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INVESTIGATION OF FUEL ADDITIVE EFFECTS ON SOOTING  
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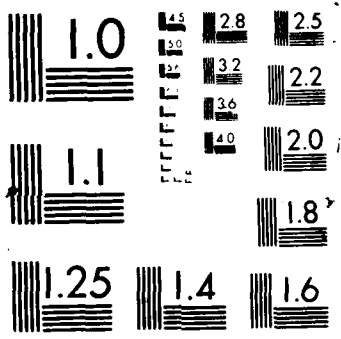
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<p>The objective of this research is to clarify the mechanisms responsible for the suppression of soot in flames by fuel additives. Measurements are limited to well-defined hydrocarbon/air prevaporized liquid- and gaseous-fueled flames. Emphasis is given to ferrocene in a diffusion flame fueled by prevaporized iso-octane. Nonperturbing laser/optical diagnostic techniques are used to relate changes in soot particulate size, number density, and volume fraction to additive concentration. Ferrocene is observed to suppress a visible soot plume completely and, in general, to intervene at a late combustion stage. Suppression is due to both size and number density reduction, which suggests that ferrocene enhances the oxidative burn-out of soot. In contrast, at an early combustion stage nearer the burner lip, a slight enhancement of soot is observed with ferrocene seeding. Also, in the seeded flame light scattering signals have been observed which are spatially</p> <p>(Continued)</p>					
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Investigation of Fuel Additive

Effects on Sooting Flames

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Principal Investigator

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Investigation of Fuel Additive  
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TABLE OF CONTENTS

	<u>Page</u>
PROBLEM/OVERVIEW . . . . .	1
PROGRESS . . . . .	1
Diffusion Flames . . . . .	1
Premixed Flames . . . . .	4
REFERENCES . . . . .	6
PUBLICATIONS . . . . .	7
PERSONNEL . . . . .	7
INTERACTIONS . . . . .	7
TABLE . . . . .	8
FIGURES . . . . .	

Investigation of Fuel Additive  
Effects on Sooting Flames

PROBLEM/OVERVIEW

Fuel additives have been used effectively to suppress soot formation in both laboratory and practical flame media (Ref. 1). Since, however, the mechanism of suppression has not been established as yet, there are no criteria for additive selection and evaluation, which unfortunately has precluded their widespread use. The goal of this research is to clarify, in detail, the nature of additive behavior in relation to soot control. In this research, emphasis is placed wholly on metal compounds as additives since selected metals have been shown, qualitatively (i.e., without clarification), and in the past, to be very effective soot suppressants. Specifically, organo-iron compounds as well as inorganic alkali and alkaline-earth salts are the principal objects of study in this program. The metals are introduced into small laboratory-scale laminar flames. Since such flames are well-defined, their study optimizes the prospect of clarifying additive behavior. Both diffusion and premixed flames are included in this research since the latter flame types may be shown to complement each other significantly. In all circumstances, measurements are carried out or planned for gaseous fuels alone. This includes the diffusion flame fueled by iso-octane and described below since the liquid fuel is heated and vaporized prior to combustion. One of the objectives of this program is to establish the one or more soot formation stages at which the additive intervenes. This is not a trivial task since its execution requires the measurement of a significant number of different gaseous and particulate flame species. To this end, both in-situ laser as well as sampling probe measurements are relied on. The laser is used principally, though not exclusively, to characterize soot particulates, whereas the sampling probe and subsequent mass spectrometric or gas chromatographic analysis is used to characterize soot precursor and intermediate gaseous species.

PROGRESS

Recent results, the status of as yet incomplete work, and near future plans are summarized below. The discussion is separated into two parts. First, diffusion flame measurements are described. This has been our area of most intense activity recently, and significant progress has been made beyond that achieved under our previous AFOSR-sponsored program (Contract No. F49620-83-C-0113). Second, the status of incomplete premixed flame measurements is described.

