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Soot formation during combustion of unsupported methanol/toluene mixture droplets in microgravity

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An experimental study is reported concerning the influence of liquid composition on soot formation and burning rate of a droplet composed of a binary miscible mixture of liquids. The mixture components represented a highly sooting fuel, toluene, and a non-sooting fuel, methanol. The experimental observations were made in a microgravity environment to create near spherically symmetric burning of the droplets. Mixtures of 5%, 25% and 50% by volume toluene in methanol were burned in room temperature air. Initial droplet diameters ranged from 0.47 mm to 0.60 mm, and the available experimental time was sufficient to record the complete droplet burning history.

Toluene concentration in methanol was shown to dramatically influence flame luminosity and soot production. However, neither burning rates nor propensity for flame extinction appeared to be significantly affected by toluene mixture fractions. 5% toluene mixture droplets behaved like pure methanol droplets in terms of burning rate, lack of flame luminosity, and extinction. Increasing the toluene concentration in the droplets to 25% increased flame luminosity, yet no visible soot agglomerates were observed. The 50% mixture droplets, however, burned with highly luminous flames and large amounts of soot agglomerates collecting inside the flame. None the less, all the mixture droplets showed similar burning rates to those of pure methanol and likewise exhibited flame extinction before complete droplet vaporization.

1. Introduction

The non-luminous nature of alcohol flames and the highly sooting luminous flames that are produced when burning aromatic liquids suggest the possibility of controlling flame luminosity and particulate formation by mixing these two classes of liquids. On one hand, alcohol flames exhibit very low, and in the case of methanol, almost no visible radiation flux so that these flames may be difficult to perceive by human senses and thus potentially hazardous. This problem can be particularly important in connection with the use of alcohols as a vehicle fuel. Doping an alcohol with another fuel that burns with extensive soot radiation may produce a flame which provides luminosity for visibility and yet maintains the relative lack of soot emissions of alcohol flames. On the other hand, alcohols may also be an effective additive to aromatics, which are an important class of hazardous waste liquids. Disposal of liquid aromatics by incineration can pose a challenge because of their propensity to produce soot particulates (Glassman 1987) in the incineration process.

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Some of the environmental concerns of the incineration process might be mitigated through reducing particulate formation by burning the aromatic in the presence of an alcohol.

The present study was undertaken to examine the burning characteristics of droplets which are mixtures of an alcohol and an aromatic, in this case, methanol and toluene. Any influence of fuel composition on flame luminosity was expected to be pronounced for these mixture droplets because methanol droplet flames produce virtually no soot (Yang *et al.* 1990) and toluene droplet flames are extremely sooty (Avedisian *et al.* 1988). Also, the ease of finding or calculating the physical properties of methanol, toluene, and their mixtures allows for the possibility of developing models for the combustion of these mixture droplets. The droplet flame provides a fundamental configuration for approaching problems pertaining to spray-fired incineration and perhaps automotive applications.

Prior studies of the burning characteristics of methanol and toluene mixtures have examined composition effects on flame radiation for wick flames and pool fires (Sorek *et al.* 1984, 1985, 1986; Anderson *et al.* 1985). This work revealed a significant increase with toluene concentration of both flame luminosity and soot formation. Pool fire luminosity was found to vary with time, and this behaviour was conjectured to be the result of one mixture component preferentially vaporizing and enriching the pool with the remaining component. Such preferential vaporization can occur in droplet combustion of multicomponent mixtures and may influence such factors as the propensity for extinction and microexplosions as shown in a previous study involving the combustion of methanol/dodecanol mixture droplets (Yang *et al.* 1990).

The present experimental method endeavoured to create an environment in which droplet combustion would be spherically symmetric. To this end, the experiments were performed in a buoyancy-free environment with the test droplets being nearly stationary and free-floating. In this way, the droplets would burn without the complicating effects of asymmetries induced by axial vapour flows around the droplet. Such flows may create internal liquid circulation within the droplet and reduce the ability to observe soot inside the flame by sweeping soot precursors and/or particles through the flame before significant soot agglomeration can occur. In addition, data obtained from droplets burning in a buoyancy-free environment can, in general, be compared with models in which spherical symmetry is assumed for computational simplicity (Spalding 1953). No such comparisons are made in the present study, however, because no droplet combustion model has yet been developed which accounts for the presence of soot around the droplet. Soot around the droplet may affect heat transfer to the droplet and thus the droplet burning rate (Jackson *et al.* 1991).

Gravity was the parameter through which buoyancy effects were minimized in the present experiments. The experiments were performed in a drop tower to create a microgravity environment. The droplet burning process can be spherically symmetric if the droplet diameter and/or gravity level (which are factors that determine the magnitude of the Grashof number) are low enough. In the present experiment, initial droplet diameters ranged from 0.47 to 0.60 mm. For initial droplet diameters in this range, buoyancy effects were minimal at the low gravity levels in the moving frame of reference of the present experiments (about 10^{-3} that of Earth's gravity).

No previous studies have addressed the influence of liquid composition on soot formation in the combustion of miscible mixture droplets in a microgravity

Figure 1

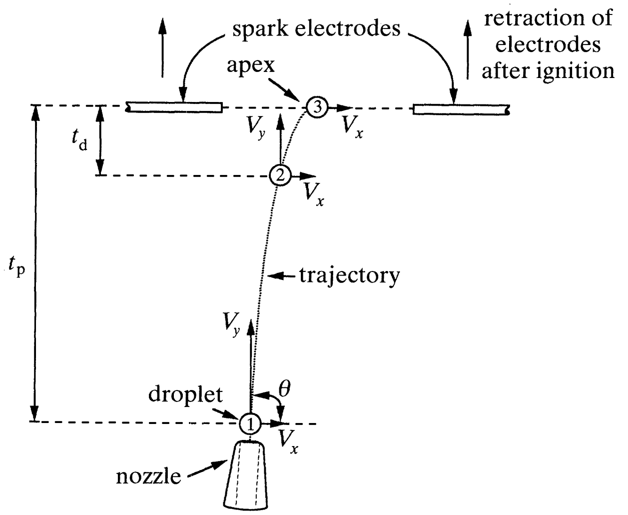

Figure 2

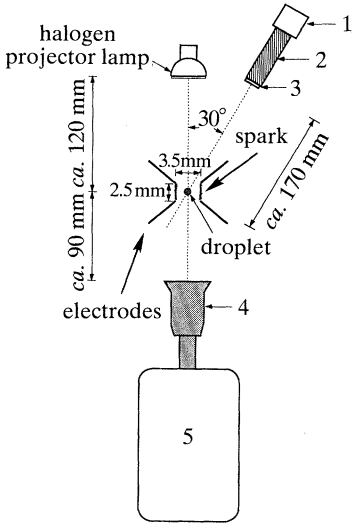

Figure 1. Schematic of the droplet trajectory which shows the apex at the plane of the electrodes where the droplet is levitated. t_p, the time for the droplet to reach its apex, and t_d, the delay time for the package to separate from the magnet, must be known to successfully levitate the droplet.

