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(54) Title: OPTICAL BALL LENS LIGHT SCATTERING APPARATUS AND METHOD FOR USE THEREOF

(57) Abstract: The inventive optical spherical lens light scattering apparatus and method for use thereof comprises an incident light source, a spherical or ball optical lens system and a photo detector. The spherical or ball lens optical system comprises at least one ball or spherical lens being in contact with a specimen of fluid, dispersed particles; said ball lens having, as a result of its spherical geometry, and as compared to a plano-convex or convex lens, a relatively short focal point. When the incident light source is used to generate a beam of light, and the beam of light is then focused through the spherical or ball lens, it lands at a point that is much closer to the lens, as compared to the point it normally lands on, when it is focused through a plano-convex, or a convex lens. It is the proximity of the spherical or ball lens' focal point to the spherical or ball lens itself, and the contacting of the ball lens to the specimen of fluid-dispersed particles, that minimizes, if not eliminates all light scattering masking of particles' light scattering results. The method of use of the said apparatus comprises the steps of: (i) using the incident light source to generate a beam of energy having a certain wavelength and frequency; (ii) focusing said beam of energy through a ball lens; (iii) placing a specimen of fluid-dispersed particles, in contact with the ball lense and within the focal point of the ball lens so that the focused beam of energy hits the suspended particles at exactly the focal point of the ball lens and the suspended particles scatter the beam of energy; and (iv) directing that scattered light produced by the suspended particles through a ball lens onto a photodetector.



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**NON-PROVISIONAL APPLICATION
FOR
UNITED STATES LETTERS PATENT**

**OPTICAL BALL LENS
LIGHT SCATTERING APPARATUS
AND METHOD FOR USE THEREOF**

SPECIFICATION

To All Whom It May Concern:

Be It Known, that we, Jianping (Lily) Zu, an individual and a resident alien of the United States of America, having an established place of residence at 4 Bayberry Road, East Setauket, New York 11733; and Dr. Walther Tscharnuter, an individual and a resident alien of the United States of America, having an established place of residence at 8 Harbor Court, Mount Sinai NY 11766, have invented certain new and useful improvements as embodied in an OPTICAL BALL LENS LIGHT SCATTERING APPARATUS AND METHOD FOR USE THEREOF, of which the following is the specification:

CLAIM OF PRIORITY

The present international, patent cooperation treaty application hereby claims priority of the earlier filed U.S. Provisional Application No. 60/746,642 by virtue of the fact that the applicants are the inventors of the subject matter for which protection was sought by way of said earlier filed provisional application.

BACKGROUND OF THE INVENTION

1. Field of invention.

The present invention relates to a method and apparatus for use in light scattering instrumentation. More importantly it relates to a new and improved optical lens system for use in conjunction with light-scattering detectors utilized for, among other things, the determination of molecular and colloidal properties and structure functions for complex fluids, protein characterization, particle sizing, particle size distribution analysis, zeta potential distribution and zeta potential in low mobility suspensions. More particularly, the present invention relates to an optical lens system that can be utilized to integrally form many light scattering systems' components, as for example a sample cell for use with light scattering detectors, and a light scattering detector probe.

2. Description of the Prior Art

Light scattering techniques are analytical science techniques that use certain properties of light to determine, among other things, the size of particles, particle size distributions, and molecular weights. The term “particles” as being used herein, denotes

1 objects whose size can range from macromolecule size at one extreme end of the
2 spectrum, to large mammalian cell or even phytoplankton size at the other end.
3 Macromolecule size in turn, can vary from one nanometer (one billionth (10^{-9}) of a
4 meter) in length to tens of micrometers; a size that can be determined by using the
5 properties of light.

6 When particles are suspended, i.e., when they are in suspension, in a fluid or
7 even in air, they actually move in a random pattern. This movement of small particles
8 suspended in a medium or a resting fluid is termed "Brownian Motion." Observation
9 and comparison of the "Brownian" motion of larger particles to the Brownian motion
10 of smaller particles reveals that the smaller particles move much faster than the larger
11 particles. As a result, small particles diffuse faster and large particles diffuse slower.