Diffusion Flames

Under our previous AFOSR-sponsored program cited above, the influence of alkaline-earth salt additives on soot in a  $C_2H_4$ /air Wolfhard-Parker diffusion flame was brought to a credible level of understanding (Refs. 2, 3).

Left in a less satisfactory state, however, were measurements to determine the effect of ferrocene on soot in a prevaporized liquid-fueled axisymmetric diffusion flame. Additional, significant work in this area is described here.

Light scattering measurements have been made for a sooting, axisymmetric diffusion flame with and without ferrocene present. The fuel was preheated iso-octane introduced to the flame as a vapor, while the surrounding oxidant was air. The ferrocene was dissolved in the fuel at 0.3% by weight concentration ( $1.86 \times 10^{-3}$  mole fraction). At the fuel vaporization temperature of  $125^{\circ}\text{C}$ , the ferrocene,  $\text{Fe}(\text{C}_5\text{H}_5)_2$ , is chemically stable and has a roughly 15 Torr vapor pressure. The effect of ferrocene on soot for a flame operating above its smoke point is given in Figs. 1 and 2. These results were derived from a Mie analysis of laser light scattering data taken along the z-axis of the flame. The volume fraction is calculated from  $f_v = (\pi D^3/6)N$ , where soot diameter, D, and number density, N, are determined from experiment (Ref. 4). Specifically, D is inferred from the angular dissymmetry of scattered light intensities at 20 and  $160^{\circ}$ , whereas N is evaluated from the absolute intensity at either angle and the previously inferred value of D. Fig. 1 shows clearly that ferrocene has a pronounced effect at a late combustion stage, as evidenced by the sharp decrease in volume fraction which sets in near  $z = 35$  mm. This behavior suggests that ferrocene suppresses soot by enhancing its oxidative burnout. This is confirmed by the data in Fig. 2 which shows a corresponding decrease in particulate size. The data in Fig. 2 are particularly significant since previously we were unable to establish a size decrease convincingly (Ref. 5). Recently improved detection sensitivity and spatial resolution have made this important conclusion possible. Specifically, the viewing solid angle was increased from  $10^{-5}$  to  $4.6 \times 10^{-3}$  steradians, and the optical sample length was decreased from 1.5 to 0.5 mm.

The data in Figs. 1 and 2 do not establish possible ferrocene effects at early combustion stages. Spurious signals from presumably unvaporized fuel molecules prevented probing at lower z-positions. On the other hand, data similar to that in Figs. 1 and 2 have been obtained for a ferrocene seeded iso-octane flame operating just below its smoke-point. In this case, it was possible to show that just subsequent to soot inception ferrocene tends to enhance soot formation slightly but perceptibly. An example of this behavior is shown in Fig. 3 where enhancement is most evident in the interval  $10 < z < 15$  mm. The soot reduction at  $z > 30$  mm in Fig. 3 is similar to that at  $z > 40$  mm in Fig. 1, but the former reduction is less pronounced owing to more significant soot oxidation in this flame when ferrocene is not present. In order to confirm ferrocene enhancement of soot at small "z", measurements were made at lateral positions coincident with peak soot volume fraction for  $z = 5, 7, 9$  and 11 mm. These results are summarized in Table 1, where ferrocene is seen to enhance soot consistently. In order to rule out the possibility that this enhancement was due to soot promotion by the dicyclopentadiene product of ferrocene decomposition, measurements were repeated for a dicyclopentadiene seeded flame, where the mole fraction of the latter present in the fuel ( $= 1.86 \times 10^{-3}$ ) was the same as that for the ferrocene case. For  $z = 5$  mm and a lateral position corresponding to peak volume fraction, no measurable effect of the dicyclopentadiene on sooting was observed.

