments IRY-512 N) served as the detector. Data were collected over 160 nm spectral windows; overlapping data sets were taken in order to cover the entire spectral region. The optical system was spectrally calibrated against NIST-traceable standard lamps: deuterium lamp OLUV-40 (Optronic Lab, Inc.) in the region 200–400 nm and tungsten halogen lamp EH-132 (Eppley Lab) in 350–800 nm. A long pass filter with $\lambda_{\text{cutoff}} \sim 350 \text{ nm}$ was used for data collection at wavelengths above 400 nm in order to eliminate second-order light from the OH^* emission at 310 nm.

Results

As this work is intended to probe the conditions inside cavitation bubbles in water, the first requirement was to find substrates that would yield sufficiently intense C_2 emission for analysis. In addition, the substrate must lead to emission at very low liquid concentrations so as not to perturb the motion of the bubble from that experienced in pure water. In other words, the C_2 precursor must behave only as a probe and not affect the gross physics of cavitation in water. To this end, the following organic substrates were tested as precursors for C_2 formation when added to water at low concentrations: pentane, cyclohexane, benzene, ethanol, *tert*-butyl alcohol, glycerol,

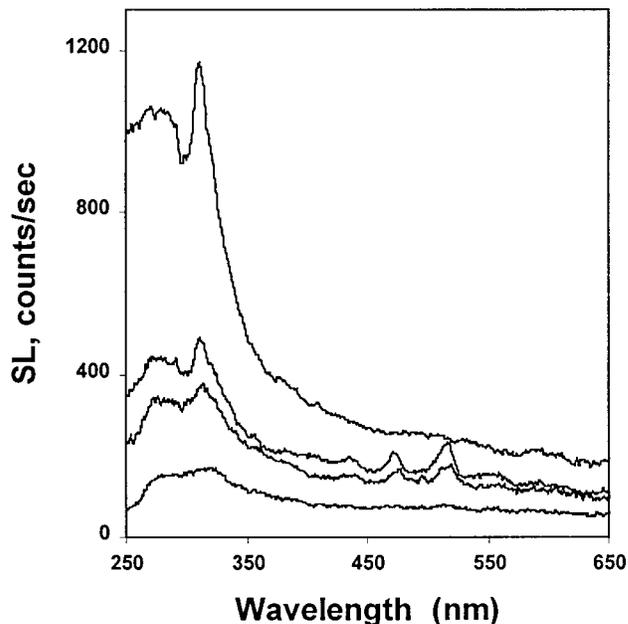


Figure 2. Sonoluminescence spectra of water–benzene mixtures at 278 K under Ar at 20 kHz and 50 W/cm^2 . The spectra from top to bottom are for MBSL from pure water, 0.01% v/v benzene in water, 0.04% v/v benzene in water, and 0.08% v/v benzene in water, respectively.

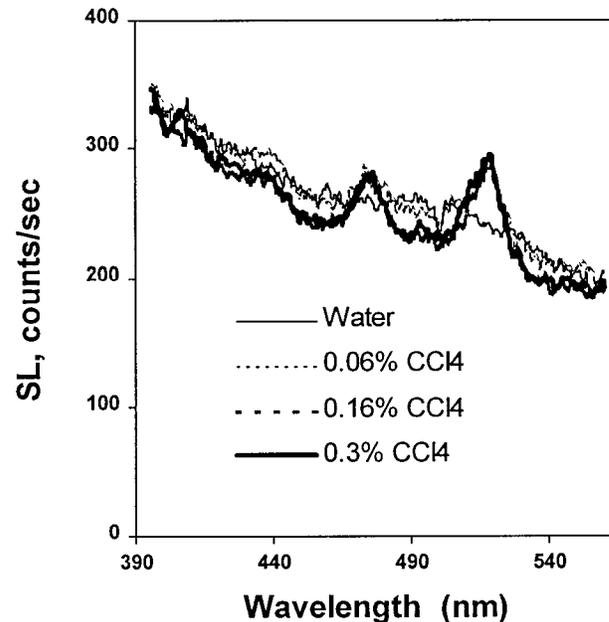


Figure 3. Sonoluminescence spectra of water–carbon tetrachloride mixtures at 278 K under Ar at 20 kHz and 50 W/cm^2 .