Figure 2. Top view (not to scale). 1, Cohu CCD camera model 6800; 2, Bausch & Lomb MonoZoom-7 lens; 3, 0.5× objective; 4, Cosmicar lens (75 mm, f/1.4); 5, Millikan DBM-55 movie camera.

environment. This study demonstrated the effect of composition on soot formation in the burning of isolated and stationary alcohol/aromatic mixture droplets by examining mixtures with initial compositions of 5%, 25% and 50% by volume toluene in methanol, which correspond to methanol mole fractions of 0.98, 0.89, and 0.72, respectively. This range of concentrations illustrated a dramatic effect of composition on soot production.

2. Description of the experiment

To create a low gravity environment for droplet combustion, the experiments were performed in a drop tower aboard a free-falling package. The experiment involved releasing into free-fall the combustion chamber and associated optical instrumentation. During the period of the fall, the droplet was ignited and its burning history recorded by a 16 mm high speed cine camera using direct backlighting from a 13 V halogen lamp. The camera was run at framing rates between 200 and 400 frames per second. The period of observation was about 1.2 s, long enough to observe the combustion history of the droplets, and was terminated when the drop package impacted an air-tunnelled foam shock absorber. The droplets were burned in a chamber charged with room temperature air at 0.101 MPa and with a relative humidity of about 30%. The importance of the relative humidity resides in the influence of water vapour condensation on the droplet burning rate and extinction.

Details of the experimental apparatus and methodology have been reported elsewhere (Avedisian *et al.* 1988; Yang 1990; Yang *et al.* 1990). For a brief description, droplets formed by a variant of the so-called 'ink-jet' method at a frequency of 2 Hz were propelled upward in a near vertical trajectory to produce a

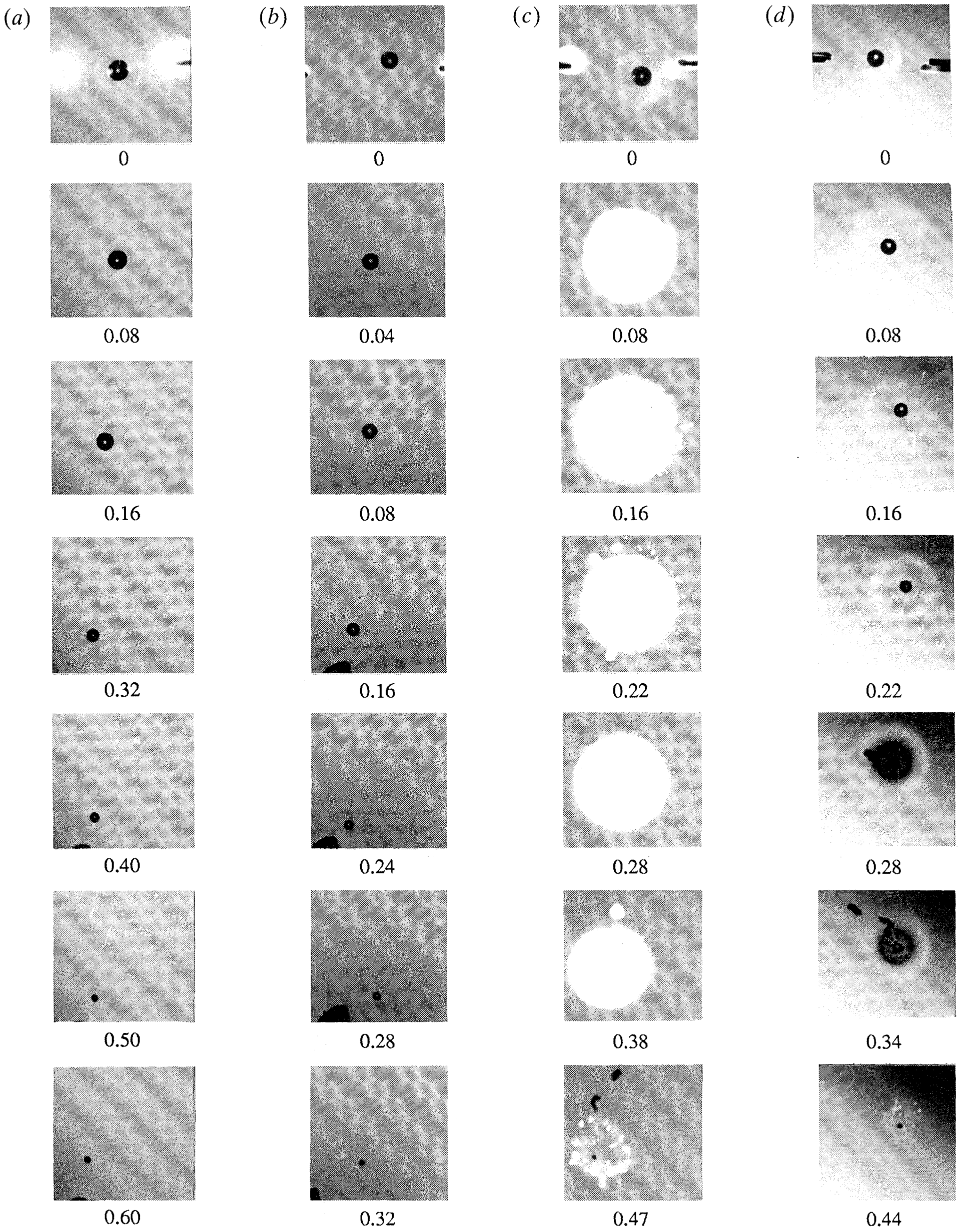

Figure 3. Methanol/toluene mixture droplets burning at low gravity. (*a*) 5% toluene by volume, $D_0 = 0.57$ mm, normal backlighting; (*b*) 25% toluene by volume, $D_0 = 0.48$ mm, faint backlighting; (*c*) 50% toluene by volume, $D_0 = 0.55$ mm, normal backlighting; (*d*) 50% toluene by volume, $D_0 = 0.52$ mm, intense backlighting.

steady stream (figure 1). The droplet generator was then turned off and the instrumentation package was released into free-fall when the final droplet reached the apex of its trajectory. This droplet was ignited about 15 ms after release of the package by two pairs of electrodes, placed on opposite sides of the droplet and subsequently retracted away from the flame by a solenoid-activated spring