12 Properties of light that can be used to determine particle size are its wavelength
13 and its frequency. Frequency is a measure of how many waves pass through a given
14 point during one second. The more waves cross the point, or the closer the distance
15 between the waves, the higher the frequency. When a beam of light, such as a laser
16 beam, having a known wavelength and frequency, is focused on moving particles, i.e.,
17 when a beam of radiant energy is incident upon a particle, a portion of this energy will
18 be scattered in all directions. However, the light scattered will be at a different
19 frequency. The frequency or intensity of this scattered light or radiant energy will
20 depend on the wavelength of the incident radiant energy, upon the difference in the
21 refractive index of the particles with respect to the medium in which they are

1 suspended, upon the size and shape of the particles and upon the angle at which the
2 scattered energy is observed.

3 In other words, one of the things that the shift in the light frequency of scattered
4 light is related to is the size of the particles causing the shift. As was set forth herein
5 above, the smaller particles move faster. They have a higher average velocity, which
6 in turn causes a greater shift in the light frequency than the shift caused by the larger
7 particles. It is this difference in the frequency of scattered light among the particles of
8 different sizes that is used to determine the size of the particles present.

9 Light Scattering techniques can be separated into static light scattering and
10 dynamic light scattering. Static light scattering measures the intensity of the scattered
11 light, $I(q)$, as a function of the scattering angle. Static light scattering has been used to
12 measure the equilibrium structure factor, the particle form factor, the molecular weight
13 and the thermodynamic quantities such as the radius of gyration and the second virial
14 coefficients in polymer solutions.

15 Dynamic light scattering, in turn, measures the decay of the intensity
16 fluctuations via the intensity auto-correlation function $G(q,t)$. It can provide a
17 convenient way to measure diffusion coefficients of macromolecules in dilute solutions
18 and the dynamics of polymers in solutions of varying concentrations.

1 Light scattering techniques have found great application in many types of
2 industrial and health oriented activities. For example **Wyatt, United States Letter**
3 **Patent No. 4,693,602** and **Wyatt, United States Letter Patent No. 4,548,500** disclose
4 the use and improvement of light scattering techniques for measurement and counting
5 of aerosol, and to a lesser extent, hydrosol properties for the purpose of maintaining the
6 cleanliness of so-called manufacturing clean rooms. Such clean rooms can include
7 areas where very large scale integrated circuits are manufactured, asbestos removal
8 work areas, operating rooms, laboratories, and pharmaceutical production areas.

9 Likewise, **Wyatt, United States Letters Patent No. 4,541,719** discloses the use
10 and improvement of light scattering techniques for the characterization of a particle or
11 an ensemble of particles as a means for guaranteeing the quality of beverages, the
12 consistency of chemical reactions, the integrity of the food industry, the efficacy of
13 antibiotic chemotherapy, the determination of adulterants and/or toxicants in food and
14 water supplies. As for **Wyatt, United States Letters Patent No. 4,490,042**, it discloses
15 the use of light scattering techniques in connection with the evaluation of the quality
16 of wine.

17 The prior art apparatus used in light scattering studies generally comprises: an
18 incident source of light; a sample or scattering cell; a lens located outside the sample
19 or scattering cell, on the side of the incident light source, but interposed between the
20 incident source of light and the scattering cell, so that it is in the pathway of the
21 incident light source; and a photodetector. Figures 11, 12 and 13 marked prior art

1 shows at least three different schematics of the prior art apparatus traditionally used in
2 light scattering studies. The process of using the prior art light scattering apparatus
3 includes the steps of using the incident source of light to generate a beam of light,
4 focusing said beam of light using the lens through a first opening on the incident light
5 source side wall of the sample or scattering cell and out of a second opening on the
6 sample cell wall directly opposite and across the first opening, insuring that the beam
7 at the focal point of the lens inside the sample cell or scattering cell enters the
8 suspension fluid at a zero degree angle relative to the axis of the lens or the path of the
9 incident beam fashion and collecting the scattered light generated by the contents of
10 the sample cell using the photodetector.

11 The incident light source can be any number of lasers because of their properties
12 of coherency, high monochromaticity, and ability to reach extremely high powers.
13 Coherency is a property of wave-like states that enables a laser to exhibit interference.
14 As most commonly used, interference usually refers to the interaction of waves which
15 are correlated with each other either because they come from the same source or
16 because they have the same or nearly the same frequency. Thus, a typical laser emits
17 light in a narrow and well defined beam and with a well defined wavelength or color
18 that is extremely useful as an incident light source in light scattering studies.

