Fluorescence-Doped Particles for Simultaneous Temperature and Velocity Imaging

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Abstract

Polystyrene latex microspheres (PSLs) have been used for particle image velocimetry (PIV) and laser Doppler velocimetry (LDV) measurements for several decades. With advances in laser technologies, instrumentation, and data processing, the capability to collect more information about fluid flow beyond velocity is possible using new seed materials. To provide additional measurement capability, PSLs were synthesized with temperature-sensitive fluorescent dyes incorporated within the particle. These multifunctional PSLs would have the greatest impact if they could be used in large scale facilities with minimal modification to the facilities or the existing instrumentation. Consequently, several potential dyes were identified that were amenable to existing laser systems currently utilized in wind tunnels at NASA Langley Research Center as well as other wind and fluid (water) tunnels. PSLs incorporated with Rhodamine B, dichlorofluorescein (DCF, also known as fluorescein 548 or fluorescein 27) and other dyes were synthesized and characterized for morphology and spectral properties. The resulting particles were demonstrated to exhibit fluorescent emission, which would enable determination of both fluid velocity and temperature. They also would allow near-wall velocity measurements whereas laser scatter from surfaces currently prevents near-wall measurements using undoped seed materials. Preliminary results in a wind tunnel facility located at Virginia Polytechnic Institute and State University (Virginia Tech) have verified fluorescent signal detection and temperature sensitivity of fluorophore-doped PSLs.

1. Introduction

This report describes progress made during Phase I of an Aeronautics Research Mission Directorate “Seedling Fund” project supported at NASA Langley in FY11-12. The project focused on synthesis and characterization experiments using polystyrene latex microspheres (PSLs) doped with a dye or series of dyes that are mechanically bound within PSLs. This novel seed material would aid in the simultaneous characterization of multiple parameters in fluid flow environments, where only single measurements are currently performed. For example, it can potentially measure temperature, pressure, velocity and/or scalar concentration instead of just velocity.

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Several authors have described efforts to dope particles, both liquid and solid, with fluorescent dyes and luminescent paints to measure scalar flow properties. Much work has been done to assess methods for doping liquid particles with dyes and temperature sensitive paints as reviewed by Sutton et al.1 Both Dunand et al.2 and Sutton et al.1 have indicated that two-dye techniques could be attractive for enhanced temperature sensitivity. Fog-based seeding is problematic in large scale wind tunnel facilities because of the large resulting particles and large particle size distribution. Emission directivity is an important phenomenon unique to droplet and particle-based fluorescence and has been discussed by various authors. As shown by Frackowiak and Tropea, the fluence field within a single illuminated particle is complex due to Lorenz-Mie scattering effects; such directivity can be exploited to optimize fluorescence signals.3 Thermographic phosphor particles or coatings have also been investigated for their potential in simultaneous temperature and velocity imaging.4,5,6,7,8 However, such particles, typically containing rare-earth elements such as Europium, are not suitable for large flow facilities because of health risks. Dye doped PSLs have been fabricated, studied and used in a variety of applications for more than three decades.9 PSLs doped with fluorescent dyes are available commercially mainly for bio-medical applications but to our knowledge these have not been used for thermometry. These commercially available dye-doped particles are prohibitively expensive for large-scale wind tunnel tests. A review of the literature revealed no mention of PSL-based thermometry. While some researchers have used PSLs coated with pressure-sensitive paint10, only the current study is leveraging single-particle fluorescence signals from PSLs.

This paper reports the feasibility of generating dye-doped PSLs for measurement of fluorescent emission in flow experiments. Also, the use of dye-doped PSLs for thermometry was verified. Along with enabling temperature and possibly pressure characterization, this method can potentially improve signal-to-noise ratios under certain circumstances compared to signals generated by Mie scattering. Some other important goals of this research are that the resulting dye-doped PSLs should be non-hazardous and inexpensive to generate so they can be used in large-scale wind tunnel experiments. PSLs are often used in the large scale wind tunnels at NASA due to their selectable, monodisperse (or uniform) diameter distribution and high index of refraction. Uniform, known-diameter particles have a predictable response to flow fluctuations, enabling prediction of lag effects in all tests, and particles of high index of refraction produce high-contrast scattering signals (high signal-to-noise ratio), enabling high fidelity flow measurements. They can also be manufactured cost-effectively, quickly and to specification in-house.