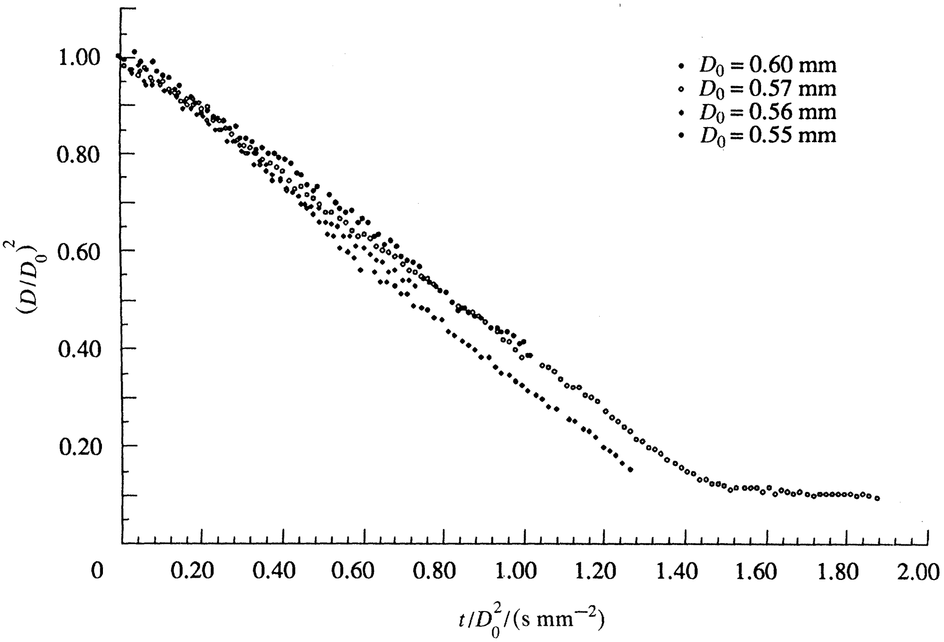

Figure 4. 5% toluene in methanol droplets.

mechanism (Yang 1990). The spark duration ranged from 12 to 15 ms. Figure 2 illustrates the relative placement of the electrodes and the optical set-up. A CCD video camera, mounted on board the instrumentation package, aided in positioning the droplet stream before an experiment and also provided in-flight views of the droplet.

A frame-by-frame analysis of the motion picture sequences yielded information about the burning characteristics of the droplets, including the evolution of droplet diameter and qualitative information about flame luminosity and the soot shell structure around the droplet. Data extraction from the movie record was performed with a computer-based image analysis system (Automatix Inc., Billerica, Massachusetts). The film images were magnified through a microscope and captured by a CCD camera which sent the image to the computer. The software package calculated an average diameter for the droplet after determining the droplet boundary with a threshold grey level. This technique worked well except when dense soot shells surrounded the droplet and soot agglomerates did not allow for the entire droplet boundary to be identified. For such cases, droplet diameters were determined every fifth frame by manually identifying the vertical and horizontal diameters as described by Yang *et al.* (1990). The two methods were compared for the time domain where soot did not obscure the droplet boundary and showed very good agreement. The two methods also were previously shown to provide consistent burning rates for unsupported *n*-heptane droplets burning at low gravity (Jackson *et al.* 1991).

The ability to observe the droplet through heavily sooting flames was found to depend on the intensity of the backlighting. Intense backlighting tended to overpower soot radiation and flame luminosity to provide clear images of the droplet and soot structures inside the flame. For very low backlighting, the droplet image was usually obscured by soot radiation in the highly sooting flames. Examples of the effect of backlighting intensity on droplet/soot/flame images are illustrated in figure 3 (discussed in §3). The photographic sequences labelled 'normal' and 'intense' for

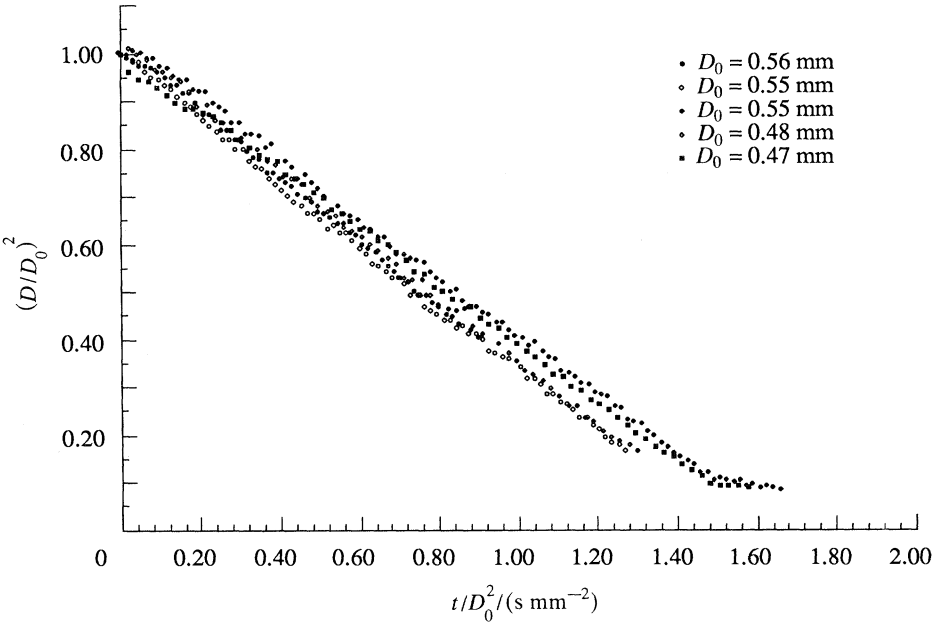

Figure 5. 25% toluene in methanol droplets.

the 50% toluene in methanol mixture droplets (figure 3*c* and *d*) illustrate the influence of backlighting on the recorded image for the same mixture. A comparison between photographs taken with similar backlighting provided a qualitative measure of the influence of liquid composition on soot radiation intensity.

3. Discussion

Combustion of droplets containing initially 5% by volume toluene (figure 3*a*) was found to be similar to that of pure methanol (Yang *et al.* 1990) in terms of burning rates and flame luminosity. The average burning rates were within 5% of those of similar sized pure methanol droplets. The flame lacked the luminosity associated with soot particle radiation. As with methanol, flame extinction was consistently observed for this mixture and was thought to have occurred when the movie record began to show almost no further change in droplet size, as brought out in the last two photographs of figure 3*a*. The measured droplet diameters at the period of negligible vaporization ranged from 180 to 200 μm. The mechanism for flame extinction in methanol droplet combustion is suspected to be water accumulation in the liquid phase (Choi *et al.* 1990), and thus extinction of these mixture droplets suggests that the small concentrations of toluene added to methanol do not suppress water vapour condensation at the liquid surface.

The evolution of droplet diameter is displayed in figure 4 in the form suggested by the classical quasi-steady droplet burning theory (Spalding 1953):

$$d^2 = d_0^2 - Kt, \tag{1}$$

where d and d_0 are the instantaneous and initial droplet diameters respectively, t is time, and K the so-called burning rate. Figure 4 shows that for the 5% toluene in methanol droplets, this linear variation is obeyed for a large fraction of the burning history. The linearity, however, does not necessarily imply quasi-steady burning, which also requires a constant d_f/d (where d_f is the instantaneous flame diameter). The repeatability of the measurements indicates uniform burning conditions from run to run. Extinction is indicated by a relatively constant $(d/d_0)^2$ at the end of burning.