carbon tetrachloride, carbon disulfide, and acetone. Most of the substrates led to weak C_2 emission and a drop in the SL intensity of water when added to the solution. Figure 2 shows the representative case of the addition of benzene to water.

Three substrates generated anomalous MBSL results. Glycerol did not exhibit emission from C_2 but did quench MBSL. Carbon tetrachloride did not change the intensity of water sonoluminescence in this region, but C_2 emission was clearly present in the spectra (Figure 3). Finally, the addition of CS_2 to water increased the SL intensity, but no C_2 emission was observed (see Figure 4).

The strongest C_2 emission among the substances studied arose from the irradiation of water/benzene mixtures, although it

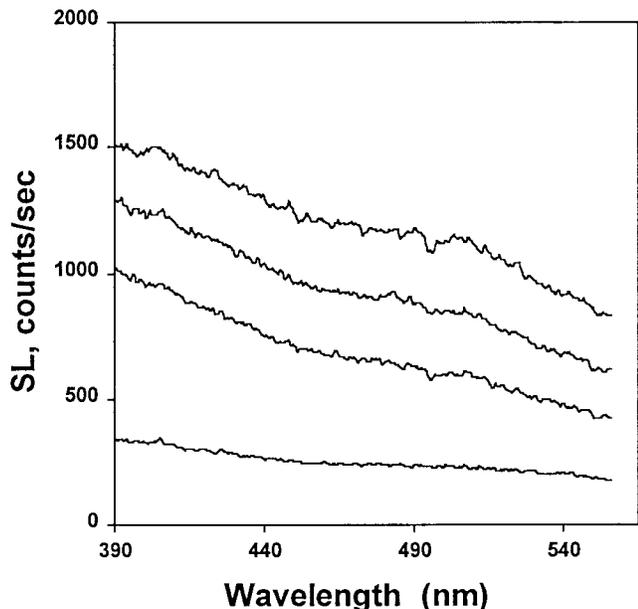


Figure 4. Sonoluminescence from water-carbon disulfide mixtures at 278 K under Ar at 20 kHz and 50 W/cm². The spectra from bottom to top are for MBSL from pure water and for 0.02, 0.04, and 0.08% CS₂ in water, respectively.

should be noted that the emission was very weak relative to C₂ emission from neat silicone oil or long chain hydrocarbons. The SL spectra of water and water/benzene mixtures are presented in Figure 2. The intensity of SL decreases across the entire spectral region as the concentration of benzene is increased. It is interesting that the emission from both C₂ (470 and 515 nm) and OH (310 nm) are significantly suppressed at a benzene concentration at or above 0.08%.

The C₂ emission from water/benzene mixtures was used to estimate the temperature of cavitation following the method of Flint and Suslick.^{8,21} They compared the MBSL spectra of silicone oil saturated with argon to synthetic spectra calculated using the well-understood theory of diatomic emission. Synthetic spectra for C₂ emission as a function of temperature were generated for the thermally equilibrated case, as shown in Figure 5; details of these calculations are given elsewhere.^{8,21} The high pressures associated with the cavitation event, however, prevents resolution of individual rovibronic lines, so vibrational and rotational temperatures were obtained by fitting the shape of each of the two dominant C₂ bands and by comparing the relative intensities of these two bands. The spectrum of C₂ emission from the dilute benzene solution in water is shown at higher resolution in Figure 6. We find that C₂ emission from the aqueous mixtures is significantly less intense than MBSL from neat silicone oil. For this reason, our temperature determinations are made by comparison of the relative intensities of the two most intense C₂ bands (the $\Delta\nu = 0$ and $\Delta\nu = +1$ bands, as shown in Figure 6). This gives an effective emission vibrational temperature of 4300 \pm 200 K.