One example of ferrocene effects at an early combustion stage is shown in Fig. 4. These data give measured intensities at a 20-degree scattering angle for a fixed height of  $z = 4$  mm and variable  $x$ -position. In Fig. 4a, there are two signals. The first at  $x=0$  is due to unvaporized fuel and is not of immediate interest; the second between  $x = 4$  and 5 mm is the first detectable soot signal. When the flame is seeded with ferrocene and the  $x$ -traversal is repeated, the data are decidedly different as indicated in Fig. 4b. The cause for enhancement at  $x = 0$  is unclear, but this signal is probably not relevant to additive behavior vis-a-vis soot. Note the increased soot intensity between  $x = 4$  and 5 mm in Fig. 4b in agreement with the comments and observations above. The signal near  $x = 3$  mm in Fig. 4b is present at reduced intensity even before soot inception, i.e., at  $z < 4$  mm. It has been tentatively identified as due to solid  $Fe_xO_y$ ; this was enforced by observing an intensity increase with slight  $O_2$  addition to the fuel stream. An example of this effect is shown in Fig. 5, where light scattering intensities (in the presence of ferrocene) are shown with and without  $O_2$  present. In Fig. 5a, the signal is measured at a 20-degree scattering angle and for a  $z = 2$  mm vertical position. Also, the iso-octane flow is approximately (2/10) ml/min, accompanied by a 60 cc/min flow of  $N_2$  which conducts the fuel vapor to the lip of the 11 mm i.d. burner tube. In Fig. 5b, conditions are the same except for the addition of a 13 cc/min flow of  $O_2$  to the fuel/ $N_2$  stream. The increase of signal intensity is evident. Based on both the large magnitude of the 20-degree signal (as compared, for example, to that due to molecular Rayleigh scattering) and the insensitivity (not shown) of the 20/160° scattering ratio to  $O_2$  addition, it appears indeed that the data in Fig. 5 indicate an increase in the number density of solid particles with said addition. Although the data in Fig. 5 are highly suggestive of solid  $Fe_xO_y$  formation, further confirmation of this interpretation is desirable. Accordingly, measurements are planned for which soot from ferrocene seeded flames will be collected with a probe and then examined via Auger spectroscopy for evidence of Fe or  $Fe_xO_y$  occlusion within the soot particulates.

In order to understand the behavior of ferrocene [ $=(C_5H_5)_2Fe$ ] in relation to soot even better, it would be useful, perhaps, to seed the iso-octane flame with closely analogous compounds like chromocene [ $=(C_5H_5)_2Cr$ ] or nickelocene [ $=(C_5H_5)_2Ni$ ]. Our several attempts to pursue this course have not been successful to date for the following reason. In order to study a metallocene additive in the iso-octane flame, at least three conditions must be satisfied. These are that the additive must 1) be stable against decomposition up to and slightly above the fuel boiling point temperature, 2) be soluble and chemically inactive in the liquid fuel, and 3) have a significant (several Torr or more) and known vapor pressure at the temperature in 1) above. In this connection, for example, we have been unable, despite intense searching, to find reliable vapor pressure data for chromocene in the open literature and, moreover, have observed it to be insoluble in iso-octane. Equally disappointing, although good vapor pressure data for nickelocene exist, its thermodynamic stability is considerably less than that of ferrocene (Ref. 6). In addition, although nickelocene initially will dissolve well in iso-octane, it subsequently reacts with even small

amounts of air (oxygen) to form an observable precipitate. This latter difficulty may be circumvented, possibly, by purging the fuel of air with an inert gas, but in general extension of the ferrocene studies to other metallocenes is not straightforward. In view of the above, our emphasis most probably will continue to be on ferrocene alone.

