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NASA-CR-190739

IN-36-CR
117782
P-17

N92-34225
Unclas
G3/36 0117782

(NASA-CR-190739) DEVELOPMENT OF A VERSATILE LASER LIGHT SCATTERING INSTRUMENT Final Technical Report (Case Western Reserve Univ.) 17 p

# DEVELOPMENT OF A VERSATILE LASER LIGHT SCATTERING INSTRUMENT

by

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Submitted as a Final Report on Cooperative Agreement NCC3-74
to the NASA Lewis Research Center Technical Officer
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August 1992

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## LASER LIGHT SCATTERING CAPABILITIES

A modular approach for assembling a laser light scattering instrument from basic building blocks (Optical Lego) will allow us to implement dynamic light scattering, static light scattering, dynamic depolarized laser light scattering, etc. in a manner ideal for each of NASA's Principal Investigators. Dynamic light scattering, as used in this report, is a digital technique for measuring and correlating the intensity fluctuations in the scattered light that arise from the Brownian motion of particles in the sample. This gives the diffusion coefficient from which particle sizes in the range of 3 nanometers to above 3 microns are determined. Static light scattering - the measurement of time-averaged intensity scattered by dispersions of particles and macromolecules (e.g. polymers, proteins, micelles, microemulsions, etc.) - is an important tool for the determination of particle structure, weight-average molecular weight and particle interactions. Dynamic depolarized laser light scattering is different from regular dynamic light scattering in that it examines the weak, horizontally polarized scattered light coming from the sample. This depolarized signal contains dynamic and structural information which is not otherwise readily available.

## JUSTIFICATION

NASA Lewis Research Center is developing and coordinating the development of a versatile light scattering instrument for use in microgravity. Laser Light Scattering (LLS) will be used in microgravity to measure microscopic particles of 30 angstroms to above 3 microns. Since it is an optical technique, LLS does not affect the sample being studied.

Critical phenomena, nucleation, spinodal decomposition, gelation, aggregation, diffusion, etc. are influenced by gravity and can be better studied with LLS in a microgravity environment. LLS can enhance protein crystal growth experiments which need quantitative information about the growth process and an indication of the onset of nucleation. The above experiments and many others have already been presented to NASA in the form of "pre-proposals", which were solicited by this ATD project from experts in the field desiring an opportunity to use LLS in microgravity. A number of very well qualified investigators have identified a group of basic science experiments which require microgravity for their performance and which will be enabled by LLS instrumentation. These potential Principal Investigators (PIs) will be responding to the Laser Light Scattering section of NASA's forthcoming Announcement of Opportunity (AO) when it is released at the end of 1990.

## PROGRAM OBJECTIVES

The major objectives of this Advanced Technology Development program are to:

1. Define and ascertain instrument needs of potential PIs.

2. Modify a commonly available instrument to make it compact, rugged, power efficient, and microgravity-ready.

3. Make the LLS instrument modular to allow it to be easily reconfigured and optimized for a wide range of experiments which await the forthcoming Microgravity Fluids NASA Research Announcement (NRA or AO).

4. Automate and enhance data taking and analysis for the modular LLS instrument.

5. Collect data sets on the traditional state-of-the-art, room-size instrument in our laboratory for evaluation of the hardware and software under development and to further develop in-house expertise in preparing and analyzing different classes of samples.

6. Test miniature LLS modules attached to fiber optic probes with protein crystal growth experiments in collaboration with Professor Wm. Wilson of Mississippi State University / MSFC.

7. Simulate vibration and low-gravity performance.

8. Test back-scatter fiber optic probes.

9. Acquire and evaluate a simultaneous multiangle LLS instrument.

10. Provide assistance to the STDCE (Surface Tension Driven Convection Experiment) flight hardware project when it is in need of LLS diagnostics. While not a specific objective of the work, since we have the capability we have taken on this other program of interest of MSAD.

**APPROACH**

NASA Lewis Research Center is providing and coordinating the technology for placing a compact Laser Light Scattering instrument in a microgravity environment. This will be accomplished by defining and assessing user requirements for microgravity experiments, coordinating needed technological developments and filling technical gaps. This effort is striving to brassboard and evaluate a miniature multiangle LLS instrument. We expect to see this ATD effort become a flight hardware development project once NASA's Microgravity Fluids AO is released and the many LLS proposals we have solicited are then submitted in response to NASA Headquarters. FY90 funding supports one of the two Lewis people on the project and has forced a freeze in the developments supported by grants and in some of the hardware advances. We do believe that the reader will still be impressed with what follows.