Discussion

Sonoluminescence is a consequence of cavitation: the formation and collapse of bubbles in a liquid. Cavitation nuclei are always present in a liquid, and these grow when acted upon by the rarefaction phase of the acoustic field. As a bubble grows, its internal pressure is maintained by both vaporization of the surrounding liquid and by diffusion of dissolved gases into the bubble. When the acoustic pressure becomes positive, the bubble

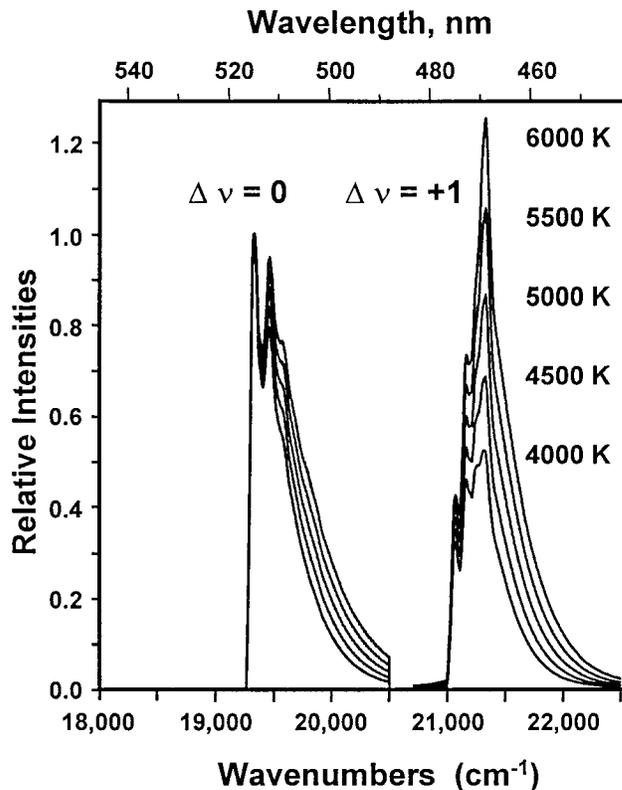


Figure 5. Calculated emission spectra comparing the intensities of $\Delta\nu = 1$ to $\Delta\nu = 0$ bands of the $d^3\Pi_g - a^3\Pi_u$ transition (Swan bands) of C₂; modified from ref 19.