This report primarily focuses on the development of the method for generating the particles as well as some preliminary proof-of-concept measurements. Ultimately, the efficacy of these particles will be evaluated in wind tunnels using traditional experimental techniques (i.e., laser Doppler velocimetry, LDV, and particle image velocimetry, PIV) to measure a variety of flow characteristics possibly including the following: velocity, temperature, pressure, multi-phase flows, fluid mixing, etc. These measurements also may benefit from the ability to collect data closer to the boundary wall than is currently possible with traditional seed materials.

This project involves three key research aspects:

- Identifying an appropriate measurement scheme for the intended application
- Identifying dyes that would meet the requirements of the measurement scheme and developing a method of manufacturing these dye-doped seed particles
- Demonstrating the new measurement technique(s) on relevant flow problems

This report focuses on the first two aspects of the research and especially the second aspect: identifying promising dyes and doping them into the seed materials. By way of motivation, the first section describes some possible measurement techniques using the fluorescent seed materials. The second section focuses on progress towards developing the seed materials. The third section describes some preliminary experiments that show proof-of-concept for temperature measurement using the new seed materials.

Figure 1. Three potential measurement schemes using dye-doped polystyrene latex spheres.
2. Measurement Schemes:

Figure 1 shows examples of possible measurement schemes that could use dye-doped particles for different measurements in fluid flows. Laser illumination would excite fluorescence in the dye-impregnated PSLs which are seeded into a flow in the wind tunnel. In the first instance [Fig. 1(a)], the laser-induced fluorescence (LIF) intensity decreases as the temperature of the particles increases. Mie scattering, on the other hand, is mostly independent of temperature. By obtaining both Mie scattering and LIF signals, and taking their ratio, the temperature of the particles can be measured. Figure 1(b) gives another example of a thermodynamic measurement using seeded particles. In this case, a pulsed laser is used and the LIF intensity is measured as a function of time. The lifetime depends on the gas pressure owing to increases in collisions with molecules present. In Figure 1(c) the temperature approach of Fig. 1(a) is combined with laser Doppler velocimetry to measure temperature and velocity simultaneously. In LDV, two crossed laser beams generate a fringe pattern. When a particle passes through the pattern, an oscillating time signature is collected. The frequency is proportional to the speed of the particle. Ratioing the LIF to Mie scattering can again provide temperature simultaneously with the velocity.

The approaches described in Fig. 1 are single-point measurements using single point detectors such as photomultiplier tubes (PMTs). A slightly more complicated arrangement can allow imaging (i.e. planar measurements) of the flow parameters. This temperature-velocity imaging experiment is schematically shown in Fig. 2. In this experiment, a pulsed laser is formed into a laser sheet to illuminate a flowfield. The particles seeded into the flow are doped with a dye which changes color as the particle temperature changes (as shown on the left side of the figure). Two cameras detect this fluorescence. The cameras have filters that pass different bands of the fluorescence (red and blue). The ratio of the fluorescent emission passing through the filters and detected by the cameras depends on temperature. Simultaneously, the image pairs can be used to measure the velocity of the particles using particle image velocimetry (PIV).

Many such measurement schemes are possible, for example measuring temperature, pressure, or gas concentration using either continuous wave or pulsed lasers of different wavelengths. A variety of dyes or even combinations of dyes could be used to optimize the measurement techniques. Measurements could be single point (and possibly high repetition rate) or image-based.

3. Development and Characterization of the Seed Material:

3.1 Particle synthesis

The seed material generation effort involves acquisition or synthesis of candidate dye materials with the development of procedures to functionalize certain dye systems for chemical
incorporation into PSL matrices. Research involving the direct integration of commercially available dye materials into PSLs has focused on two dyes: Rhodamine B and dichlorofluorescein (DCF, also known as Fluorescein 548 or Fluorescein 27). These were selected due to the capability of using these dyes for multi-parameter measurements (i.e., velocity and temperature). Other dyes including Kiton Red, Fluorescein, Tetraphenyl Prophyrin, Erythrosine, and Pyronin Y were also examined as possible candidates, but the two dyes above showed the most promise.