**PROGRESS** (Elaborates Program Objectives)

1. NASA Lewis held an Advanced Technology Development (ATD) Laser Light Scattering Workshop in September of 1988. Major LLS contributors, manufacturers, and potential PIs from around the world attended the workshop. Many issues were settled in formal and informal discussions during the two day workshop. A draft version of the Workshop Proceedings was edited and issued soon after the workshop and a final, 308 page version of the Workshop Proceedings (NASA CP-10033) was published August 1989.

In January of 1990 another meeting of potential LLS PIs took place in Washington DC. At this meeting, more than a dozen researchers discussed and shared their ideas and proposal outlines.

We exhibited the back scatter fiber optic probes discussed below at the Microgravity Fluids workshop in mid 1990. This gave us a chance to discuss the LLS project with additional potential PIs and to excite the LLS community with a live demonstration of technology that is on the cutting edge.

The forthcoming Microgravity Fluids Announcement of Opportunity (December 1990) contains a section for laser light scattering which will solicit not only proposals for microgravity LLS experiments, but additional PI instrument parameters and details of how the PIs plans to store samples and initiate experiments (e.g. - chemically, thermally, through photopolymerization, etc.).

2. Dr. Robert G.W. Brown has served as a hardware consultant to this project. He invented and has patented many of the advances needed for making the miniature modules and improving the detectors used in LLS. He has provided detailed lists of the necessary miniature module specifications and ways of insuring these specifications have been met. The core of these specifications are provided for the reader in Appendix 1. At the January Washington DC meeting, Dr. Brown, associated with Royal Signals and Radar Establishment (RSRE), showed RSRE developments in miniature LLS systems in a presentation to potential PIs. Some of these systems had been designed specifically for NASA needs, and they are about a year ahead of the rest of the world. This meeting gave potential PIs a chance to look ahead at what is technologically possible and it gave NASA and RSRE important feedback on how well the needs of the potential PIs are being met.

3. Single angle detector and laser modules have been breadboarded, but have yet to be finished and delivered to the LLS laboratory at NASA Lewis for evaluation. This delay is the result of a reorganization of Defense Technology Enterprises (DTE), which was the commercial arm of RSRE. The Laser Dynamics group of DTE has since broken off and formed their own company (in alliance with a large parent company) and should be able to deliver the modules before the year is out. We intend to check the miniature instrumentation by repeating measurements which are difficult to make with traditional LLS instrumentation. Details follow in section 5.

4. The maximum likelihood correlator algorithms being developed by Professor Robert Edwards of Case Western Reserve University (CWRU) for this project are in their second year of development. The analysis portion of the prototype software for extracting particle size distributions, errors, and goodness of fit from single angle Quasi Elastic Light Scattering (QELS) data and later automating the LLS instrument is nearly completed and early results are very promising. The prototype code was written in APL, which allowed it to be rapidly modified. However, the APL code now needs to be translated to a language that can be compiled to give rapid results. The code is in the process of being documented and one of our next programming tasks at the LLS laboratory at NASA Lewis will be recoding this program using a fast compiled language. This portion of the project is on a no cost extension while we await funding for its completion. The addition of multiangle algorithms will give more information and promises to decrease the stiffness of the numerical problem and make it easier to fit histograms to the data sets. The development of the multiangle code by Professor Cheung at the University of Akron, in collaboration with Professor Edwards, is

going well, and is now consistently extracting bimodal and trimodal particle size distributions from the data libraries discussed in the next section. Professor Cheung's work is also on a no cost extension since the ATD project was only funded for one of its two Lewis scientists (the other's salary was provided by the MMSL at NASA Lewis) and hence could not offer continued support for its grant research.

The core of correlator control program for single detector input is now finished and is being used at Lewis to automatically take data, analyze it, and print out the results. The analysis algorithms are being enhanced at Lewis with additional Mie algorithms. These allow the experimenter to account for scattering asymmetries that arise with samples larger than 1/10 the wavelength of the incoming light. Two out of three Mie routines planned for the enhancements are now coded and running. The automatic LLS instrument parameter setup routines provided by the maximum likelihood algorithms remain to be added to the correlator program.

5. LLS experiments conducted in our laboratory have given us many data sets for analysis and testing of both the software and hardware being developed for this ATD effort. While this library of data is needed for evaluating the modular miniaturized hardware in its different configurations, it also perfects in-house expertise in challenging LLS experiment areas. An eight page listing of ATD LLS experiments prior to March of 1990 was previously submitted to Stuart Glazer at NASA Headquarters. We have attached several tables giving an overview of our more recent dynamic LLS work with single, bimodal, trimodal, and multimodal distributions made in-house from standards. Additional work in static light scattering on polymeric systems is provided Zimm/Berry plots and has been included in Appendix 2. These data libraries have been and will continue to be essential for evaluating both our multiangle algorithm developments and the miniature LLS modules when they arrive.