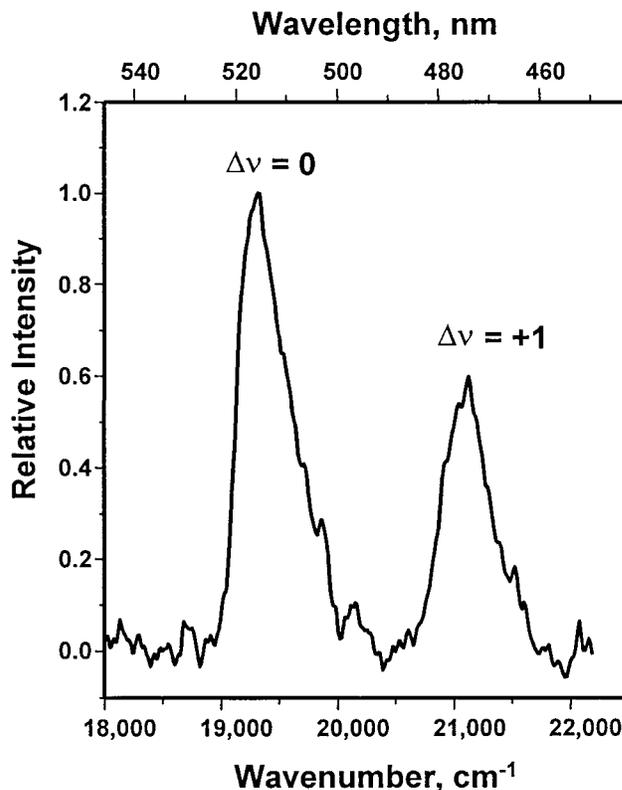


Figure 6. Sonoluminescence spectrum of water-0.01% benzene mixture at 278 K under Ar at 20 kHz and 50 W/cm². Comparison of the intensities of $\Delta\nu = 1$ to $\Delta\nu = 0$ bands of the $d^3\Pi_g - a^3\Pi_u$ transition of C₂ gives an estimated emission temperature of 4300 \pm 200 K.

is compressed. During this compression the vapor recondenses at the interface, and the gas redissolves into the liquid. At high

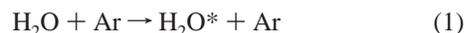
enough acoustic intensity the bubble pulsations become non-linear and the bubble collapses violently during the positive pressure phase of the acoustic field.

The first stage of bubble compression is isothermal. The heat generated by the compression of the bubble contents and by vapor condensation at the bubble wall is dissipated via the thermal diffusivity of the gas–vapor mixture inside the bubble and by the thermal conductivity of the surrounding liquid. The next stage of bubble compression is nearly adiabatic, and heat is produced by bubble compression faster than it can be dissipated in the surrounding liquid through thermal conductivity. This adiabatic collapse is violent enough to heat the bubble content to temperatures of 5000 K in nonaqueous liquids.^{6–8}

There have been few attempts in the literature to estimate cavitation temperatures in water. Seghal and co-workers used MBSL from aqueous alkali metal salt solutions in an attempt to probe the temperature and pressure inside a cavitation bubble.⁹ In particular, they used the broadening and shift of sodium and potassium lines in the SL spectra of water–salt solutions to estimate the relative density of the cavitation bubble contents. Note here that relative density in spectroscopy means the ratio of the density at experimental conditions to the density of the same gas composition at 0 °C and 1 atm. From their measured relative density of 40, they calculated the compression ratio required to attain that relative density, where the compression ratio is the ratio of the bubble radii at the stage when the *adiabatic* compression starts vs when it is complete. Seghal and co-workers then calculated the resultant temperature and pressure inside a hypothetical bubble undergoing that extent of adiabatic compression and gave estimates of 3360 ± 330 K and 313 ± 50 atm.⁹ They made the implicit assumption, however, that the conditions inside the bubble when adiabatic compression begins are 1 atm and 285 K. While those conditions may represent conditions in the bubble at the beginning of collapse, the initial stage of bubble compression is isothermal. Unfortunately, in these measurements, the point at which the compression becomes adiabatic is not known:²² thus, no accurate estimate of final temperature and pressure are possible through this approach. They made the further assumption that argon was the sole collisional partner for the metal; this is highly unlikely, given the high vapor pressure and large collisional cross section of water. In addition, their method for determining the full width at half-maximum (fwhm) of the metal lines and the shift in wavelength of those lines was not precise due to the high asymmetry of lines and low resolution of the optical system. Finally, since the vapor pressure of alkali metal ions is negligible, the location of the emission from excited-state metal atoms may or may not be coming from the gas phase of the cavitating bubble. For all these reasons, the temperature and pressure reported by Seghal and co-workers⁹ should be considered as qualitative measurements, indicating only the existence of high temperatures and pressures inside the bubble.

Another approach was taken recently by P. Riesz and co-workers, who analyzed isotope effects in the formation of radicals during aqueous sonolysis.¹⁰ In particular, they used spin traps to study the relative rates of O–H and O–D bond cleavage during ultrasonic irradiation of argon-saturated H₂O and D₂O mixtures. Unfortunately, as the authors note, there are large error limits intrinsic with this technique. They found that the temperature inside the bubble was between 2000 and 4000 K, depending on the spin-trap used. In addition, Hoffmann and co-workers have recently estimated the interfacial temperature during remediation of nitrophenols,¹¹ but these results do not probe the gas phase of the collapsing bubble.