In the near term, we plan to look for ferrocene perturbation of soot precursor and/or intermediate species. There is past evidence gathered by others (Ref. 7) that ferrocene may simultaneously decrease soot and polycyclic aromatic (i.e., pcah) concentrations. We plan to attempt to observe such an effect using, as a first choice, in-situ laser spectroscopic techniques. This may be possible for the following reason. Coe and Steinfeld (Ref. 8) have shown that it is possible to excite fluorescence emission from pyrene, among other pcah as well, in an atmospheric pressure premixed ethylene-air flame using pulsed laser light near 300nm wavelength. Similar measurements were made recently in a pyrene-doped premixed flat  $\text{CH}_4/\text{O}_2$  flame at 80 Torr pressure by Peterson, et. al (Ref. 9). Since pyrene (Ref. 10) fractional concentrations in relation to total pcah are often significant in flames (Ref. 10), our position is that it appears feasible to laser-excite pyrene in the iso-octane flame and to look for the effect of ferrocene on it. Accordingly, such measurements are planned and will be attempted.

#### Premixed Flames

Although diffusion flames have certain practical advantages in that they 1) soot copiously, 2) are inherently more stable than their premixed counterpart and 3) more nearly approximate practical combustion devices, they have disadvantages as regards the ease of understanding additive mechanisms. In a diffusion flame, it is not possible to separate soot formation from burnout, and aerodynamic and chemical processes are inextricably linked. In contrast, in premixed flames soot burnout may be suppressed entirely and the aerodynamics reduces to elementary one-dimensional diffusion. For the premixed flame, there is a one-to-one relation between various stages of soot formation and the z-ordinate. Another advantage is that species concentrations may be calculated from equilibrium data for the various reactions occurring in the flame. Omitting details, this may be done for seeded flames as well, thereby making it possible to predict the equilibrium distribution of a metal among its species, i.e., mole fraction of  $\text{M}$ ,  $\text{MO}$ ,  $\text{M}^+$ , etc. For the reasons above, preparations are in progress to pursue additive measurements in premixed flames. The primary objective of this research is to determine what effect, if any, alkaline-earth salt additives have on neutral, soot precursor and/or intermediate species. This important issue was not addressed during the course of our previous alkaline-earth metal studies (Refs. 2 and 3).

An apparatus is in place which is suitable for measuring soot via Mie scattering as well as soot precursor and intermediate species via on-line mass spectrometric probe sampling, where the soot and related precursor species occur in a nearly flat one-dimensional flame operating at subatmospheric

pressure. The burner is made entirely of brass, is water cooled, and the seeded premixed gas flow emerges through 357 1 mm diam holes spaced equally within the perimeter of the 7 cm diam exit plane of the burner. Initial efforts with this apparatus have been encouraging; however, several difficulties have emerged as well. First, although a reasonably flat, sooting  $C_2H_4/O_2/N_2$  flame was obtained, at low 20-30 Torr pressure the flame tended to lift from the burner near its periphery. This could be prevented only by operating the burner enclosure at a pressure well above 100 Torr, which defeats important advantages characteristic of operation at lower pressure. Second, we have not as yet successfully adapted a conventional pneumatically operated atomizer to seeding of the flame with an aqueous spray of dissolved alkaline-earth salts. This despite the fact that an identical atomizer was used successfully by us in previous alkaline-earth additive measurements in atmospheric pressure diffusion flames (Refs. 2, 3). Low pressure flames have been historically difficult to seed and, indeed, only a few examples have been cited in the literature (Ref. 11). The principal difficulty appears to be one of maintaining a stable flame in the presence of liquid spray injection. Third and finally, there is a sooting limit beyond which probe sampling of gas phase species is obstructed by soot deposition on the probe; we estimate this to occur at or near a  $10^{-8}$  cc/CC soot volume fraction. For this volume fraction, the maximum, then, single-pass optical absorption in our flame may be shown to be about 0.6%. This should be sufficient to carry out planned absorption measurements required for soot characterization, but not without some difficulty. The preceding, potential problem areas are being assessed rigorously at present.

