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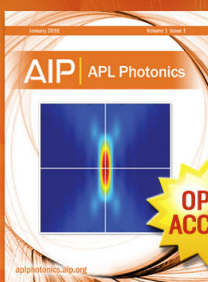
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# The use of spectroscopic measurements to evaluate cataphoretic segregation<sup>a)</sup>

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A study has been made on the influence of neutral end volumes on the segregation rates (time-dependent cataphoresis) in the positive column of a 0.01% Ar-Ne gas mixture. The Ar(4200 Å) and Ne(5882 Å) spectral-line intensities were monitored using optical techniques 5 cm from the anode and cathode. The effectiveness of the segregation as inferred from the Ne(5882 Å) intensity is inconsistent with a 0.01% Ar-Ne gas mixture. It is believed that this inconsistency is due to an axial variation of the electron-energy distribution.

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In the past, light-emission-measurement techniques have been used extensively to investigate the cataphoretic segregation of gas mixtures.<sup>1-3</sup> The widespread popularity of these techniques among investigators is primarily associated with their relative simplicity compared to other methods. However, during recent studies of time-dependent dc cataphoresis using the binary mixture of 0.01% argon in neon, certain general observations have resulted which seem worthwhile noting.

In order to study the effectiveness of the cataphoretic segregation of a gas mixture using optical techniques, spectral-line intensities are monitored as a function of distance from the cathode of the dc discharge for given values of various discharge parameters. It has been the common practice among investigators to assume that the density, temperature, and axial drift velocity of the electrons do not vary axially in the positive column.<sup>4,5</sup> The resulting (Boltzmann) distribution of atomic states would then produce a spectral-line intensity directly proportional to the neutral-atom density since the spectral intensity is proportional to the density of atoms in the upper state of the line.<sup>6</sup> Hence, the spectroscopic measurements should provide a measure of the relative neutral-atom density along the discharge tube. These linear relationships may, however, be altered by radiation trapping effects and other phenomena such as axial changes in the electron-energy-distribution function. It is the purpose of this communication to draw attention to the limitations in using the variation in the spectral-line intensity in the positive column to infer the effectiveness of the cataphoretic segregation within a gas mixture.

A schematic diagram of the apparatus used in the time-dependent cataphoresis studies is shown in Fig. 1. The discharge tube is constructed from Pyrex glass tubing having an internal diameter of 2.5 cm and a distance between the anode and cathode of 44 cm. Since the neutral volumes at each electrode are believed to influence segregation rates, an additional volume (six times the discharge tube volume) was con-

nected near each end of the discharge tube through a glass valve.<sup>7</sup> The valves were constructed from glass in order to confine the plasma to the region of the discharge tube between the electrodes. Each valve could be opened or closed by moving two glass-encapsulated metal slugs with the aid of two external magnets. In this manner measurements could be made on the effect of several different electrode end volume geometries on the times required to achieve a steady-state condition. The discharge tube and neutral end volumes were mounted on a standard high-vacuum gas-handling system. Research-grade gas samples were used in the present study.

The voltage applied to the electrodes was obtained from a regulated dc power supply with a continuously variable output up to 2.0 kV. Currents up to 100 mA could be obtained at the maximum output voltage. Spectral emission from locations 5 cm from each electrode—regions found to be within the positive column—was transmitted to a Jarrel-Ash Spectrometer via fiber-optic light pipes. After suitable amplification with a photomultiplier, the data were fed into the multichannel analyzer (MCA). A shutter at the entrance to the spectrometer was synchronized to the MCA in such a

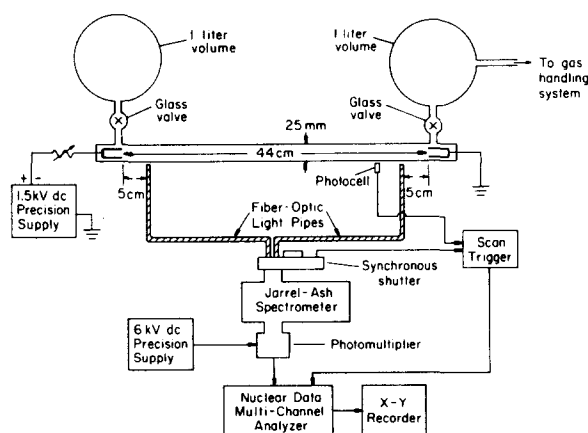


FIG. 1. A schematic diagram of the time-dependent cataphoresis apparatus.