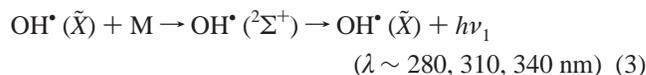
Water Sonoluminescence. The kinetic energy of particles inside the bubble increases throughout the course of the bubble collapse, and it can be converted into internal energy by means of inelastic collisions:



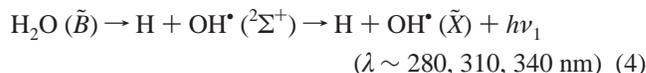
Water molecules can be excited into various rotational, vibrational, and electronic states via inelastic collisions (eq 1): $\tilde{A} \ ^1\text{B}_1$ (energy of excitation $E_v \sim 7.5$ eV), $\tilde{B} \ ^1\text{A}_1$ ($E_v \sim 8.3$ eV), and $\tilde{C} \ ^1\text{B}_1$ ($E_v \sim 9.9$ eV).²³ If enough energy is absorbed by a water molecule during such collisions, it will dissociate, giving a hydrogen atom and a hydroxyl radical in the ground or excited states.²³ For example, the \tilde{A} state is repulsive and H₂O (\tilde{A}) dissociates to give hydrogen atoms and OH radicals in the ground electronic state:



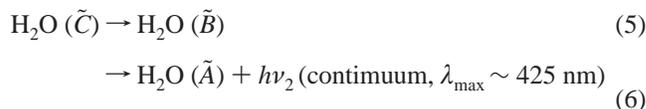
The resulting hydroxyl radicals and hydrogen atoms can initiate chemical reactions both inside the bubble and in the liquid after bubble collapse. Hydroxyl radicals in the ground state can also be directly excited by collisions to give an emission with a maximum at 310 nm:



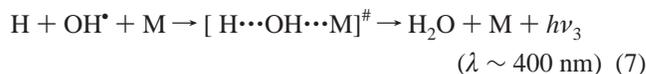
Dissociation of H₂O ($\tilde{B} \ ^1\text{A}_1$) also gives excited hydroxyl radical OH* ($^2\Sigma^+$):



Water molecules excited to the $\tilde{C} \ ^1\text{B}_1$ state can relax through either \tilde{B} or \tilde{A} states:

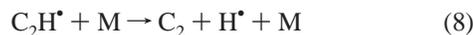


The spectra of water sonoluminescence consists of both emission from OH* radical at 280, 310, and 340 nm (eq 3) and a continuum of uncertain origin. One possible source of continuum is shown in eq 6, with emission in the region from 380 to 600 nm and a maximum at 425 nm.¹⁹ Another possibility is the recombination outlined in eq 7, where # denotes a vibrationally excited complex.^{18,24}

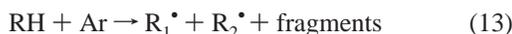
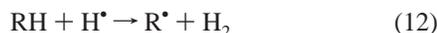
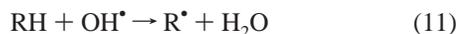


SL from Water/Organic Mixtures. Almost all the organic liquids examined exhibit weak C₂ emission as a feature of the sonoluminescence spectra when added to water. At the same time, the overall intensity of SL in the spectral region under study decreases. This is most likely due to the consumption of H atoms and OH radicals through reactions with the organic species, thus decreasing the likelihood of radiative recombination (eq 7). Another possibility is collisional quenching of emission from the excited-state water molecules by the organic substrates and, consequently, a decrease in the intensity of sonoluminescence across the entire region.