In contrast to the 5% mixture, the 25% toluene mixture flame became visible as burning proceeded. The luminosity of the flame could only be detected by reducing the intensity of the backlighting (labelled 'faint backlighting' in figure 3*b*) from the 'normal backlighting' in figure 3*a*. Though no soot agglomerates were observed, radiation in the visible characteristic of soot and its precursors became apparent for a portion of the burning history. The flame became visible about 0.08 s after ignition in figure 3*b* and then disappeared near the end of burning. This variability in flame luminosity suggests a delay period before significant amounts of soot and/or soot precursors developed in the gas phase to provide significant visible radiation. Such a 'delay' may be explained by a preferential vaporization mechanism where methanol, which burns without producing soot, initially dominates the vaporization. As methanol was preferentially depleted from the liquid surface, the fractional vaporization of toluene eventually increased to promote soot formation in the gas phase.

Figure 5 shows the evolution of normalized droplet diameter over the complete burning history of droplets containing 25% toluene in methanol. The near linearity of the variation and repeatability of the measurements is apparent. Flame extinction was consistently observed for this concentration as evidenced by the constant $(d/d_0)^2$ for $t/d_0^2 > 1.5$. Extinction diameters for this mixture were in the range of 150 to 180 μm, values similar to that of pure methanol. The occurrence of extinction for these mixture droplets suggests an ineffectiveness of toluene as an additive to methanol for suppressing water vapour absorption at the liquid surface.

Initial diameters for the droplets containing initially 25% by volume toluene in methanol varied from 0.47 to 0.56 mm for the data displayed in figure 5. The movie records of the burning of those droplets revealed differences in flame luminosity that suggest an influence of initial droplet diameter on soot formation. Flames surrounding the smaller droplets appeared less luminous than those surrounding the larger droplets. Figure 6 illustrates two series of photographs, one of the smaller (figure 6*a*) and one of the larger (figure 6*b*) droplets studied for this mixture. All conditions were the same for the two droplets shown in figure 6; in particular the backlighting was the same for the two photographic sequences. Comparing the flame luminosity at $t = 0.08$ s and $t = 0.16$ s shows that the flame was more luminous for the larger droplet than for the smaller. This indicates more soot radiation in the flame of the larger droplet, which may have resulted from an increase in soot production.

It has been demonstrated that for spherically symmetric quasi-steady droplet combustion, average residence times of fuel molecules in the gas phase between the droplet and its flame are proportional to d^2 (Jackson *et al.* 1991). Thus larger droplets allow more time for fuel molecules to break down and form soot precursors and nucleate soot particles. Such an influence of droplet size may explain the increase in flame luminosity (i.e. soot radiation) of the larger droplets of the 25% mixture. Nevertheless, the influence of initial droplet diameter for the 25% mixture on flame luminosity does not apparently impact the evolution of droplet diameter as shown in figure 5, at least within the resolution of the present data acquisition arrangement. The low amount of soot produced in these flames did not significantly affect the measured burning rates. The influence of soot formation (and other factors which

Figure 6

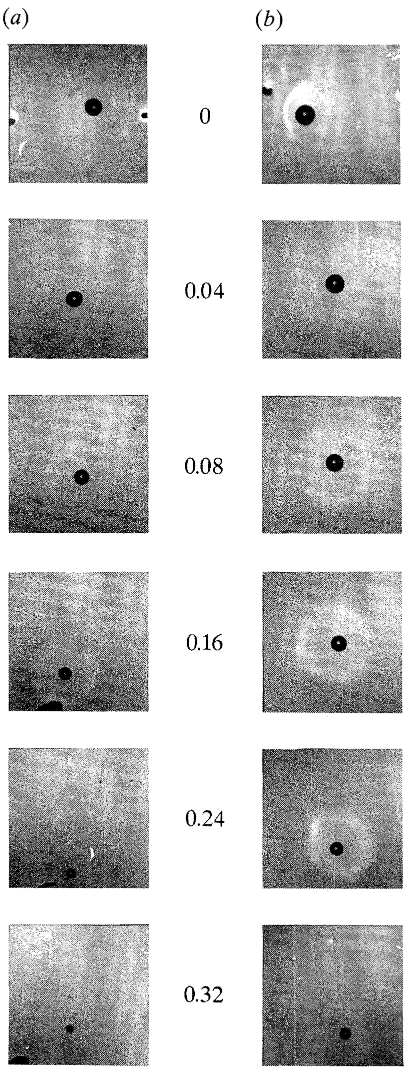

Figure 7

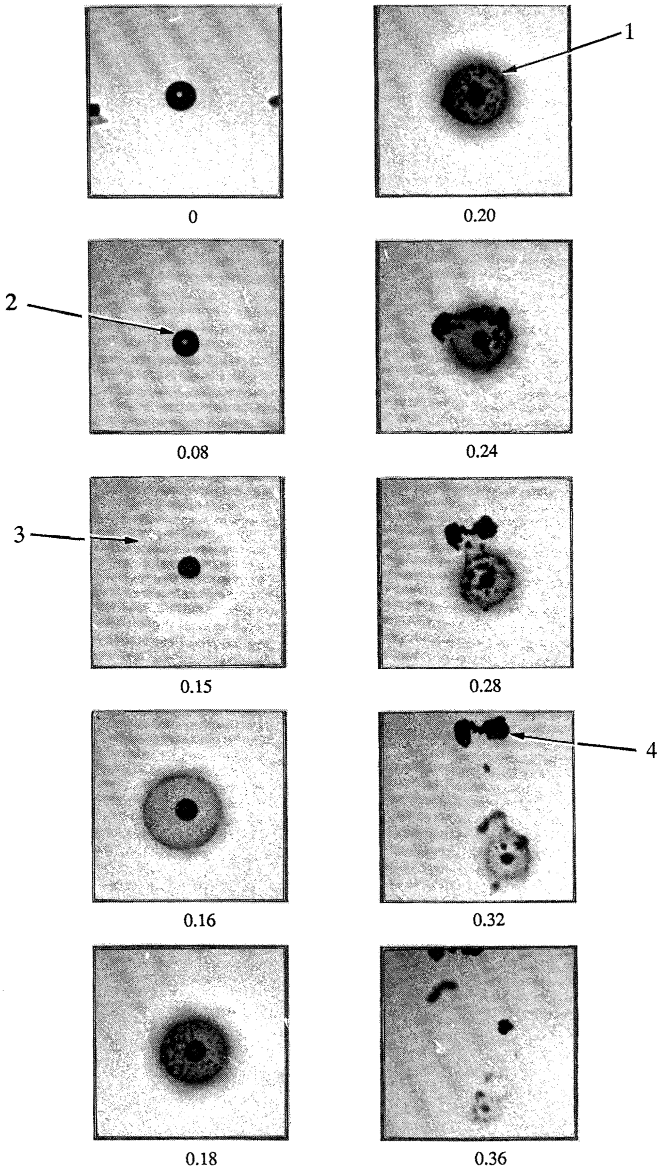

Figure 6. 25% v/v toluene/methanol droplets burning. (*a*) $D_0 = 0.48$ mm; (*b*) $D_0 = 0.55$ mm.

Figure 7. 50% v/v toluene/methanol droplet burning at low gravity. $D_0 = 0.49$, intense backlighting. 1, soot shell; 2, droplet; 3, luminous flame front; 4, soot agglomerate.

may be dependent on the initial droplet diameter, such as the rate of condensation of water vapour on a droplet) may have important consequences concerning comparisons of droplet burning data.