Undoped PSLs—that is, PSLs without a fluorescent modifier—were synthesized using modification of a dispersion polymerization techniques developed by our group.\textsuperscript{11} The reaction volume for the PSLs described here was approximately 1/10 that described in the technical memorandum (Ref. 11). Briefly, a reaction kettle was outfitted with a gas inlet, a thermocouple, a mechanical stirrer, and a condensation column. A mixture of 242 mL of deionized water, 33 mL of freshly distilled styrene, and 0.0444 g of MgSO$_4$ were added to the kettle. This mixture was stirred for 45 minutes at approximately 40 RPM while nitrogen was used to purge the liquids of any dissolved oxygen. Next, the stir speed was increased to 250 RPM and a kettle heating mantle was utilized to bring the temperature up to 70 °C under a positive pressure of N$_2$. Once the temperature was equilibrated, K$_2$S$_2$O$_8$ was dissolved in 8 mL of 70 °C deionized water and rapidly added to the reaction kettle. The reaction mixture was stirred overnight under N$_2$. The next day, the solution was poured through a cheesecloth filter to remove large aggregates from the solution.

The synthesis of dye-doped PSLs was conducted in a similar fashion with the exception that the dye was dissolved in the styrene monomer prior to addition to the aqueous solution in the reaction kettle. This ensured that the dye would be dissolved in the organic phase prior to generation of the dispersion solution.

### 3.2 Analysis Techniques

The size and morphology of the PSLs generated in this work were characterized. Particle size measurements were conducted on a Particle Sizing System Model 780 AccuSizer. The minimum and maximum detectable particle diameters for this instrument are 500 nm and 100 micrometers, respectively. A typical size distribution is shown in Fig. 3. The morphology of the particles was characterized using optical and confocal microscopy. Both of these techniques were utilized to measure diameter variation of the PSLs in two dimensions with the assumption that these measurements would be representative of the variation in the third dimension. To measure the diameter variation, images of PSLs were fitted to rectangular shapes such that the perimeter of the rectangle overlaid the edges of the PSL. Variation of the length and width was utilized to assess the deviation of PSL circularity. Although these data are not shown, the rectangles fitted around the PSL images did not demonstrate a significant deviation from squares, at least to the resolution of the images captured.
Four different spectral analysis techniques were available to test the resulting particles. Each method has advantages and disadvantages. The first method uses a low-power fixed-wavelength laser as the light source. In this case, the fluorescence was collected with an optical fiber and directed into an Ocean Optics USB2000 spectrometer where it was dispersed. A 532 nm blocking filter in front of the fiber blocked the laser’s wavelength. For these laser-based measurements, an aliquot of the liquid/PSL particle mixture was placed on a microscope slide and the carrier liquid was dried, leaving the dye-doped particles behind. The temperature of the microscope slide could be varied using a hot plate in order to study temperature effects. Unfortunately, this process was not completely repeatable in absolute intensity, but it produced high-quality spectra and was safe and easy to use.

The second instrument available was a spectrophotometer, which could measure the absorption spectrum of the dye-doped particles.

The third instrument was a fluorimeter. In the fluorimeter, the excitation wavelength could be varied (and an absorption spectrum identified) and the emission spectrum could be obtained. The advantage of this instrument is that the sample concentration and optical thickness could be carefully controlled because the samples are liquid. The drawbacks of this instrument are twofold: First, the particles had to be studied in a fluid, which changed their spectral characteristics compared to the eventual application in air (for LDV or PIV). Second, light from the light source could contaminate the collected emission, so the spectra had to be interpreted with care.

The final method of testing the particles was to use a high-powered continuous wave laser to probe the particles after they were dispersed into a gaseous flow. Fluorescence was captured by a lens-coupled fiber and directed to a photomultiplier tube. A filter in front of the tube blocked the laser’s wavelength and transmitted the fluorescence. This final method, set up at Virginia Tech in Blacksburg, VA, was the most time consuming as it involved a much larger setup, a setup to atomize the particles, laser safety concerns, etc. Consequently, the first three methods were used as screening and learning tools and only promising batches of particles were tested at Virginia Tech using this fourth method.