C₂* formation inside the cavitation bubble could be due to the following reactions, which have been documented in shock wave studies:²⁵



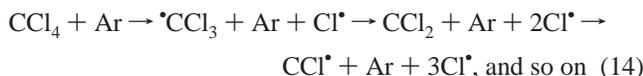
The initial steps in the formation of C₂ are the reactions of substrates with H or OH radicals or direct destruction of the organic substrates by collisions with Ar:



Fragment recombination (eqs 8–10) can form excited-state C₂. The complete set of possible reactions of organic substances inside the cavitation bubble, and in the solution after bubble collapse, is very complex (see, for example, recent papers on sonochemistry of water–organic mixtures^{26–29}) and will not be considered here.

Sonoluminescence intensity decreases upon addition of small amounts of benzene to water. At benzene concentration above 0.08%, both C₂ emission and OH* emission disappear (Figure 2). One possible explanation of this effect is that at high intrabubble concentrations of benzene, the concentration of OH is insufficient to completely oxidize benzene to the short fragments required to produce C₂ emission. The reaction is limited to a few initial steps, OH* is consumed with no production of C₂, and the intensity of both OH* emission and C₂ emission are consequently diminished. Note here that the absence of OH* emission at a high concentration of benzene in solution confirms the importance of the reaction in eq 3 in the gross scheme of water sonoluminescence.

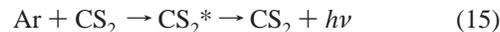
Formation of C₂ from Water/CCl₄ Mixtures. Interestingly, the intensity of sonoluminescence relative to that of pure water does not decrease upon the addition of small amounts of carbon tetrachloride (Figure 3). We take this to mean that the reactions of carbon tetrachloride inside the bubble do not compete with recombination (eq 7) and that CCl₄ and its decomposition products do not quench the emission of water in this region. Carbon tetrachloride can thus be destroyed through a process analogous to eq 13:



The subsequent radical recombinations and transformations will occur both inside the bubble and in the solution after bubble collapse.²⁶ The formation of C₂ probably occurs through reactions of C and CCl_x fragments in a manner similar to that suggested above (eqs 9 and 10) for CH radicals.

Sonoluminescence from CS₂/Water mixtures. The addition of small amounts of CS₂ to water increases SL intensity relative to that of pure water in the region studied, but no C₂ emission is observed (Figure 4). The increase in SL intensity can be explained by the known fluorescent emission from CS₂ in this region, which exhibits structureless emission from 350 to 580 nm with a broad maximum around 450 nm,³⁰ similar to the CS₂/water MBSL spectra reported here. The increase in SL intensity

can thus be accounted for by direct excitation of carbon disulfide inside the cavitation bubble during its compression:



Note here that CS₂ can be destroyed sonochemically, giving elemental carbon and sulfur.³¹ It is possible that some degree of C₂ emission is present in the spectra but that it is masked by CS₂* luminescence.

Estimation of Temperature Inside the Bubble. Flint and Suslick⁸ used medium-resolution spectra of C₂ emission arising from irradiation of silicone oil (silicone oil was used because of its strong emission) to determine the temperature within sonoluminescing bubbles. Experimental spectra were compared with spectra calculated using the well-understood theory of diatomic molecular emission.³² Pressure broadening of the rovibronic lines made it impossible to resolve individual transitions, and Spier's method³³ for overlapping dense spectral bands was used to generate synthetic spectra. The relative intensity of each rotational line was calculated for a given temperature, and the contribution of each line to the spectrum was weighted by a triangular aperture function that reflected the resolution of the spectrometer and the broadening of the lines. There were thus three adjustable parameters for the calculation of each spectrum: the vibrational and rotational temperatures and the spectrometer aperture function. Spectra were generated for the thermally equilibrated case: details of these calculations are published elsewhere.^{8,21}