In comparison to the minimal soot production and flame luminosity of the first two mixtures, dramatic increases in soot production and flame luminosity were found for droplets with initial toluene concentrations of 50% by volume. Figure 3*c* and 3*d* illustrate typical results for two different droplets. Comparing the photographic

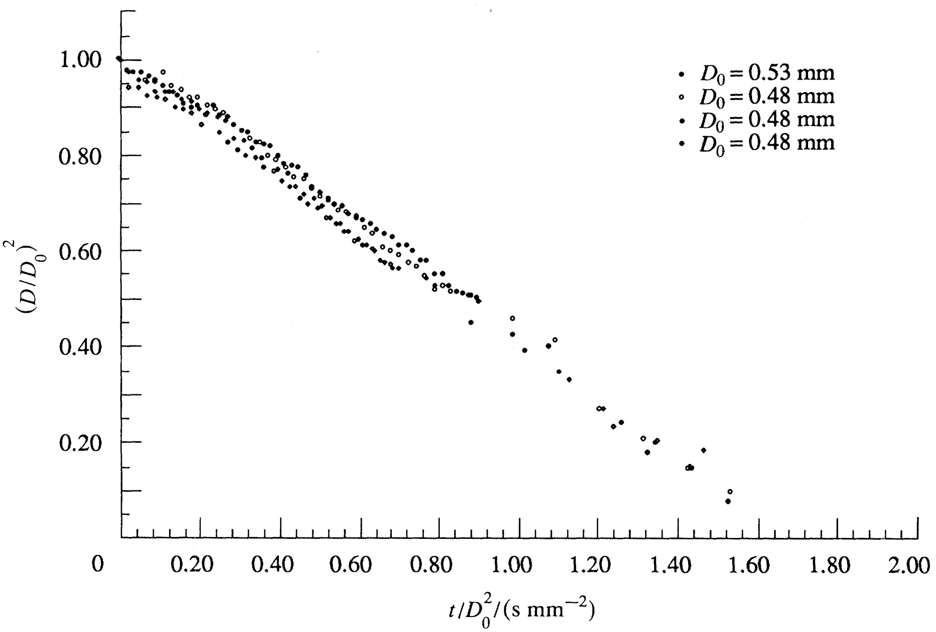

Figure 8. 50% toluene in methanol droplets.

series in figure 3*a* and *c*, which were obtained with the same backlighting, reveals the increased luminosity and soot production for the 50% mixture. Soot radiation completely obscured the 50% mixture droplet in figure 3*c*. Increasing the backlighting intensity (figure 3*d*) allowed for observation of the droplet and of the development of a spherical soot structure inside the flame. Extinction occurred for this mixture also and was characterized by the soot structure collapsing with the flame around the droplet before complete vaporization. This is illustrated in the last photographs in figure 3*c* and *d*.

An additional photographic sequence in figure 7 shows further details of the evolution of the soot structure during combustion of an initially equivolume mixture of methanol and toluene. An initial period of relatively little soot formation was characterized by a faintly luminous flame, which signifies a high percentage of methanol vaporization ($t < 0.16$ s in figure 8). With the subsequent increase in toluene vaporization, soot formation became pronounced with the shell-like structure forming around the droplet, similar to that observed for pure toluene droplets (Avedisian *et al.* 1988). The soot shell development arose from thermophoretic forces pushing the soot agglomerates in toward the droplet (Knight & Williams 1980). The agglomerates reached a radial position between the flame and the droplet where the thermophoretic forces were balanced by particle drag from the radially outward gas flows created by evaporation.

Photographs in figure 7 at $t = 0.16$, 0.18, and 0.20 s reveal separate soot agglomerates forming the structure of the soot shell. The agglomerate density in the shell initially increased after its formation, and this density increase promoted coagulation of agglomerates which may have encouraged the eventual disruption of the shell (Jackson *et al.* 1991). As time progressed, the shell began to disintegrate (generally in the opposite direction of any small drift of the droplet) with larger agglomerates escaping the flame unoxidized ($t = 0.32$ s in figure 7). A final stage in the combustion was characterized by both the flame and shell moving in toward the droplet ($t = 0.32$ and 0.36 s in figure 7). When the collapsing flame crossed the soot shell, the soot was heated, as shown by the illuminated particles in the last pictures in figure 3*c* and *d*. The soot shell then appeared to rapidly break up and flame extinction occurred before the droplets were completely vaporized. The diameters of the remaining droplets, although not clear due to their rapid motion after the disintegration of the soot shell, were definitely smaller than the extinction diameters of the 5% and 25% mixtures studied.

Initial droplet diameters for the initially equivolume methanol/toluene mixture ranged between 0.48 and 0.53 mm. Figure 8 shows the evolution of normalized droplet diameter. The data for the early period of combustion were obtained from the computer-based image analysis system, but due to soot agglomerates obscuring portions of the droplet boundaries during the latter period, a manual method as discussed in §2 was used to find droplet diameters for every fifth frame. This explains the relative sparsity of data in the latter half of the droplet evolution in figure 8.

The combustion characteristics of the methanol/toluene mixtures can be partially understood in light of the fractional vaporization of the two fuel components. At atmospheric pressure, methanol/toluene vapour–liquid equilibrium data exhibit a negative azeotrope at a methanol mole fraction of 0.89 (Burke *et al.* 1964), which corresponds to the 25% by volume toluene mixture. Because of the negativity of the azeotrope, the heating of any non-azeotropic mixture during combustion will tend to push the droplet composition further away from the azeotrope. For a mixture with an initial methanol concentration lower than the azeotropic mixture, such as the 50% by volume toluene mixture, the methanol mass fraction in the liquid phase will decrease as vaporization proceeds. As a result, the proportional amount of toluene to methanol will increase in the liquid and subsequently the gas phase. This increase in toluene composition with time may result in the burning process becoming more like that of pure toluene, characterized by luminous flames and significant soot accumulation inside the flame. Such behaviour was illustrated by the combustion of the 50% mixture droplets (figure 3*c* and *d*). On the other hand, the proportional amount of toluene in a mixture with a composition significantly richer in methanol than the azeotrope, such as the 5% toluene mixture, may tend to decrease in the liquid and gas phase as vaporization proceeds. It may then be expected that combustion of mixtures richer in methanol than the azeotrope will approach that of pure methanol. The 5% toluene mixture droplets exhibited such behaviour, characterized by non-luminous flames (figure 3*a*).