4. Results

4.1 Incorporation of dyes into PSLs

Dispersion/emulsion polymerization, which was the technique used to generate these dye-doped PSLs, is very sensitive to the chemical composition of the dispersion, among a myriad of other environmental factors. Thus, incorporation of another chemical constituent, such as a fluorescent dye, is likely to impact the resultant PSL size and size distribution. In some cases, the dye is not incorporated at all. To directly address the impact on PSL characteristics, all of the...
batches of dye-doped PSLs were synthesized with a target particle diameter of 1 micrometer using compositions well established in our research facility. Based on a review of commercially available dyes with spectroscopic properties relevant to existing wind tunnel facilities, several dyes were identified and tested. Several were chosen for low toxicity and temperature sensitivity. An additional dye, tetraphenylporphyrin, was identified due to the potential to detect pressure variations as well as modify the spectral properties readily by incorporation of different metallic ions in the center of the porphyrin macrocycle. Based on the literature, a concentration range of 0.6-0.006 mM was selected. As can be seen in Table 1, several of the dyes used did not result in significant incorporation into the particles. These particles did not exhibit fluorescence under illumination and were not further studied.

<table>
<thead>
<tr>
<th>Fluorescent Dye</th>
<th>PSL Light Scattering Characterization</th>
<th>Fluorescent Characterization</th>
<th>PSL Incorporation</th>
<th>notes</th>
</tr>
</thead>
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<tr>
<td>No Dye</td>
<td>Average Diameter, µm: 0.76</td>
<td>Standard Deviation, µm: 0.14</td>
<td>λ&lt;sub&gt;exc&lt;/sub&gt;, nm: --</td>
<td>λ&lt;sub&gt;em&lt;/sub&gt;, nm: --</td>
</tr>
<tr>
<td>Rhodamine B</td>
<td>0.63</td>
<td>0.63</td>
<td>575</td>
<td>595</td>
</tr>
<tr>
<td>DCF</td>
<td>0.77</td>
<td>0.17</td>
<td>512</td>
<td>526</td>
</tr>
<tr>
<td>Kiton Red 620</td>
<td>0.59</td>
<td>0.13</td>
<td>554</td>
<td>575</td>
</tr>
<tr>
<td>Tetraphenyl Porphyrin</td>
<td>1.08</td>
<td>0.30</td>
<td>400</td>
<td>655</td>
</tr>
<tr>
<td>Pyronin Y</td>
<td>--</td>
<td>--</td>
<td>547&lt;sup&gt;a&lt;/sup&gt;</td>
<td>570</td>
</tr>
<tr>
<td>Erythrosine</td>
<td>--</td>
<td>--</td>
<td>530&lt;sup&gt;a&lt;/sup&gt;</td>
<td>548</td>
</tr>
</tbody>
</table>

<sup>a</sup>Due to poor incorporation, the fluorescent properties could not be determined with the dye incorporated in a PSL matrix. These values are those determined in solution and would likely shift slightly upon incorporation into a PSL particle.

As detailed in the table, since most of the dyes did not incorporate into the particles or had leaching problems, the remainder of this report mainly focuses on the most promising dyes: DCF and Rhodamine B. Note that Rhodamine B is considered more toxic than DCF.

4.2 The effect of dye and salts on particle diameter

Incorporation of fluorescent dyes was determined to impact the PSL particle diameter and size distribution. For most cases shown in Table 1, the presence of the fluorescent dye reduced the average particle diameter significantly. Similarly, the standard deviation, as a percentage of the particle size, increased considerably. Both of these results suggest that the fluorescent dye impacted the radical chain polymerization processes that generate the PSLs. A common method to modify PSL diameter is to change the ionic strength of the reaction solution (for example, changing the concentration of MgSO<sub>4</sub>). Figure 5 shows how DCF-doped particle sizes changed with dye concentration (a) and MgSO<sub>4</sub> concentration, presented as ionic strength (b). Ionic strength is a measure of the concentration that also takes into account the charges of the ions and also includes the ions from the initiator (K<sub>2</sub>S<sub>2</sub>O<sub>8</sub>). Surprisingly, as seen from the graphs, neither
dye concentration nor MgSO$_4$ concentration exhibited a significant influence on the diameter of the PSLs. In past work$^{11}$ the MgSO$_4$ concentration significantly influenced particle size. This discrepancy is currently unresolved.