19 Some of the lasers used in light scattering techniques include but are not limited
20 to gas lasers such as helium-neon lasers which emit light having wavelengths of 543
21 nm and 633 nm; argon-ion lasers capable of emitting light having wavelengths of 458

1 nm, 488nm, and 514.5 nm; Helium-Silver lasers capable of emitting light having a
2 wavelength of 224; and neon-copper lasers capable of emitting light having a
3 wavelength of 248 nm.

4 The lens that is interposed between the incident light source, i.e. the laser, and
5 the scattering cell is preferably a plano-convex lens. It is capable of taking the
6 collimated or parallel beam of light generated by the laser and traveling parallel to the
7 lens axis and passing through the lens and converging it (or focusing it) to a spot on the
8 axis of the lens at a certain distance behind the lens (known as the focal length) and
9 found within the light scattering cell where it hits the particles that are suspended in the
10 suspension fluid within the light scattering cell.

11 The sample or scattering cell holds the specimen of particles that are to be
12 analyzed and tested. Prior to being placed in the sample or scattering cell, the particles
13 are dispersed in an appropriate suspension fluid. Any one of the many types of sample
14 cells or scattering cells available in the market for light scattering techniques can be
15 used, depending on the light scattering application for which the sample cell will be
16 used. For example it can be the scattering cell disclosed in the **Trundle United States**
17 **Letter Patent No. 3,700,338**, which provides a light scattering cell with an inexpensive
18 optical surface that may be disposed of when it is scratched or otherwise damaged; that
19 may be rapidly changed when the need arises; that minimizes angular distortion; and
20 that minimizes unwanted absorption, refraction and reflection. Such scattering cell
21 comprises a cell machined from a length of cylindrical metal tubing or similar material

1 having a light entrance and viewing apertures. The optical surfaces over the apertures
2 are formed from a thin transparent disposable film which is clamped around the outside
3 of the cylindrical metal tubing (pipe).

4 Alternatively, it can be the scattering cell or straight path sample cell disclosed
5 in **Wertheimer, United States Letters Patent No. 4,265,538** which has the ability to
6 provide a structure that makes possible the measurement of the forward scattered light
7 and the 90 degree scatter in two orthogonal directions without the introduction of wells,
8 insertions, or bends in the sample stream, which can create turbulence. The cell
9 structure has an entrance and an exit window, which respectively form a front and back
10 for the cell, so that the incident light beam is received by the front and the forward
11 scattered light is transmitted through the back as one of the components. The cell also
12 has a prism means with at least one of its faces optically contacting the sample along
13 a side of the cell. Two other faces of the prism means are oriented so that they each
14 pass another component of the scattered light.

15 Still yet, it can be the scattering cell or sample cell disclosed in **Phillips et.al.**
16 **United States Letters Patent No. 4,616,927** which permits the measurement of the
17 light scattering properties of very small liquid-borne sample with negligible
18 background interference from the illumination source. It comprises a right cylinder
19 with a hole bored through a diameter. The cylinder and hole are optically polished and
20 the cylinder is surrounded by an array of detectors lying in the plane of the hole and
21 parallel to the base. Means are provided for introducing and removing a particle

1 bearing sample fluid. The sample introduced into the hole thereby can be illuminated
2 by a collimated light beams whose diameter is much smaller than the diameter of the
3 hole. This beam passes directly through the hole and enters and leaves the cell by
4 means of special windows mounted externally to the cell. Because of the slight
5 difference of refractive index between the fluid and the surrounding glass cell, very
6 little stray or background light enters the detectors, even at very small scattering angles.
7 The invention also provides means for attenuating small angle scattered intensities
8 which are a source of detector saturation in conventional light scattering instruments.

9 Or, if the light scattering technique used is dynamic light scattering then the
10 scattering cell or sample cell can be the one disclosed by **Freud et. al, United States**
11 **Letters Patent 5,485,270**. It comprises a cell housing and a cell chamber, the cell
12 chamber being formed within the cell housing such that the cell chamber is exposed to
13 a membrane which is capable of retaining within the cell chamber the particles of a
14 sample introduced into the cell chamber while allowing a liquid carrier to diffuse across
15 the membrane and an out of the cell chamber.