6. Our collaboration with Professor Wm. W. Wilson (MSFC) has been on hold while we await the arrival of our miniature LLS modules. We have kept in touch and have shared some recent experimental published work from the Journal of Crystal Growth with Professor Wilson discussing the detection of nucleation in protein crystals using LLS.

7. An electronic vibration isolation system with seismic control (a next generation EVIS design from Newport) has been installed and is ready to test the miniature single angle LLS modules when they are delivered.

8. Significant advances in back-scatter fiber optic probes have resulted from a grant initiated by this LLS ATD project with Professor Dhadwal at SUNY-Stony Brook. These fiber optic probes will allow LLS to be used in a solution that does not allow a laser beam to pass through it. Their larger viewing volume may prove to be of great importance when LLS is applied to protein crystal growth experiments. While waiting for the LLS modules to arrive, we have invested significant amounts of research time characterizing the revolutionary fiber probes. Tables summarizing some of the hundreds of data sets taken in this study are included in Appendix 3. With the back scatter probes, we have been able to study milky concentrations ranging up to 10% weight concentration without multiple scattering problems. These probes will be useful for both protein crystals and, in another form, for fractal studies. Multiple angle and low angle probes will be developed when funding allows.

9. Evaluation of the miniature modular multiangle LLS unit will begin when it is received. It can hopefully be purchased with 1991 funding. Note that the currently planned level of funding is sufficient only to cover salaries of the two Lewis investigators. Additional funds are required for the purchase of brassboard items. It will be used to study polydispersity, cross-correlation, and dynamic depolarized light scattering (DDLS). This brassboard of optics and simultaneous detectors will allow testing of our multiangle software with simultaneous multiangle scattering on samples that change too quickly to be evaluated with currently available equipment.

10. We have run hundreds of LLS tests on samples of particles (alumina, glass microspheres, pliolyte) proposed for use by the STDCE project. Two particle size distributions are illustrated in Appendix 4. We have presented them with written reports outlining our findings. These studies showed aggregation problems and a background of fine residual (dust) particles which destroy the background contrast for the cameras. After finding the problem, we identified a possible solution. Professor John Ugelstad in Norway supplied us with 70% porosity particles which are nearly density matched when they fill with the solution they are placed into. They are also all spherically symmetric and monodisperse (ie. - identical in size). Professor Ugelstad is willing to make and sell 90% porosity particles. This information has been passed to the STDCE project.

**KEY PERSONNEL**

William Meyer - NASA/LeRC via CWRU contract
Project Manager

* overall management of the ATD effort
* reporting to NASA Headquarters
* submitting progress reports to NASA/LeRC
* Progress Items 1-4, 9

Dr. Rafat Ansari
Research Scientist - NASA/Lewis via CWRU contract

* Progress Items 5-10

**CONCERNING THE APPENDICES WHICH FOLLOW**

The following appendices are provided to show individuals knowledgeable in the field of LLS the kind of data analysis presently available for this project. While enhancements are being added, the software available to and developed by this project is quite advanced. The data presented in the following appendices is presented prior to publication and we ask that it be cited accordingly by the reader.

**APPENDIX 1**

SUMMARY OF DR. R.G.W. BROWN'S 26 PAGE REPORT CONCERNING THE SPECIFICATION OF AVALANCHE PHOTODIODE DETECTORS FOR USE IN STATIC AND DYNAMIC LIGHT SCATTERING

| **PARAMETER** | **VALUE** | **PROOF/DOCUMENTATION** |
|---|---|---|
| Pulse height distribution | Clean, unimodal | Graph/Plot |
| Dark count Rate | <300 cts/sec PCS<br><1000 cts/sec LDV | Value @ Operating Temperature |
| Dead Time | <50nsec (<20ns if possible) | Pulse picture |
| Count rate stability | <+/- 1/2% over experimental time | Graph/plot over over 30 minutes |
| Temperature stability | <+/- 1/50 °C | Plot of constant source |
| Voltage stability (bias) | <+/-2 or 3 mV | Plot of constant source |
| Quantum efficiency | >25% SLIK (if available)<br>>7.5% C30921S | State method of proof |
| Linearity of count rate | up to 10 MHz at rate | Graph |
| Afterpulsing | <0.04% | Calculations +9 |
| Factorial moments | up to 3rd or 4th | Calculations for $n \geq 2$ |
| Power consumption | depends on cooling typically less than 6 Watts | Result of measurement |
| Operational temperature range | Constant ct rate over T | Graph/Plot |
| Anti-lock up after saturation | | |
| Output pulses | Compatible with chosen correlator | |