For the azeotropic mixture, thermodynamic equilibrium at the vapour–liquid interface might seem to suggest that the mixture would maintain its initial composition as combustion proceeds. However, the initially azeotropic composition can change during combustion because of gas phase diffusion away from the liquid surface: the liquid composition will tend to decrease in the proportional amount of the species which diffuses away from the liquid surface more rapidly. With methanol/toluene mixtures, binary diffusion coefficients of methanol in air are reported to be about twice as high as those of toluene in air (Vargaftik 1975). Because during combustion both species must have concentration profiles in the vapour phase which go to zero at the flame, the concentration gradients should be larger for methanol, being initially the richer species at the vapour–liquid interface, than for toluene. Diffusive transport away from the liquid surface should then be greater for methanol with the initially azeotropic mixture, the 25% toluene mixture. The more rapid removal of methanol away from the gas–liquid interface and toward the flame can then result in an increase in the proportional amount of toluene to methanol in the liquid phase and the vapour phase as well. This could result in the combustion process becoming more like that of pure toluene. However, for mixtures near the azeotrope, the liquid and vapour compositions at the interface remain close to each other, and it may be expected that the increase in the proportional amount of toluene will remain relatively small. As shown by combustion of the 25% toluene mixture droplets (figure 3*b*), the increase in toluene concentration apparently remained small enough to prevent significant soot formation.

As noted in §1, flame luminosity of pool fires and wick diffusion flames of methanol/toluene mixtures is affected by liquid composition. The same has now been observed with droplets in the present experiments. The present results show that small droplets containing highly sooting toluene can be burned without producing particulates if enough methanol is mixed with toluene (in this case being three parts of methanol with one part of toluene). This result cannot be generalized at this time, because a theoretical framework does not exist for predicting the influence of additives on particulate formation in droplet combustion. Nevertheless, the notion of reducing soot formation during droplet combustion by selective addition of a less sooting liquid fuel may have important consequences in applications such as spray-fired incineration of liquid hazardous wastes. Particulate formation presents one of the major environmental concerns with the incineration of liquid wastes. If additives can be found which reduce particulate emissions to environmentally acceptable levels during incineration, wider acceptance of incineration for disposal of hazardous waste liquids could result.