16 Or, still, it can be the modified light scattering cell disclosed in **Wyatt et.al**
17 **United States Letters Patent 5,530,540** whereby an eluant of a very small dimension,
18 transverse to its direction of flow, is entrained successively by two sheath flows and
19 presented to a fine light beam that illuminates the entrained eluant as it flows though
20 the light beam. The light scattered by the entrained eluant is collected by detectors
21 outside of a transparent flow cell enveloping the sheath flow entrained eluant. The

1 windows of the transparent flow cell through which the light beam enters and leaves
2 are far removed from the scattering eluant and kept clear of eluant contained particles
3 by means of flow components that will form subsequently one of the eluant sheath flow
4 employed. The eluant source is typically for a fine capillary such as found in capillary
5 electrophoresis, capillary hydrodynamic fractionation and flow cytometry applications.

6 Likewise, the photodetector can be any one of the many photodetectors available
7 for light scattering techniques, in the market today. Generally, the photodetector is a
8 sensor of light or other electromagnetic energy. It acts to collect all of the scattered
9 light beams generated by the fluid suspended particles, in the pathway of the laser
10 beam, at the focal point of the lens inside the sample or scattering cell; and to convert
11 them into a signal that can be measured and interpreted. At its most basic, the
12 photodetector comprises some sort of optic and electronic system that responds to
13 photons hitting it, produces a current, and converts the current into a voltage.

14 The optic and electronic system that responds to photons hitting it to produce a
15 current can be a photo diode detector. It can comprise an optic lens system that guides
16 the scattered light onto the photodiode. The photodiode is a semi-conductor diode that
17 functions as a photodetector. A diode is basically a component that restricts the
18 direction of flow or charge carriers such as electrons or ions. Essentially it allows an
19 electric current to flow in one direction, but blocks it in the opposite direction. Thus,
20 a diode is essentially an electronic version of a check valve. A photodetector is a
21 sensor of light or other electromagnetic light. It is usually provided either with a

1 window or an optical fiber connection so that it can let in the light to the sensitive part
2 of the device where it excites electrons and creates a current. The current is then
3 converted into voltage, using the current-to-voltage converter, which voltage is used
4 to record and interpret the results of shining the laser beam on the suspended particles
5 in the sample or scattering cell.

6 Or, the optic and electronic system that responds to photons hitting it to produce
7 a current can be a photomultiplier. A photomultiplier is an extremely sensitive detector
8 of light in the ultraviolet, visible and near infrared wavelengths of light. This detector
9 multiplies the signal produced by light that hits it as much as 10^8 , from which single
10 photons can be resolved. It comprises a photocathode, several dynodes and an anode.
11 The photocathode is a negatively charged electrode coated with a photosensitive
12 compound. When it is struck by light, it emits electrons. A dynode is one of a series
13 of polished metal electrodes within the photomultiplier. Each dynode is more
14 positively charged than its predecessor. Secondary emission occurs at the surface of
15 each dynode. In other words, the electrons emitted from the photocathode, as a result
16 of being hit by photons, are accelerated towards the dynode where if they strike with
17 sufficient energy, they will knock off many more electrons from the surface of the
18 dynode. These new electrons are then accelerated toward another dynode where even
19 more electrons are emitted. This process occurs typically ten or so times. The result
20 is that the tiny and normally undetectable current from the photocathode becomes a
21 larger and more easily measurable current flowing in the final anode circuit. In fact, by
22 the time this process has been repeated at each of the dynodes, 10^5 to 10^7 electrons have

1 been produced from each incident photon that hits the photocathode. The anode is an
2 electrode through which electric current flows into a polarised electrical device, such
3 as a current-to-voltage converter.