SUMMARY OF DR. R.G.W. BROWN'S 20 PAGE REPORT CONCERNING THE SPECIFICATION OF SEMICONDUCTOR LASER DIODES FOR USE IN STATIC AND DYNAMIC LIGHT SCATTERING

| PARAMETER | VALUE | PROOF/DOCUMENTATION |
|---|---|---|
| Mode structure | $TEM_{00}$ | Data sheet/far field photograph |
| Mode hopping | Zero after warm-up | Graph/plot over 30 minutes |
| Wavelength | 780 or 850 nm typically | Monochromator output |
| Output power | >20mW over 30 minutes | Graph of plot over 30 minutes |
| Factorial Moments | Up to 3rd or 4th +/- error | Calculations for n>2 |
| Excess correlations | <0.04% | Results of Auto-measurements at 20K and 100K cts/sec |
| Coherence length/ linewidth | >3m / <80MHz | Manufactures data sheet or Spectrum-analyzer plot |
| Laser beam diameter/ collimation | as chosen | Photo, Ronchi test or knife edge convolution |
| Polarization | 500:1 if possible +/- 1/10° alignment | Alignment accuracy fading plot |
| Power consumption | depends on cooling | Result of measurement |
| Operational temperature range | Constancy over T | Graph/Plot |
| Temperature stability | +/-0.5 °C | Graph/Plot |
| Voltage stabilities | +/-2 or 3mV | Plot of constancy |

## APPENDIX 2

## (GLOSSARY)

This glossary is provided with the assumption that the reader has some knowledge of LLS. Many volumes have been written on the terms listed below. We are providing this glossary to insure that the abbreviations in the following tables are meaningful.

1. **Conc.**: Weight/volume solids concentration in percent.

2. **Diff.Coeff.**: Diffusion coefficient. The rate at which a particle diffuses is strongly dependent upon this parameter. In simpler terms, the driving force results from a gradient in concentration which causes particles to move from regions of high concentration to regions of low concentration.

3. **2dCum**: Second cumulant of a fourth order cumulant analysis. This analysis describes the size distribution of the particles by its moments. Information on the shape of the distribution function is not provided by this method.

4. **PDP**: Polydispersity parameter of the system being studied.

5. **DblExp**: Double exponential. A double exponential often fits data quite well, but not much can be inferred unless additional evidence of a bimodal model exists.

6. **ExpSam**: Exponential sampling is also known as Pike-Ostrowski analysis. This technique does not assume a fixed number of modes.

7. **NNLS**: A non-negatively constrained least squares algorithm described by C.L. Lawsen and R.J. Hansen in "Solving Least Squares Problems", Prentice-Hall, 1974.

8. **Contin**: Contin is capable of analyzing a variety of related numerical problems for the regularized solution of linear algebraic and linear Fredholm integral equations of the first kind. It contains options for peak constraints and linear equality and inequality constraints. It was developed by S.W. Provencher in the Federal Republic of Germany.

9. **RMS ERROR**: Root-mean-square error from residual plots as a function of correlator channel.

10. **CNTRT**: Total number of photons counted in one second.

11. **GAMMA**: Half-width at half-maximum of the intensity fluctuation spectra due to Brownian motion of particles. The diffusion coefficient is extracted from Gamma.

12. **% ERROR** indicates % error in Gamma, and hence in diffusion coefficient measurement.

13. **Radius of gyration** is defined as the square root of the weight average of all the mass elements.

14. **2nd Virial Coeff.** provides a measure of particles interactions in the system being studied.