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Figure 1

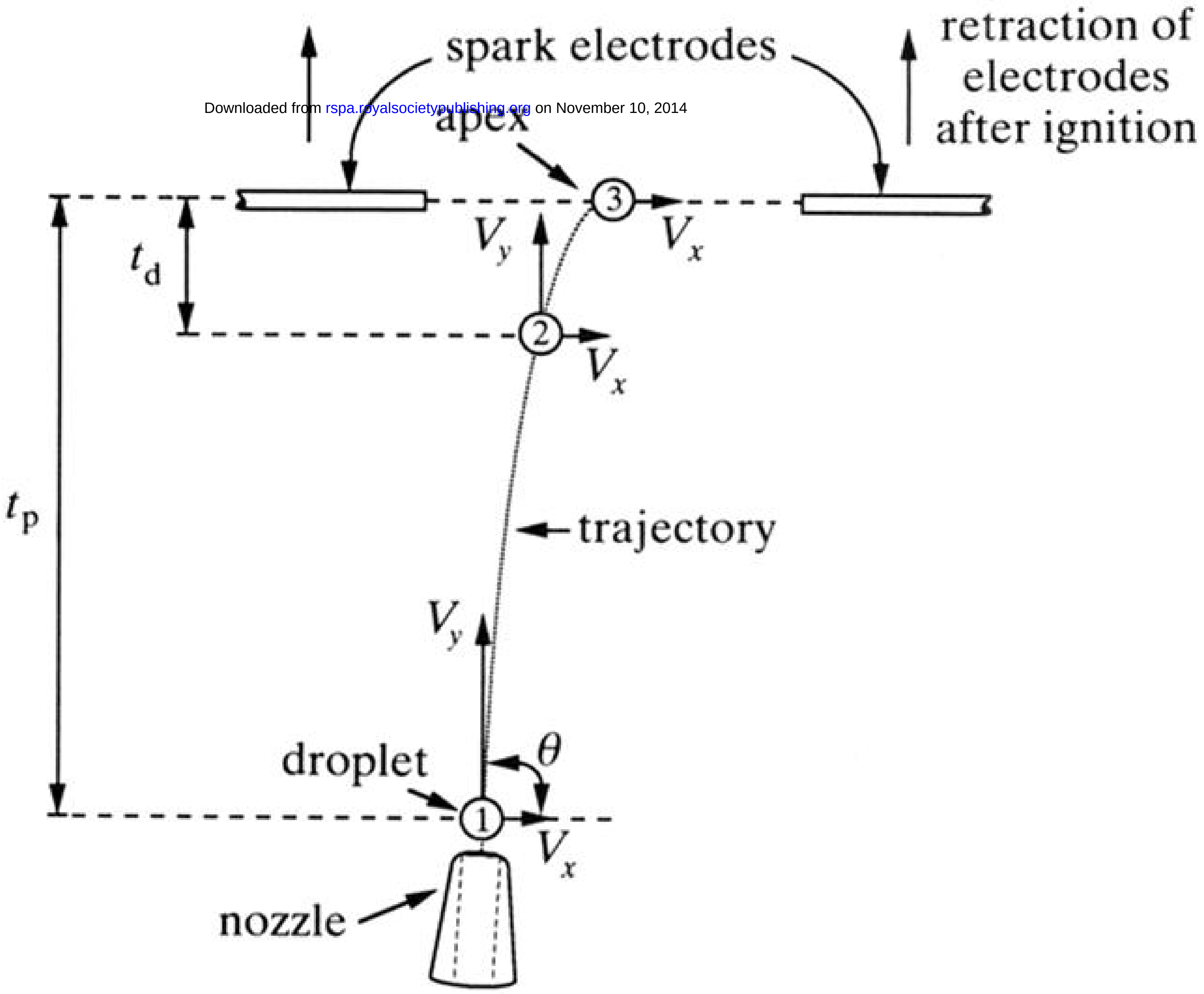

Figure 2

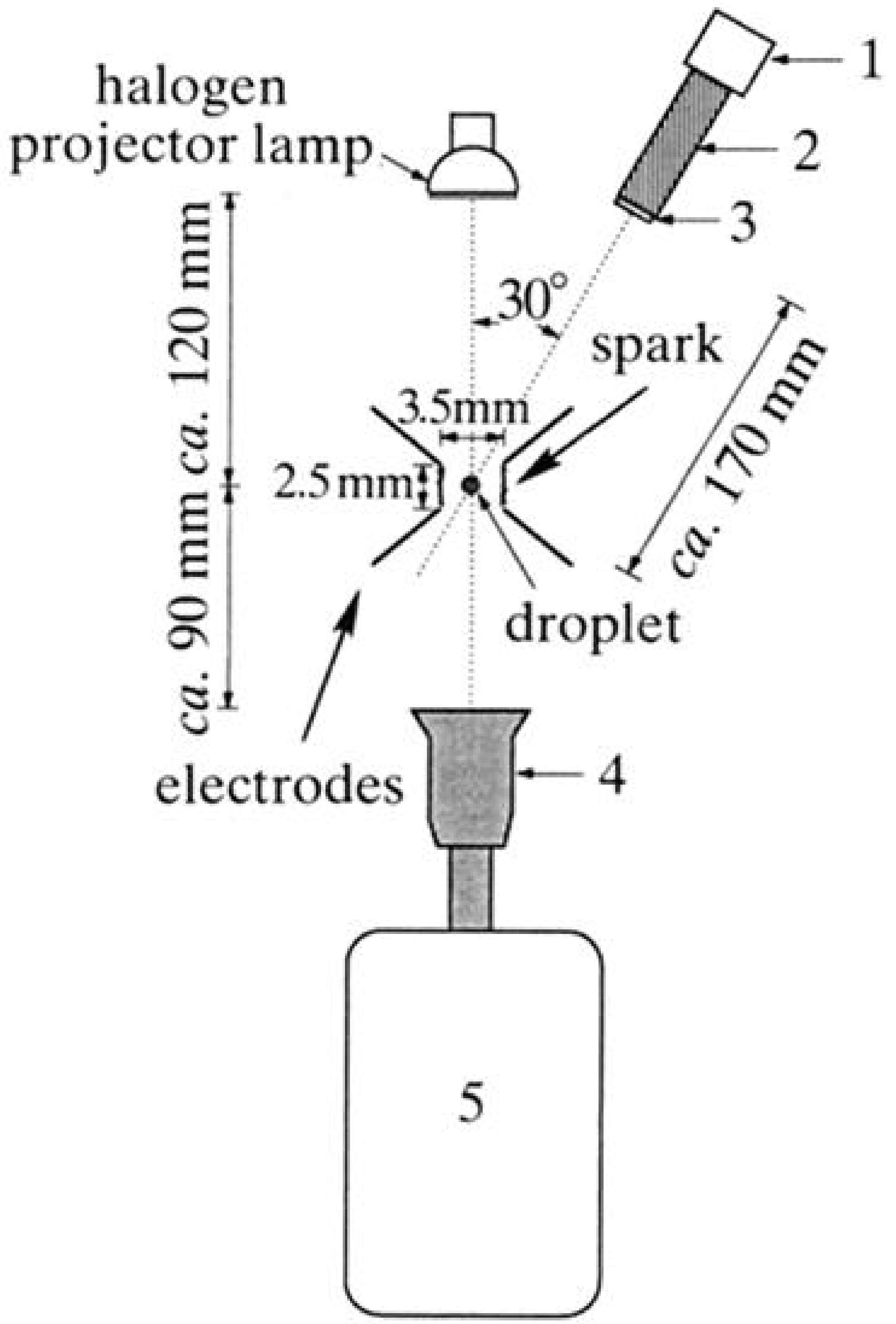

Figure 1. Schematic of the droplet trajectory which shows the apex at the plane of the electrodes where the droplet is levitated. t_p, the time for the droplet to reach its apex, and t_d, the delay time for the package to separate from the magnet, must be known to successfully levitate the droplet.

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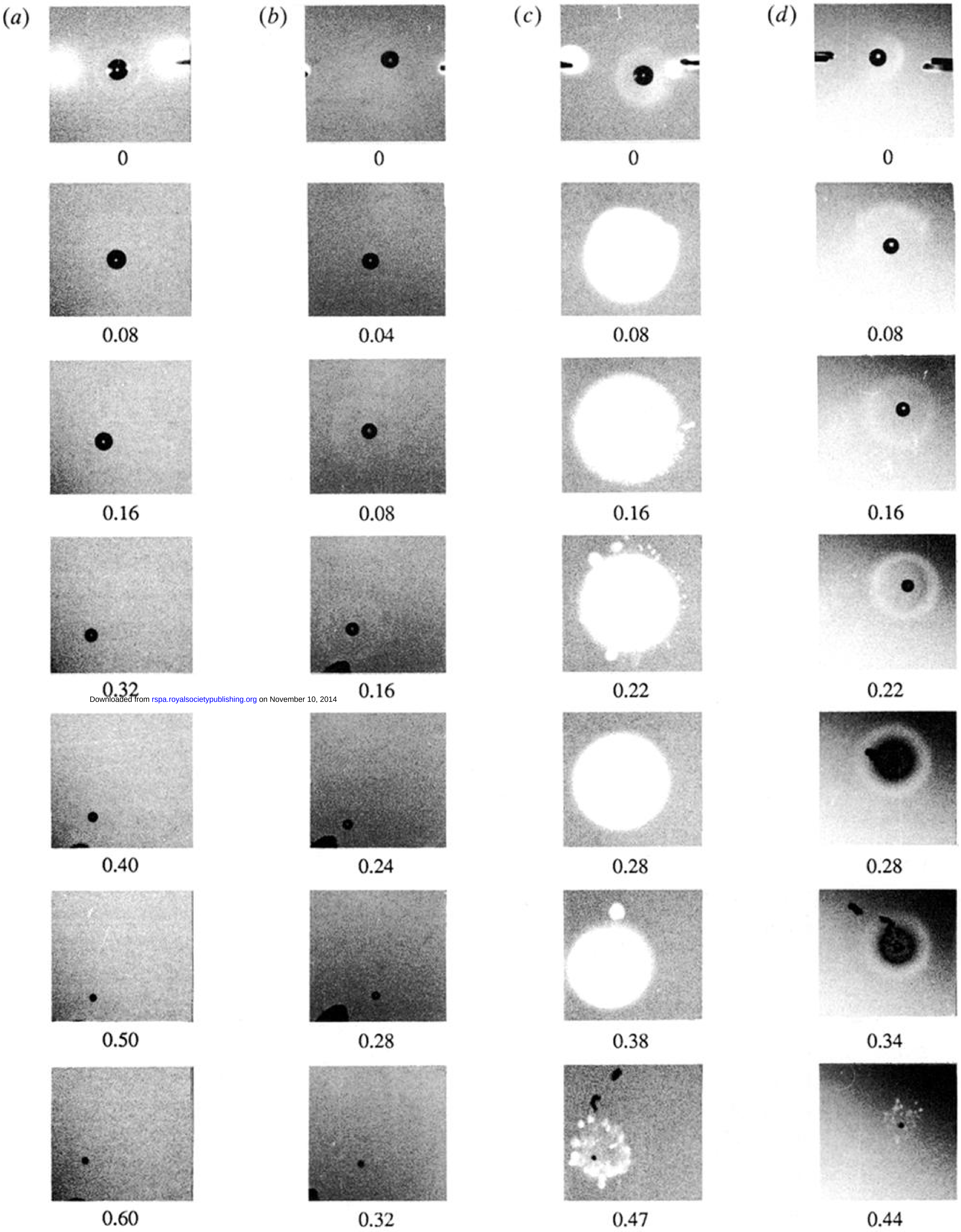

Figure 3. Methanol/toluene mixture droplets burning at low gravity. (*a*) 5% toluene by volume, $D_0 = 0.57$ mm, normal backlighting; (*b*) 25% toluene by volume, $D_0 = 0.48$ mm, faint backlighting; (*c*) 50% toluene by volume, $D_0 = 0.55$ mm, normal backlighting; (*d*) 50% toluene by volume, $D_0 = 0.52$ mm, intense backlighting.