4 Finally, the optic and electronic system that responds to photons hitting it to
5 produce a current can be an avalanche photodiode. An avalanche photodiode is a
6 photodetector that can be regarded as the semiconductor analog to a photomultiplier.
7 By applying a high reverse bias voltage (typically 100-200 V in silicon) an avalanche
8 photodiode can show an internal current gain effect (around 100) due to impact
9 ionization (avalanche effect). Impact ionization in an avalanche photodiode is the
10 process by which a photon is absorbed to create electrons. If the absorption occurs in
11 a region of high electrical field then it can result in an avalanche breakdown, a process
12 that is exploited in an avalanche photodiode to provide gain. Avalanche breakdown
13 is a phenomenon, a form of electric current multiplication that can allow very large
14 currents to flow. However, the avalanche current must be limited if the avalanche
15 breakdown is to work successfully without any breakdown.

16 The process of using the light scattering apparatus described herein above is
17 fraught with problems and opportunities for errors and in the particle light scattering
18 results. These errors in turn can lead to many erroneous conclusions in connection with
19 the particles being studied. As a result, since the inception of light scattering
20 technology it has been imperative to address these problems and minimize the
21 opportunities for errors in the data generated and the conclusions made there from.

1 The errors arise from the background effects or contributions created by the
2 sample cell or scattering cell itself (hereinafter "the sample cell"), and from the
3 suspension fluid. For example, one background effect arises at the entrance point of the
4 beam through the first entrance opening in the sample cell. The refractive index of the
5 material covering such first entrance point can be silica, quartz, glass or even plastic.
6 The refractive index of such materials is different from the refractive index of the
7 outside environment through which the beam travels before it enters the sample cell,
8 and different from the suspension fluid in which the particles are dispersed, inside the
9 sample cell. As a result the beam of light entering the cell gets bent as it travels from
10 the outside environment through the material covering the first opening in the sample
11 cell and then gets bent again as it travels through the suspension fluid. Consequently,
12 the chances that the beam of light will enter into the suspension fluid at a zero degree
13 angle relative to the axis of the lens or the path of the incident light beam is much lower
14 than theorized.

15 Another background effect arises from the light scattering effects of the
16 impurities contained in the materials covering the first entry opening of the sample cell,
17 the second exit opening of the sample cell, the walls themselves of the sample cell, and
18 the suspension fluid, itself. As pure as such materials and fluids can be made, they will
19 still contain impurities. For example, an article entitled "Liquid Phase Particulate
20 Contaminants in Water" by Wilbur Kay, *Journal of Colloid and Interface Science*,
21 Volume 46, No. 3, March 1974 pages 543-44, describes the range of contaminants
22 which have been encountered in pure water. Thus, as the light beam hits the particles

1 dispersed in the suspension fluid it will also hit these impurities. Just as the dispersed
2 particles will cause the light beam to scatter, so too will the impurities cause the light
3 beam to scatter. The impurities' light scattering can very well mask or add to the light
4 scattering of the particles themselves leading to errors in the results.

5 Yet another background effect can arise from the light exiting the second exit
6 opening of the sample cell, opposite to and coaxial to the first entry opening. Some of
7 the light traveling through the suspension fluid will not necessarily make it out the
8 second exit opening. Partly as a result of hitting the particles themselves and partly
9 because of the refractive index of the suspension fluid and partly because of the
10 spreading of the beam of light through the suspension fluid, it will hit the walls of the
11 sample cell framing and forming the outer perimeter or edges of the second exit
12 opening. When it does, it will bounce back or reflect back into the suspension fluid,
13 thereby further masking the light scattering results of the particles and further skewing
14 the results therefrom.

15 Still another background effect can arise from air bubbles. Some liquids,
16 notably water, will not wet, clean silica surfaces easily. If air does get into the sample
17 cell, it will form at least one air bubble on any one of the silica windows covering the
18 openings of the sample cell. This air bubble acts like a lens scattering the incident light
19 or even the exiting light, rendering meaningful measurements impossible.

1 Some of the prior art has attempted to provide solutions for the elimination of
2 the errors that can arise as a result of the background effects created or contributed by
3 the sample cell, or the suspension fluid. One technique has been to stream the particle-
4 containing/suspension fluid specimen through a small circular orifice, perpendicularly,
5 *i.e.*, across the pathway of the light beam generated by the incident light source. This
6 works quite well for aerosols, where the air stream carrying the particles has essentially
7 no focusing power. However, it does not work so well with hydrosols, *i.e.*, particles
8 suspended in liquids, because the liquid stream spreads the incident light beam, which
9 can both mask the particles' scattered light and distort it.