**TABLE 1**

**LASER-LIGHT SCATTERING STUDIES OF MONODISPERSE SYSTEMS**
(SPHERICALLY SYMMETRICAL IDENTICAL PARTICLES)

| Angle degrees | Diff.Coeff. $Cm^2Sec^{-1}$ | DIAMETER IN NANOMETERS 2dCum | PDP | DblExp | ExpSam | NNLS | Contin | BEST FIT | RMS ERROR | CNTRT KHz | GAMMA rad/s | % ERROR |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| (DOW 91 nm) | | | | | | | | | | | | |
| 15 | 4.83E-8 | 92.9 | 0.15 | N/A | 159 | 82 | 101 | NNLS | 2.25E-3 | 485 | 95.5 | 1.00 |
| 20 | 4.58E-8 | 90.5 | 0.24 | 88 | 117 | 90 | 75 | 2dCum | 7.23E-4 | 356 | 173.3 | 0.32 |
| 30 | 5.19E-8 | 91.0 | 0.07 | 89 | 79 | 85 | N/A | NNLS | 4.26E-4 | 288 | 384.1 | 0.29 |
| 45 | 5.60E-8 | 85.1 | 0.05 | N/A | 68 | 83 | 101 | Contin | 5.68E-4 | 164 | 896 | 0.33 |
| 60 | 5.73E-8 | 85.0 | 0.01 | N/A | 85 | 84 | 101 | ExpSam | 4.15E-4 | 218 | 1530 | 0.27 |
| 90 | 5.62E-8 | 87.2 | 0.002 | N/A | 88 | 86 | N/A | 2dCum | 6.22E-4 | 134 | 2983 | 0.42 |
| 120 | 5.45E-8 | 89.6 | 0.008 | N/A | 90 | 87 | N/A | 2dCum | 5.70E-4 | 171 | 4356 | 0.34 |
| 135 | 5.43E-8 | 88.7 | 0.03 | N/A | 88 | 90 | 101 | 2dCum | 3.69E-4 | 198 | 5009 | 0.22 |
| 150 | 5.32E-8 | 87.1 | 0.09 | N/A | 144 | 83 | N/A | NNLS | 1.92E-4 | 262 | 5571 | 0.85 |
| 160 | 5.26E-8 | 89.4 | 0.07 | 87 | 90 | 84 | 92 | ExpSam | 2.25E-4 | 344 | 5645 | 0.17 |
| (DOW 482 nm) | | | | | | | | | | | | |
| 30 | 1.06E-8 | 466.4 | 0.005 | N/A | 472 | 461 | N/A | 2dCum | 1.31E-4 | 2155 | 466.4 | 0.19 |
| 60 | 7.34E-9 | 627.7 | 0.11 | 690 | 404 | 571 | N/A | NNLS | 5.94E-4 | 88.7 | 627.8 | 0.86 |
| 90 | 9.68E-9 | 486.3 | 0.07 | 484 | 769 | 499 | 486 | 2dCum | 5.01E-4 | 57.5 | 535 | 0.31 |
| 135 | 3.10E-9 | 1087 | 0.45 | 952 | 1583 | 786 | 1938 | NNLS | 7.45E-4 | 26.9 | 408 | 1.34 |
| (DOW 1.082 micron) | | | | | | | | | | | | |
| 30 | 3.77E-9 | 1364 | 0.005 | N/A | 1122 | 1277 | N/A | 2dCum | 9.07E-4 | 649 | 25.5 | 0.84 |
| 45 | 3.69E-9 | 1244 | 0.14 | 1188 | 1132 | 1026 | 1328 | Contin | 5.03E-4 | 136 | 61.3 | 0.53 |
| 60 | 3.22E-9 | 1387 | 0.20 | 1101 | 762 | 1097 | 1620 | DblExp | 8.23E-4 | 48.6 | 93.8 | 1.17 |
| 90 | 3.20E-9 | 1194 | 0.28 | 1241 | 1269 | 872 | 1008 | NNLS | 7.88E-4 | 30 | 218 | 0.71 |