In view of the uncertainties cited in the paragraph above, a second experiment parallel to the one above is currently in preparation. In this case, the flame will operate at atmospheric pressure, and gas phase precursor species such as acetylenes and pcah will be skimmed with a quartz microprobe, collected in appropriate cooled and other traps, and subsequently subjected to gas chromatographic analysis (Ref. 12). This approach will have the following distinct advantages. First, at atmospheric pressure there will be sufficient soot to permit the application of both light scattering and extinction techniques to particulate characterization. Second, seeding can be accomplished successfully with a conventional atomizer. Finally, by collecting gas samples for extended time intervals via appropriate traps, sensitivity to a very broad range of pcah will be possible, including those having more than 5 or 6 aromatic rings for which concentrations relative to total pcah are small.

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## PUBLICATIONS

1. Bonczyk, P. A.: The Influence of Alkaline-Earth Additives on Soot and Hydroxyl Radicals in Diffusion Flames. *Combust. Flame* 67, 179-184 (1987).
2. Bonczyk, P. A.: Suppression of Soot in Flames by Alkaline-Earth and Other Metal Additives (submitted for publication in *Combust. Sci. Tech./in review*).
3. Bonczyk, P. A.: Laser-Induced Saturated Fluorescence of SrOH in Flames (in preparation).

## PERSONNEL

The research described in this report was carried out solely by Dr. Paul A. Bonczyk, Senior Research Scientist, Combustion Sciences.

## INTERACTIONS

The paper entitled "Fuel Additive Effects in Sooting Flames" was presented by P. A. Bonczyk at the 1986 AFOSR/ONR Contractors Meeting on Combustion, Stanford University, Stanford, Calif., June 18-20, 1986.

On 9 April 1987, Dr. P. A. Bonczyk met with Prof. J. B. A. Mitchell at UTRC, East Hartford; subsequently, a telephone conversation between these principals took place on 29 June 1987. Prof. Mitchell is affiliated with The University of Western Ontario, Canada; he is actively engaged in research similar to that of Dr. Bonczyk, which was the subject of their interaction. Prof. Mitchell currently receives support from the Physics Directorate of AFOSR in areas of research unrelated to fuel additives.

On 22-24 June 1987, Dr. P. A. Bonczyk participated in the 1987 AFOSR/ONR Contractors Meeting on Combustion, Pennsylvania State University, University Park, PA.

TABLE 1

Soot volume fraction with and without ferrocene. Measurements taken at lateral position coincident with peak volume fraction for each of z-positions given below. [(-8) =  $1 \times 10^{-8}$  e.g.]

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<u>z</u>	<u>no ferrocene</u>	<u>0.3% (wght) ferrocene</u>
5	1.2 (-8)	3.3 (-8)
7	2.1 (-7)	2.9 (-7)
9	6.2 (-7)	7.3 (-7)
11	5.9 (-7)	1.0 (-6)

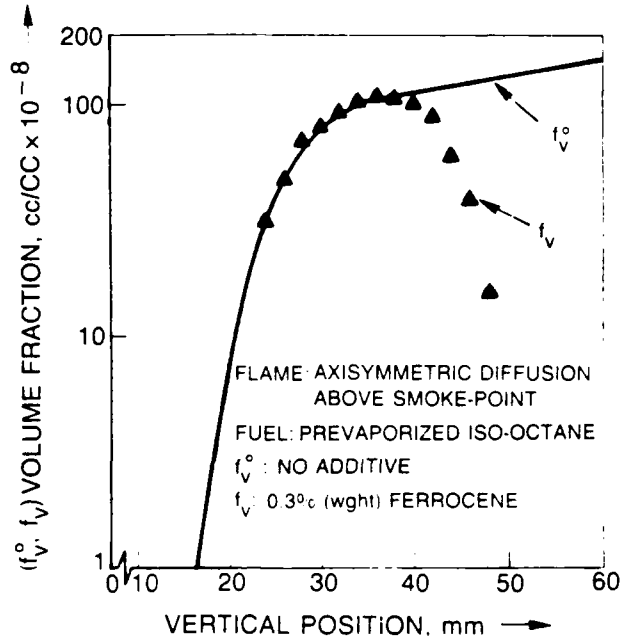


Fig. 1 Additive effectiveness on soot volume fraction.