(a)

![Graph](image1.png)

(b)

![Graph](image2.png)

**Figure 5.** Effect of dye concentration (a) and MgSO$_4$ concentration (b) on particle size for DCF-doped particles. In (a), the electrolyte concentration was constant (using 0.015 mol/L water). In (b) the dye concentration was constant (using 0.0093 mol/L styrene).

### 4.3 Spectral characterization of the resulting particles

Figure 6 shows a comparison of PSLs doped with Rhodamine B and DCF, both excited with the same 532 nm laser. The absence of signal near 532 nm was caused by the blocking filter in front of the optical fiber. The two spectra have peaks at different wavelengths (about 550 nm for DCF and 590 nm for Rhodamine B). The fluorescence from Rhodamine B is approximately 3 times stronger than that of DCF.
As is well known, the spectroscopic properties of many organic molecules with large dipole moments are sensitive to the polarity of the environment. This property is termed solvatochromism. Rhodamine B is a solvatochromic dye and the influence of solvent polarity on Rhodamine B’s fluorescent properties has been extensively studied. Therefore, to verify that the Rhodamine B was encapsulated within the PSLs—not simply present in solution or physisorbed to the surface—the fluorescent emission characteristics of the Rhodamine B-doped PSLs were compared to those obtained from Rhodamine B in an aqueous solution (Fig. 7). The shift in Rhodamine B’s fluorescent emission to longer wavelengths confirms that it is located in an environment with a polarity lower than that of water, i.e., it is located in a styrene matrix. This observation was further confirmed by leaching studies described below.

4.4 Effect of the dye concentration on fluorescence intensity

By integrating the fluorescence in the spectral graphs obtained with laser excitation, a qualitative indication can be obtained of the effect of adding increasing amounts of dye to the PSLs. Figure 8 shows that as the DCF dye concentration was increased, the LIF intensity increased, though not linearly. The leveling off of LIF intensity may be caused by self quenching of the fluorescence or too much absorption of the laser at high dye concentrations. These data are considered qualitative because the process of drying a layer of particles on the microscope slide is not repeatable since the thickness of the layer can affect the overall intensity. An improved method of making these measurements quantitatively using the fluorimeter is being explored.
4.5 Leaching Studies

In order to verify that the dye was truly contained within the PSL, leaching studies were conducted. In these experiments, approximately 5 g of the PSL suspension were filtered through a polycarbonate filter with 400 nm pores. The filtered solution was collected and visually analyzed for fluorescent emission. The filtered particles were re-dissolved in deionized water regenerating a solution of approximately the same concentration as the original solution. This solution was passed through another 400 nm pore-sized filter with the filtered solution again visually inspected for the presence of dye.

Rhodamine-doped PSLs exhibited excellent dye retention with no detectable fluorescent emission from either the first or second filtered solutions (Fig. 9(a)). Kiton red-doped PSLs, however, demonstrated significant dye leaching, with the initial filtered solution exhibiting a deep red color and the second filtered solution exhibiting a pale pink color (Fig. 9(b)). Similarly, the filtered Kiton red-doped PSLs were nearly white upon removal of the second portion of solution. DCF-doped PSLs exhibited an interesting phenomenon. The first filtrate did not exhibit fluorescent emission, while the second filtrate did. One plausible explanation of this is that the pH of the initial solution was acidic—the pH was determined to be approximately 3.5—while the second solution was neutral. Thus, dye molecules that were physi-sorbed to the surface of the PSL exhibited much stronger interactions with the PSL in an acidic environment than in a neutral environment. This hypothesis is currently being evaluated.
5. Proof of concept detectivity and temperature measurement

NASA Langley Research Center and Virginia Tech have collaborated to test the new fluorescent particle technologies in small benchtop experiments with a view towards transitioning these methods into flow rigs at meaningful conditions and scales. Benchtop experiments, detailed below, are being used for proof-of-concept studies.

Experiments have been conducted in a subsonic, variable-temperature round jet at Virginia Tech for two purposes: first to assess the single-particle LIF signal quality of Rhodamine-B-doped PSLs described above and second, to measure the temperature sensitivity of these doped PSLs. The experimental setup is shown in Fig. 10, in which a free-space diode-pumped solid state laser (Coherent, Inc., Verdi 18 W) emitting 5 to 6 W, continuous wave, 532 nm light to excite the fluorescent particles. Mie scattering (at a wavelength of 532 nm) and fluorescence (at wavelengths greater than 600 nm) from the particles were collected using a lens system. The collected light was coupled into a multimode fiber optic which led to a pair of amplified photomultipliers, one each for the Mie and fluorescence signals.