**Parameters**: Temperature: 25°C; Laser Wavelength: 514.5 nm; Particles: Polystyrene polymer.

**TABLE 2**

**LASER-LIGHT SCATTERING STUDIES OF POLYDISPERSE SYSTEMS**
(MIXTURES OF DIFFERENT SIZE PARTICLES)

| Angle degrees | Diff.Coeff. $Cm^2Sec^{-1}$ | DIAMETER IN NANOMETERS 2dCum | PDP | DblExp | ExpSam | NNLS | Contin | BEST FIT | RMS ERROR | CNTRT KHz | GAMMA rad/s | % ERROR |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| BIMODAL (CWRU 39 nm and DOW 91 nm) | | | | | | | | | | | | |
| 30 | 7.62E-8 | 56.1 | 0.20 | 55 | 50 | 49 | 71 | Contin | 2.03E-4 | 335 | 622 | 0.40 |
| 60 | 7.86E-8 | 55.6 | 0.17 | 53 | 51 | 52 | 70 | ExpSam | 3.92E-4 | 186 | 2341 | 0.39 |
| 90 | 8.07E-8 | 53.7 | 0.17 | 55 | 66 | 47 | 54 | Contin | 4.64E-4 | 157 | 4849 | 0.24 |
| 135 | 7.98E-8 | 54.4 | 0.18 | 54 | 53 | 50 | 54 | Contin | 4.39E-4 | 212 | 8161 | 0.32 |
| 160 | 7.89E-8 | 54.2 | 0.20 | 54 | 85 | 53 | 78 | DblExp | 2.68E-4 | 408 | 9314 | 0.17 |
| TRIMODAL (CWRU 39 nm, DOW 91 nm and 482 nm) | | | | | | | | | | | | |
| 20 | 1.06E-8 | 394 | 0.36 | 313 | 215 | 290 | 523 | DblExp | 3.52E-4 | 2665 | 39.9 | 1.32 |
| 30 | 1.00E-8 | 396 | 0.36 | 325 | 461 | 314 | 499 | NNLS | 1.02E-4 | 1563 | 87.9 | 1.29 |
| 30 | 1.01E-8 | 391 | 0.29 | 328 | 215 | 245 | 518 | Contin | 5.42E-4 | 1604 | 89.0 | 1.40 |
| 60 | 4.36E-8 | 82.9 | 0.27 | 78 | 78 | 69 | 171 | NNLS | 3.33E-4 | 138 | 1568 | 1.14 |
| 90 | 4.00E-8 | 88.8 | 0.28 | 84 | 77 | 70 | 167 | Contin | 3.66E-4 | 114 | 2929 | 1.08 |
| 135 | 5.72E-8 | 70.0 | 0.22 | 70 | 71 | 59 | 136 | NNLS | 6.58E-4 | 130 | 6345 | 0.55 |
| 150 | 3.16E-8 | 94.0 | 0.35 | 85 | 85 | 52 | 62 | NNLS | 4.22E-4 | 216 | 5165 | 1.49 |
| MULTIMODAL (CWRU 39 nm, DOW 91 nm, DOW 482 nm and DOW 1080 nm) | | | | | | | | | | | | |
| 30 | 0.70E-8 | 551 | 0.36 | 439 | 557 | 431 | 882 | NNLS | 2.31E-4 | 1492 | 63.2 | 1.52 |
| 60 | 1.90E-8 | 141 | 0.48 | 116 | 72 | 59 | 172 | NNLS | 3.10E-4 | 129 | 925 | 2.10 |
| 90 | 2.42E-8 | 125 | 0.28 | 96 | 91 | 46 | 261 | NNLS | 6.37E-4 | 90 | 2083 | 3.15 |
| 135 | 2.55E-8 | 106 | 0.27 | 89 | 81 | 62 | 134 | NNLS | 4.96E-4 | 102 | 4187 | 2.85 |
| 150 | 1.23E-8 | 173 | 0.40 | 111 | 148 | 61 | 410 | Contin | 3.32E-4 | 170 | 2802 | 4.59 |

**Parameters**: Temperature: 25°C; Laser Wavelength: 514.5 nm; Particles: Polystyrene polymer.

# APPENDIX 3

## FIBER OPTIC LIGHT SCATTERING RESULTS

TABLE 3: 35 NM (Lot # 1900223A)

PARAMETERS:

Particle Standard: Bangs Polystyrene 35 nm.
Wavelength=632.8 nm, T=20°C, Angle=157°
Run time: 5 minutes

| Conc. | Diff.Coeff. | DIAMETER IN NANOMETERS | | | | | | BEST | RMS | CNTRT | GAMMA | % |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| % | $Cm^2Sec^{-1}$ | 2dCum | PDP | DblExp | ExpSam | NNLS | Contin | FIT | ERROR | KHz | rad/s | ERROR |
| 0.01 | 9.99E-8 | 42.1 | 0.03 | N/A | 42 | 42 | N/A | 2dCum | 9.91E-4 | 16 | 6858 | 0.72 |
| 0.05 | 10.6E-8 | 39.2 | 0.06 | 39 | 49 | 38 | 40 | 2dCum | 2.84E-4 | 72 | 7362 | 0.17 |
| 0.10 | 10.7E-8 | 39.3 | 0.04 | N/A | 39 | 37 | 41 | 2dCum | 3.51E-4 | 53 | 7342 | 0.22 |
| 0.50 | 11.1E-8 | 37.8 | 0.04 | N/A | 37 | 35 | 40 | ExpSam | 1.40E-4 | 343 | 7633 | 0.05 |
| 1.00 | 11.4E-8 | 36.6 | 0.05 | 37 | 37 | 37 | 38 | NNLS | 6.73E-5 | 300 | 7888 | 0.03 |
| 2.50 | 11.8E-8 | 35.4 | 0.05 | 35 | 35 | 35 | 36 | NNLS | 8.18E-5 | 665 | 8146 | 0.07 |
| 5.00 | 12.2E-8 | 33.8 | 0.07 | 33 | 34 | 19 | N/A | DblExp | 6.81E-5 | 615 | 8544 | 0.26 |
| 10.0 | 12.0E-8 | 33.6 | 0.09 | 34 | 33 | 33 | 35 | ExpSam | 2.62E-4 | 177 | 8575 | 0.14 |

REMARKS: All measurements were taken using a Neutral Density Filter of OD.40

## FIBER OPTIC LIGHT SCATTERING RESULTS

TABLE 4: 85 NM (Lot # 1900214A)

PARAMETERS:

Particle Standard: Bangs Polystyrene 85 nm.
Wavelength=632.8 nm, T=20°C, Angle=157°
Run time: 5 minutes

| Conc. | Diff.Coeff. | DIAMETER IN NANOMETERS | | | | | | BEST | RMS | CNTRT | GAMMA | % |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| % | $Cm^2Sec^{-1}$ | 2dCum | PDP | DblExp | ExpSam | NNLS | Contin | FIT | ERROR | KHz | rad/s | ERROR |
| 0.01 | 4.77E-8 | 87.3 | 0.052 | N/A | 81 | 86 | 101 | 2dCum | 3.73E-4 | 38.01 | 3304.5 | 0.23 |
| 0.05 | 4.87E-8 | 86.4 | 0.035 | N/A | 86 | 82 | N/A | 2dCum | 6.66E-4 | 17.66 | 3338.9 | 0.44 |
| 0.05 | 4.95E-8 | 86.2 | 0.008 | N/A | 87 | 84 | N/A | 2dCum | 3.58E-4 | 132.3 | 3343.8 | 0.25 |
| 0.10 | 5.12E-8 | 82.3 | 0.031 | N/A | 82 | 81 | N/A | 2dCum | 2.84E-4 | 155.4 | 3502.9 | 0.22 |
| 0.10 | 5.10E-8 | 84.4 | 0.005 | N/A | N/A | 81 | N/A | 2dCum | 1.49E-4 | 253.0 | 3415.9 | 0.10 |
| 0.20 | 5.16E-8 | 83.5 | 0.005 | N/A | 84 | 80 | N/A | ExpSam | 2.87E-4 | 436.3 | 3451.8 | 0.25 |
| 0.25 | 5.30E-8 | 80.0 | 0.020 | N/A | 80 | 78 | N/A | ExpSam | 4.04E-4 | 624.2 | 3604.0 | 0.38 |
| 0.30 | 5.20E-8 | 81.3 | 0.025 | N/A | 81 | 81 | N/A | ExpSam | 2.24E-4 | 537.2 | 3547.0 | 0.09 |
| 0.40 | 5.31E-8 | 79.9 | 0.018 | N/A | 80 | 78 | N/A | ExpSam | 5.76E-4 | 694.8 | 3609.4 | 0.23 |
| 0.50 | 5.39E-8 | 77.1 | 0.054 | N/A | 71 | 74 | 76 | 2dCum | 2.64E-4 | 974.8 | 3741.3 | 0.26 |
| 0.60 | 5.43E-8 | 76.9 | 0.048 | N/A | 71 | 75 | N/A | 2dCum | 1.96E-4 | 792.4 | 3751.9 | 0.18 |
| 0.70 | 5.38E-8 | 77.0 | 0.059 | N/A | 77 | 75 | N/A | 2dCum | 1.80E-4 | 912.0 | 3745.6 | 0.18 |
| 0.80 | 5.51E-8 | 75.0 | 0.062 | N/A | 106 | 72 | N/A | 2dCum | 2.71E-4 | 949.2 | 3845.0 | 0.27 |
| 0.90 | 5.48E-8 | 75.4 | 0.061 | 76 | 95 | 74 | 76 | Contin | 4.45E-4 | 990.7 | 3822.5 | 0.18 |
| 1.00 | 5.71E-8 | 73.2 | 0.045 | N/A | 71 | 73 | 76 | ExpSam | 5.39E-4 | 1360 | 3939.9 | 0.34 |
| 5.00 | 6.25E-8 | 64.6 | 0.099 | 61 | 41 | 51 | 73 | NNLS | 9.72E-5 | 983.2 | 4464.0 | 0.51 |

REMARKS: All measurements were taken using a Neutral Density Filter of OD.40

**FIBER OPTIC LIGHT SCATTERING RESULTS**

TABLE 5: 165 NM (Lot # 1890620C)

PARAMETERS:

Particle Standard: Bangs Polystyrene 165 nm.
Wavelength=632.8 nm, T=20°C, Angle=157°
Run time: 5 minutes