The intensity of C₂ emission from water–benzene mixtures is much weaker than that from silicone oil, and we were limited to a comparison of the interband intensities of the Δν = 0 and Δν = +1 bands, which vary substantially as a function of temperature (Figure 5). The observed MBSL spectra after correction and baseline subtraction are presented in Figure 6. Comparison with the calculated spectra²¹ gives a temperature of 4300 ± 200 K. This is significantly lower than that observed from silicone oil (5080 ± 160 K),⁸ which is explained by the higher vapor pressure of water (6.6 Torr) compared to silicone oil (<0.01 Torr). The adiabatic index (i.e., C_p/C_v) of water is substantially lower than that of argon, and the rotational and vibrational modes of water molecules in the cavitating bubble serve as heat sinks for the energy deposited in the bubble during its collapse. In addition, as the bubble is heated, dissociation begins to occur, and this endothermic process absorbs a great amount of energy. The energy consumed by all these processes is thus unavailable for raising the temperature of the bubble contents.

The interpretation of this spectroscopic temperature is not without complications. The temperature within an imploding bubble will have both a temporal and a spatial profile, and the emission spectra will thus vary as a function of time during collapse and the site of emission within the bubble. The overall emission will be dominated by the hottest portion of this cycle. The effective emission temperature obviously does not provide a unique parameter with which to describe this complex profile. Furthermore, C₂ is the product of a complicated set of chemical reactions, and the initial distribution of states might reflect the thermodynamics of this chemistry rather than the conditions within the bubble. Unlike a flame, however, the contents of a cavitation bubble are subjected to high pressures and collisional frequencies. Thus, it is likely that C₂* reaches thermal equilibrium during its lifetime. These issues have, in fact, been previously discussed in the literature.³⁴

It has also been argued that the dissociation energy of C₂ provides an upper limit to its use for spectroscopic thermometry: since C₂ is destroyed at temperatures not too much above

5000 K, this could be an upper limit to the temperature range for C₂ thermometry.³⁵ It has been argued that the interior of the bubble reaches temperatures well in excess of 5000 K and that the continuum in MBSL spectra is due to emission from the resulting plasma.³⁶ Recent work in this laboratory, however, has confirmed the 5000 K temperature in low-volatility solvents using metal atom emission, which is not prone to dissociative effects.⁶ Even more importantly, the spectroscopic temperatures for cavitation in water reported here are well below any possible effects from dissociation of C₂.

We are of the opinion that this spectroscopically determined temperature reflects the conditions inside a cavitation bubble collapsing in water. The concentration of benzene in solution is very low (~0.01%), and the vapor pressure of benzene inside the bubble should be negligible compared to that of the water. It is therefore unlikely that the presence of benzene would perturb the bubble dynamics relative to that of pure water. We also note that the kinetic isotope effect studies of Misik and co-workers,¹⁰ while giving only a broad range of possible cavitation temperature (between ≈2000 and ≈4000 K) are in reasonable agreement with our results, despite the vastly different techniques employed.

Conclusions

Small amounts of organic liquids in water can give rise to C₂ emission while concurrently decreasing the overall MBSL intensity. It is proposed that this is due to the consumption of OH radicals and H atoms by reactions with the organic substrates. This is a general phenomenon, though there are some exceptions. Carbon disulfide does not yield C₂ emission, but the intensity of SL increases, probably due to fluorescence from known excited states of CS₂. Carbon tetrachloride does not change the intensity of water sonoluminescence but does yield C₂ emission. This indicates that carbon tetrachloride can directly dissociate inside the cavitation bubble without reacting with the products of water dissociation.

The temperature of C₂ emission from the cavitation bubble is 4300 ± 200 K, which is significantly lower than that observed from pure silicone oil. We believe this is due to the high vapor pressure of water relative to that of silicone oil. The vapor serves to decrease the adiabatic index (C_p/C_v) of the bubble contents. The dissociation of water molecules decreases the energy of bubble compression available for the heating of the bubble interior. All these factors reduce the temperature reached inside the bubble.

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