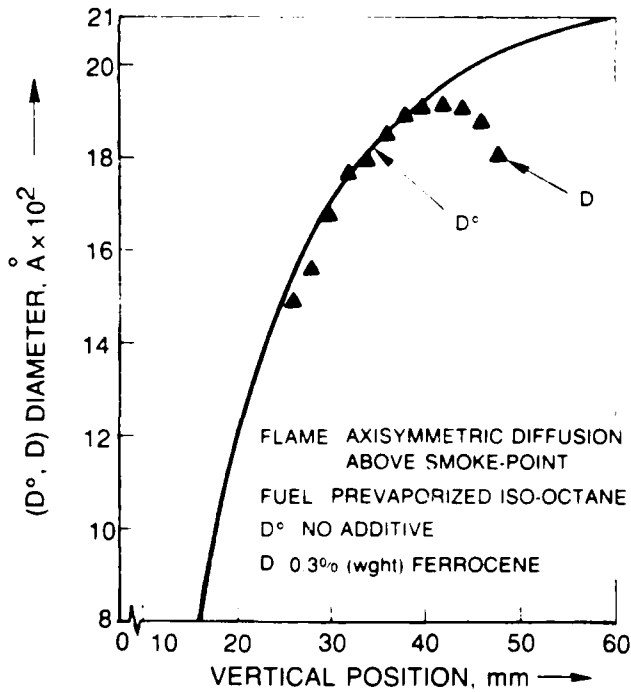


Fig. 2 Additive effectiveness on soot diameter.