10 While **Wallace, United States Letters Patent No. 4,178,103** appears to have
11 solved the problem of streaming hydrosols through a small orifice across a laser beam,
12 such solution is of limited application. It does not address those applications where
13 streaming does not work. Nor does any of the prior art address the effects of conducting
14 light scattering analysis with a probe. Finally, none of the prior art suggests or focuses
15 on providing any solutions to the problem of sample cell and suspension fluid
16 background effect contribution by changing the anatomy and configuration, as well as
17 the positioning, of the lens itself relative to the suspension fluid and the particles
18 diffused therein.

19 Accordingly, there clearly still is a need for an apparatus and method that can
20 address the problems of masking or skewing of particle light scattering results, by the
21 background effects created or contributed by the sample cell itself, and/or the

1 suspension fluid. Without such apparatus and method errors will continue to exist, or
2 at least suspected to exist; confidence in the light scattering results and the scientific
3 conclusions arising therefrom will remain at relatively the same level as currently
4 existing; and light scattering technology will continue to exist on the fringes of industry
5 and science application, as a research and development tool, instead of being widely
6 accepted as a tool for quality control and consistency in the infinite number of areas,
7 limited only by imagination, where it might find application.

8

9

OBJECTS OF THE INVENTION

10 **IT IS THEREFORE AN OBJECT** of the present invention to minimize the
11 inherent problems and errors arising therefrom, in light scattering technology
12 applications, not heretofore effectively addressed by the prior art.

13 **IT IS ANOTHER OBJECT** of the present invention to minimize, if not totally
14 eliminate, those errors in the results of light scattering technology applications, that
15 arise from the background effects created or contributed by the sample cell, *i.e.*, the
16 coverings of the entry and exit openings of the sample cell, and the walls thereof.

17 **IT IS ANOTHER OBJECT** of the present invention to minimize, if not totally
18 eliminate, those errors in the results of light scattering technology applications, that
19 arise from the background effects created or contributed by the suspension fluid.

1 **IT IS YET ANOTHER OBJECT** of the present invention to minimize, if not
2 totally eliminate, any and all refractive index effects on the light beam generated by the
3 incident light source, as the light beam travels and transitions from one medium and
4 into another.

5 **IT IS STILL ANOTHER OBJECT** of the present invention to generate a light
6 beam that will, more likely than not, enter the suspension fluid at a zero degree angle
7 relative to the axis of the lens and the incident path of the light beam.

8 **IT IS A FURTHER OBJECT** of the present invention to minimize, if not
9 completely eliminate, the light scattering masking of particles' light scattering results,
10 effectuated by the impurities found in the sample cell, *i.e.*, the coverings of the entry
11 and exit openings of the sample cell, and the walls thereof; and the impurities found in
12 the suspension fluid.

13 **IT IS YET A FURTHER OBJECT** of the present invention to minimize, if not
14 completely eliminate, the light scattering masking of particles' light scattering results,
15 effectuated by the reflection or bouncing back into the suspension fluid, of light not
16 successfully exiting the sample cell.

17 **IT IS STILL A FURTHER OBJECT** of the present invention to minimize, if
18 not completely eliminate, the light scattering masking of particles' light scattering
19 results, effectuated by air bubbles trapped in the sample cell or in the suspension fluid.

1 **ANOTHER OBJECT** of the present invention is to provide an apparatus and
2 method that can be incorporated into a light scattering sample cell, for both static and
3 dynamic light scattering applications.

4 **IT IS ANOTHER OBJECT** of the present invention to provide a light
5 scattering sample cell suitable for the use of micro-volumes of samples of liquid-
6 suspended particles, during a flow-through, dynamic light-scattering analysis of said
7 particles.

8 **IT IS ANOTHER OBJECT** of the present invention to eliminate the need for
9 large volume samples of liquid-suspended particles, during a flow-through, dynamic,
10 light-scattering analysis.

11 **IT IS YET ANOTHER OBJECT** of the present invention to permit the use of
12 truly micro-volume samples of liquid-suspended, hazardous particles during batch
13 mode, dynamic light scattering analysis.