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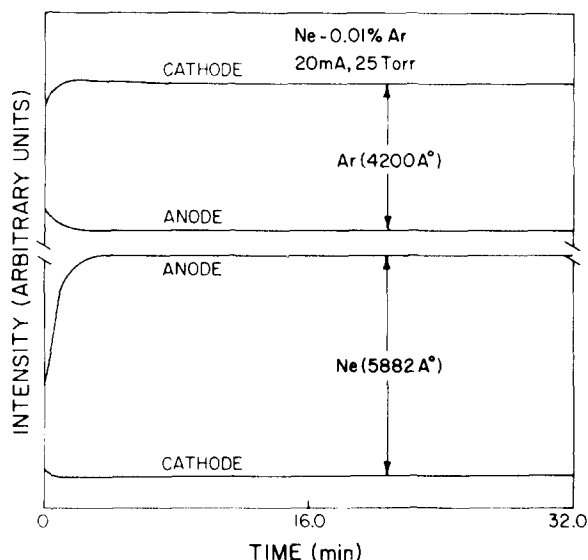


FIG. 2. Time dependence of the Ar(4200 Å) and Ne(5882 Å) spectral-line intensities. The electrode end volumes were small ( $\leq 10\%$ ) compared to the discharge-tube volume.

manner that data from the two discharge locations were alternately relayed to two separate data-storage areas within the MCA. Proper synchronization of the MCA was attained by a trigger circuit which required both the existence of a discharge and the proper shutter opening to be activated. As a result, the time dependence of any particular spectral-line intensity could be measured simultaneously at two separate locations in the discharge.

Figure 2 shows the time dependence of the intensities of the Ne(5882 Å) and the Ar(4200 Å) lines sampled near the anode and cathode. The data refers to a 20-mA discharge at 25 Torr for a 0.01% argon-neon mixture with the electrode end volumes small ( $\leq 10\%$ ) compared to the discharge-tube volume. Figure 3 shows similar data when a volume of six times the discharge-tube volume was placed behind the cathode electrode. It will be noted that the times required to achieve steady-state conditions are dependent on the relative electrode and discharge-tube volumes. This result will be explained in detail in a later report. It is important to note in Figs. 1 and 2 that for valid comparisons one should consider only the changes in the Ne(5882 Å) intensity relative to the no-discharge condition ( $t = 0$ ) as measured at either sampling position. The same comment holds for the Ar(4200 Å) line.

Examination of Figs. 2 and 3 shows that near the cathode the Ar(4200 Å) line intensity initially increases and then decays exponentially to a steady-state value. It is believed that the enhancement of this behavior in Fig. 3 is a direct result of introducing a large neutral volume relative to the discharge tube behind the cathode. Near the anode for both Figs. 2 and 3, the Ar(4200 Å) line intensity decreases exponentially as the argon is depleted in this region due to cathaphoresis. The temporal behavior of the Ne(5882 Å) line intensity is somewhat similar to the Ar(4200 Å) intensity in

that the general features can be explained by simple cathaphoresis arguments. However, if one assumes that the magnitude of the spectral lines is proportional to the neutral concentration and makes a comparison of the changes in the Ne(5882 Å) line intensity from the no-discharge condition ( $t = 0$ ) to the steady-state value ( $t \rightarrow \infty$ ) for both Figs. 2 and 3, one would conclude that the neon concentration has doubled near the anode. Such an enhancement in the neon is inconsistent with the fact that for the present gas mixture the maximum change in the Ne density must be less than 1 part in  $10^4$ . This behavior was found to hold for other neon spectral lines and also was detected regardless of the gas pressure, current, or relative magnitudes of the electrode and discharge-tube volumes.

We believe that the inconsistency of the neon enhancement for a 0.01% Ar-Ne gas mixture reflects an axial variation of the electron-energy distribution. Near the anode the electron-energy distribution is primarily determined by the neon, while near the cathode both the argon and neon will have an influence on the electron energy. Clearly, the extent to which this effect will occur is impurity concentration dependent as well as a function of other discharge parameters. The major point to be emphasized here is that changes in the major gas constituent inferred from changes in the line intensity of that component may be seriously misleading since the intensity of the spectral line not only depends on the concentration of the major gas but also on the electron-excitation conditions which vary in different parts of the positive column. Corresponding errors in estimating changes in the minor gas component from spectroscopic measurements will also occur, although these may not be as serious since for small concentrations nonlinear effects such as radiation trapping are reduced.

It seems apparent that future refinements in dc cathaphoresis theory should attempt to incorporate an axial electron-energy distribution.

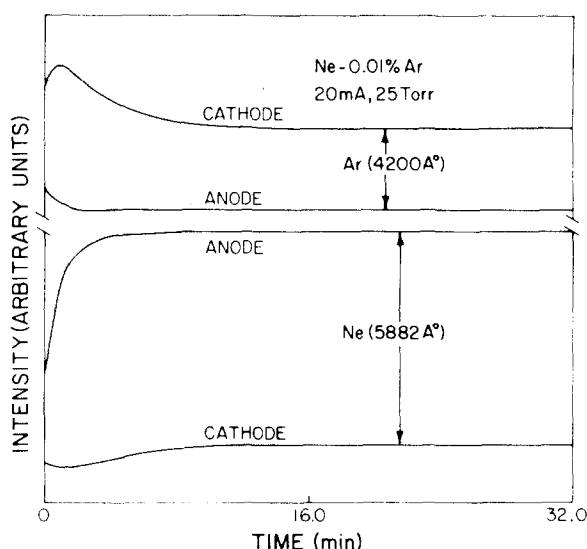


FIG. 3. Time dependence of the Ar(4200 Å) and Ne(5882 Å) spectral-line intensities. The cathode end volume was six times the discharge-tube volume, while the anode end volume remained small.

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