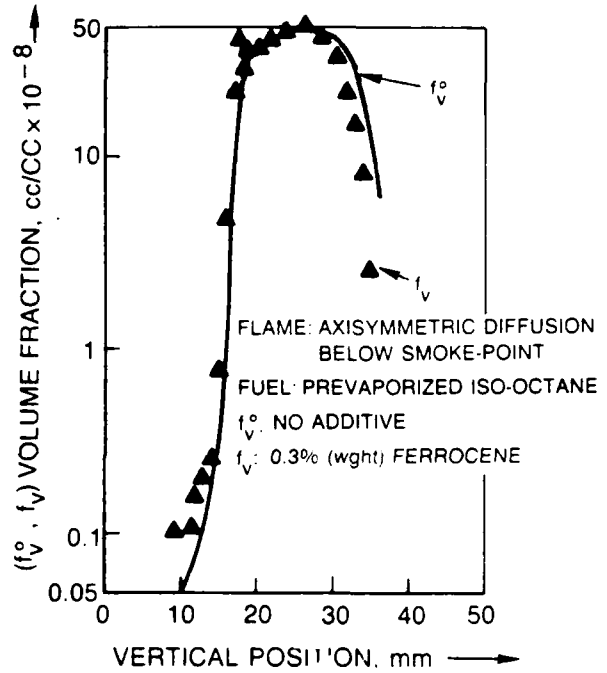
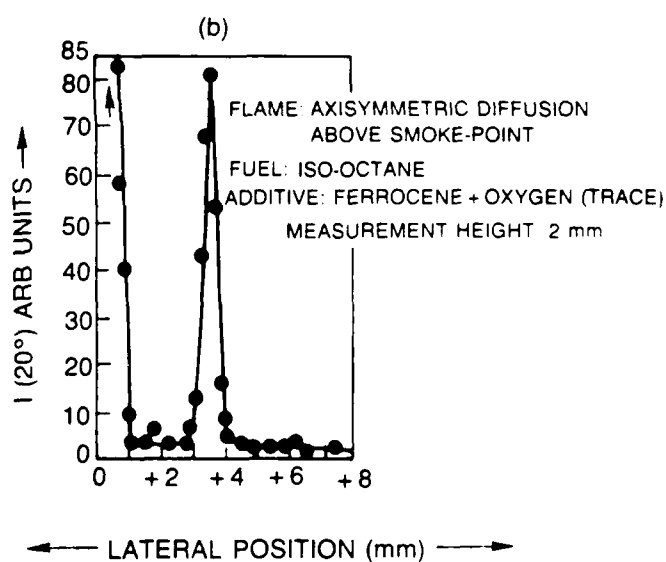
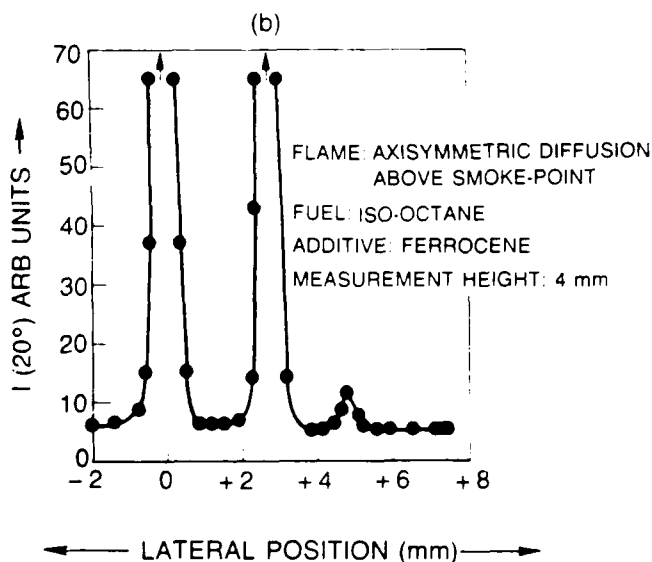
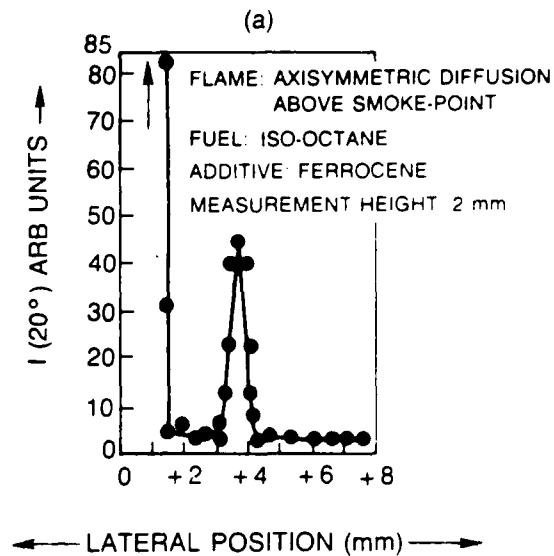
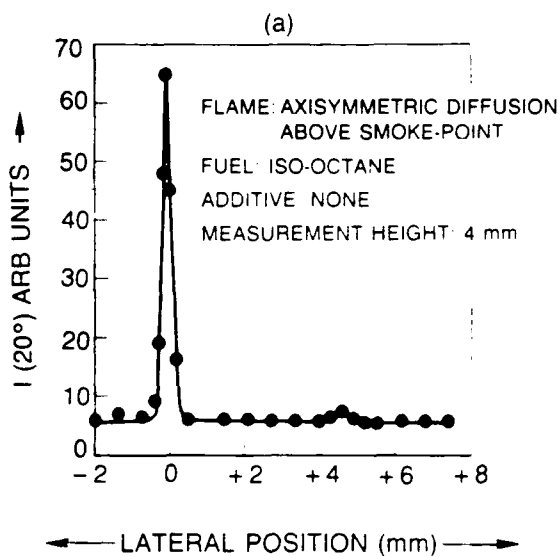


Fig. 3 Additive effectiveness on soot volume fraction.



**Fig. 4** Lateral dependence of scattered light intensity. (a) iso-octane flame. (b) iso-octane + ferrocene flame.

**Fig. 5** Lateral dependence of scattered light intensity. (a) iso-octane + ferrocene flame. (b) iso-octane + ferrocene + oxygen flame.

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