| Conc. % | Diff.Coeff. $Cm^2Sec^{-1}$ | DIAMETER IN NANOMETERS 2dCum | PDP | DblExp | ExpSam | NNLS | Contin | BEST FIT | RMS ERROR | CNTRT KHz | GAMMA rad/s | % ERROR |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 0.005 | 2.70E-8 | 161.3 | 0.005 | N/A | 163 | 151 | N/A | 2dCum | 3.95E-4 | 128 | 1788 | 0.12 |
| 0.01 | 2.67E-8 | 161.3 | 0.005 | N/A | 163 | 153 | N/A | 2dCum | 3.45E-4 | 200 | 1788 | 0.13 |
| 0.05 | 2.77E-8 | 157.7 | 0.005 | N/A | 134 | 144 | N/A | 2dCum | 8.22E-4 | 578 | 1828 | 0.22 |
| 0.10 | 2.75E-8 | 151.8 | 0.048 | N/A | 140 | 145 | 179 | 2dCum | 5.20E-4 | 811 | 1900 | 0.26 |
| 0.20 | 2.78E-8 | 147.9 | 0.070 | 144 | 148 | N/A | 106 | ExpSam | 1.81E-4 | 1098 | 1950 | 0.11 |
| 0.30 | 2.84E-8 | 147.3 | 0.043 | N/A | 143 | 145 | N/A | ExpSam | 5.86E-4 | 984 | 1958 | 0.31 |
| 0.40 | 2.82E-8 | 145.4 | 0.075 | 144 | 140 | 138 | 130 | ExpSam | 2.02E-4 | 984 | 1984 | 0.13 |
| 0.50 | 2.84E-8 | 143.8 | 0.083 | 144 | 199 | 142 | N/A | 2dCum | 4.97E-4 | 883 | 2006 | 0.28 |
| 0.60 | 2.85E-8 | 143.0 | 0.083 | 143 | 141 | 95 | N/A | NNLS | 3.05E-4 | 749 | 2017 | 0.30 |
| 0.70 | 2.88E-8 | 140.7 | 0.095 | 153 | 129 | 106 | N/A | ExpSam | 7.65E-4 | 666 | 2049 | 0.58 |
| 0.80 | 2.98E-8 | 133.6 | 0.11 | 133 | 115 | 89 | 150 | NNLS | 2.01E-4 | 651 | 2158 | 0.49 |
| 0.90 | 3.15E-8 | 121.7 | 0.16 | 131 | 113 | 49 | 136 | Contin | 4.48E-4 | 516 | 2370 | 0.82 |
| 1.00 | 3.21E-8 | 117.3 | 0.18 | 102 | 85 | 98 | 158 | ExpSam | 1.05E-3 | 536 | 2458 | 1.00 |
| 2.50 | 10.6E-8 | 32.80 | 0.15 | 36 | 35 | 21 | 32 | NNLS | 1.63E-4 | 300 | 8779 | 2.54 |
| 2.50 | 14.1E-8 | 22.30 | 0.30 | 19 | 17 | 12 | 44 | NNLS | 1.98E-4 | 339 | 12958 | 2.10 |

REMARKS: All measurements were taken using a Neutral Density Filter of OD.40

**FIBER OPTIC LIGHT SCATTERING RESULTS**

TABLE 6: 261 NM (Lot # 1B73 SERAGEN)

PARAMETERS:

Particle Standard: Seragen Polystyrene 261 nm.
Wavelength=632.8 nm, T=20°C, Angle=157°
Run time: 5 minutes

| Conc. | Diff.Coeff. | DIAMETER IN NANOMETERS | | | | | | BEST | RMS | CNTRT | GAMMA | % |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| % | $Cm^2Sec^{-1}$ | 2dCum | PDP | DblExp | ExpSam | NNLS | Contin | FIT | ERROR | KHz | rad/s | ERROR |
| 0.005 | 1.63E-8 | 260.1 | 0.017 | N/A | 260 | 258 | 267* | 2dCum | 6.76E-4 | N/A | 1108 | 0.23 |
| 0.01 | 1.65E-8 | 260.8 | 0.005 | N/A | 264 | 257 | 259* | 2dCum | 2.54E-4 | N/A | 1105 | 0.19 |
| 0.05 | 1.68E-8 | 255.2 | 0.005 | N/A | 258 | 251 | 256* | 2dCum | 1.68E-4 | N/A | 1130 | 0.13 |
| 0.10 | 1.67E-8 | 257.4 | 0.005 | N/A | 260 | 250 | 250* | 2dCum | 3.35E-4 | N/A | 1120 | 0.28 |
| 0.20 | 1.78E-8 | 233.0 | 0.06 | 224 | 234 | 217 | 245 | Contin | 3.63E-4 | 195 | 1238 | 0.23 |
| 0.30 | 1.71E-8 | 230.0 | 0.13 | 239 | 236 | 201 | 240 | NNLS | 2.48E-4 | 229 | 1255 | 0.30 |
| 0.40 | 1.72E-8 | 223.0 | 0.16 | 225 | 200 | 197 | 206 | NNLS | 3.04E-4 | 295 | 1293 | 0.39 |
| 0.50 | 1.83E-8 | 203.4 | 0.182 | 210 | 188 | 156 | 341 | NNLS | 4.94E-4 | 228 | 1418 | 0.59 |
| 1.00 | 1.03E-8 | 226.0 | 0.179 | 205 | 195 | 186 | 303 | NNLS | 3.60E-3 | 309 | 1457 | 0.78 |

REMARKS: * represents Contin calculations without Mie corrections in the limit 200,400 nm.