Figure 6

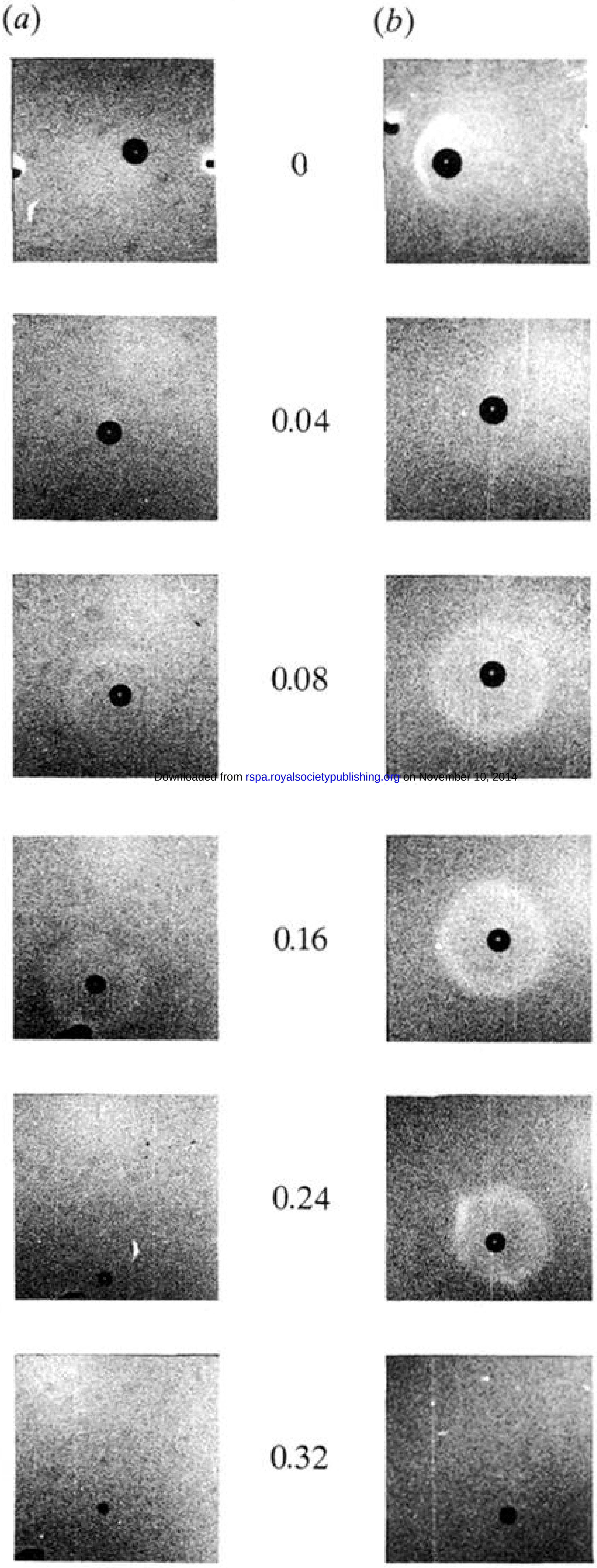

Figure 7

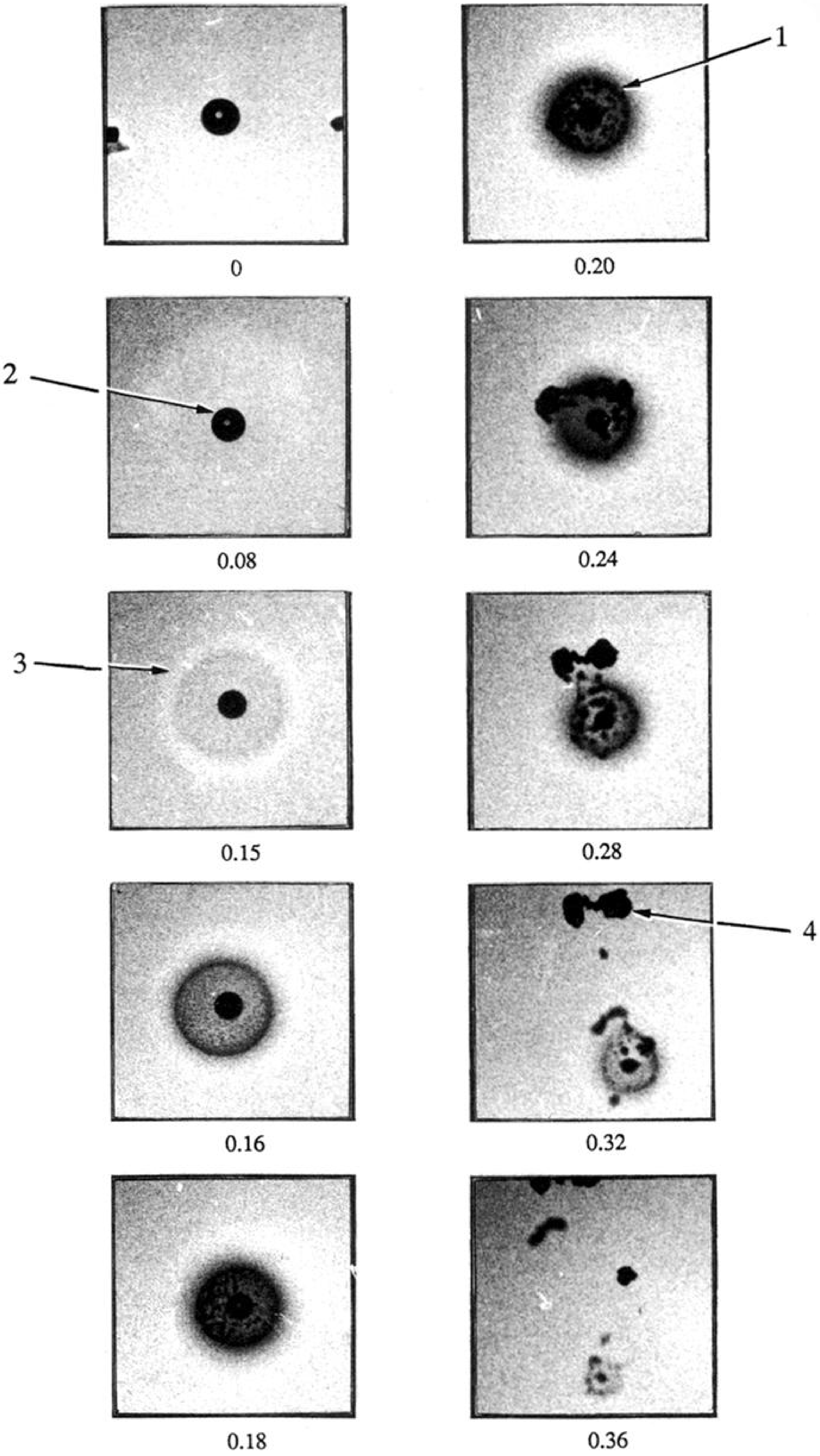

Figure 6. 25% v/v toluene/methanol droplets burning. (*a*) $D_0 = 0.48$ mm; (*b*) $D_0 = 0.55$ mm.

Figure 7. 50% v/v toluene/methanol droplet burning at low gravity. $D_0 = 0.49$, intense backlighting. 1, soot shell; 2, droplet; 3, luminous flame front; 4, soot agglomerate.