The particles were atomized inside a plenum box and mixed with dry compressed air to supply the jet. Cooling for jet temperature variation was achieved by placing dry ice inside the plenum box, and the jet temperature was measured with a thermocouple at the nozzle exit. Jet velocities were on the order of 10 m/s. With this setup, single-particle measurements were obtained with significant signals from both the Mie scattering and fluorescence. Signals from both Rhodamine B-doped PSLs [example in Fig. 11(a)] and un-doped PSLs [Fig. 11(b)] were measured to verify that fluorescence was being detected rather than Mie scattering interference. Statistical analysis of the integrated intensity from several thousand bursts such as the example in

Figure 9. Leaching study results for Rhodamine B (a) Kiton Red (b).

Figure 10. The flow and instrumentation setup for basic scattering measurements of PSL particles. ‘V18’ indicates the Verdi 18 Watt Laser.
Fig. 11(a) was conducted to produce the temperature sensitivity data of Fig. 11(c). Therein, the mean of the ratio of the LIF to Mie signal strengths, using the Mie signal as a reference for incident excitation intensity, is plotted versus jet exit temperature. There is a strong dependence of the LIF signal on temperature, evident even for the narrow temperature range studied, which may be leveraged in future experiments to directly measure flow static temperature. While the mean measurements showed temperature sensitivity, the correlation between Mie scattering and LIF was not repeatable on a single-shot basis, therefore preventing single-shot temperature measurements from being obtained. The most likely cause of this lack of correlation is particle-to-particle variations in dye concentration. Future work will explore solutions to this problem.

![Graphs](image)

(a) (b) (c)

**Figure 11.** Results from the single-particle measurements in cold jets. (a) and (b): single-particle Mie (solid line) and LIF (dashed line) photomultiplier signals from a PSL doped with Rhodamine B and an undoped PLS, respectively. (c) Ratio of the electronic signal strengths between Mie and LIF as a function of jet exit temperature.

6. Conclusion

Development of multifunctional PSL seed materials may enable a variety of experiments currently inaccessible to flow physics researchers. The capability of simultaneously measuring velocity, temperature, pressure, and/or other environmental parameters will be of paramount importance for furthering our understanding of variable-property turbulent shear flows, encompassing a wide class of applications. Applications for the new materials include studies of:

- heated supersonic jets with the intent to reduce noise and increase performance;
- supersonic boundary layers to obtain turbulent statistics to improve compressible turbulence models;
- turbomachinery film cooling;
- boundary layer heat transfer to hot and/or cold walls

Beyond the measurement of pressure and temperature using fluorescence, the fluorescence signal may also be used to measure velocity, eliminating the interference of light scattered by facility walls. In such a paradigm, high Reynolds number wall flows, which exhibit very large gradients of velocity near the wall, may be studied for fundamental understanding of boundary layer development in two- and three-dimensional flows.

In this study, various dye-doped particles have been generated and characterized. The presence of the dyes in the particles generally caused the particles to be slightly smaller and have a larger size distribution. While several of the dyes did not incorporate into the PSLs, DCF and Rhodamine B did produce strong fluorescence signals in the various instruments tested. In the
case of Rhodamine B, the fluorescence was referenced against the Mie scattering, for single-particles passing through a focused laser volume. As expected, temperature sensitivity was observed on a time averaged basis. However the correlation between Mie scattering and LIF was not repeatable on a single-shot basis. This approach can be extended to use a crossed-beam geometry so that the Mie scattering can be processed as a laser-Doppler velocimetry (LDV) signal to simultaneously obtain temperature and velocity in turbulent flows. Future applications of this technique would incorporate the temperature sensitive particles into a particle image velocimetry (PIV) system for simultaneous planar velocity and temperature measurements. We also plan demonstrations with DCF in the same apparatus to determine if it can be used instead of Rhodamine B.

References:

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Laser Doppler Velocimetry; Polystyrene Latex Microspheres

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<td>(443) 757-5802</td>
</tr>
</tbody>
</table>

19b. TELEPHONE NUMBER (Include area code)