14 **IT IS STILL ANOTHER OBJECT** of the present invention to provide a
15 dynamic, light- scattering flow cell and method for use in both batch and flow-through
16 modes of dynamic, light-scattering analyses using truly micro-volume samples of
17 liquid-suspended particles.

1 **IT IS A FURTHER OBJECT** of the present invention to provide a dynamic,
2 light-scattering flow cell and method for use in both batch and flow-through modes of
3 dynamic and static, light-scattering analyses, which flow cell and method permit a high
4 pressure seal, without the use of additional seals, gaskets, windows or other sealing
5 compounds (e.g. epoxy, silicone or similar) on the walls thereof.

6 **IT IS YET A FURTHER OBJECT** of the present invention to provide a
7 dynamic, light- scattering flow cell and method for use in both batch and flow-through
8 modes of dynamic and static, light-scattering analyses, which flow cell and method do
9 away with the need to find a broad range of chemically compatible, broad range
10 temperature, insensitive seals, gaskets or sealing compounds.

11 **IT IS STILL A FURTHER OBJECT** of the present invention to eliminate the
12 need for refractive index matching or refractive index correction during both batch and
13 flow-through modes of dynamic and static, light-scattering analyses, because the surface
14 of the lens through which a laser is focused into a micro-volume sample of liquid-
15 suspended particles is in direct contact with said sample.

16 **IT IS ANOTHER OBJECT** of the present invention to provide an apparatus
17 and method that can be incorporated into a light scattering probe capable of minimizing,
18 if not totally eliminating the light scattering masking of particles' light scattering results,
19 effectuated by a densely populated or minimally populated suspension fluid, and the
20 impurities thereof.

1 **IT IS YET ANOTHER OBJECT** of the present invention to minimize, if not
2 completely eliminate, the light scattering masking of particles' light scattering results,
3 effectuated by the impurities found in the sample cell, *i.e.*, the coverings of the entry and
4 exit openings of the sample cell, and the walls thereof; and the impurities found in the
5 suspension fluid through the use of a spherical or ball lens.

6 **IT IS STILL ANOTHER OBJECT** of the present invention to produce
7 particles' light scattering results and scientific conclusions therefrom, that inspire the
8 scientific community with confidence regarding their precision and accuracy, and the
9 good and services community regarding their widespread application to any and all
10 industries at large.

11 These objects, as well as other objects and advantages will become more
12 apparent in the description that is set forth herein below, particularly when read in
13 conjunction with the accompanying drawings.

14 **SUMMARY OF THE INVENTION**

15 The inventive optical spherical lens light scattering apparatus and method for use
16 thereof comprises an incident light source, a spherical or ball optical lens system and a
17 photo detector. The spherical or ball lens optical system comprises at least one ball or
18 spherical lens being in direct contact with the specimen of particles to be tested
19 dispersed in a suspension fluid; said ball lens having, as a result of its spherical

1 geometry, and as compared to a plano-convex or convex lens, a relatively short focal
2 point. When the incident light source is used to generate a beam of light, and the beam
3 of light is then focused through the spherical or ball lens, it lands at a point that is much
4 closer to the lens, as compared to the point it would normally land on, when it is focused
5 through a plano-convex, or a convex lens. It is the proximity of the spherical or ball
6 lens' focal point to the spherical or ball lens itself, the contact of the ball lens to the
7 suspension fluid containing the particles to be tested, and the placement of the specimen
8 of particles dispersed in the suspension fluid within such focal point, that minimizes, if
9 not eliminates all light scattering masking of particles' light scattering results.

10 The inventive method for use of the inventive optical spherical lens light
11 scattering apparatus comprises at least the following steps: (i) using an incident light
12 source to generate a beam of light or a beam of energy having a certain wavelength and
13 frequency; (ii) focusing the beam of light or beam of energy through a spherical or ball
14 lens; (iii) placing a specimen of particles, dispersed in a suspension fluid, directly in the
15 focal point of the spherical lens, so that the focused beam of light or focused beam of
16 energy hits the suspended particles at exactly the focal point of the spherical or ball lens,
17 and the suspended particles scatter the beam of light; and (iv) directing the scattered
18 light through a spherical or ball lens onto a photodetector.

19 In one embodiment of the inventive optical spherical lens light scattering
20 apparatus, the spherical or ball lens optical system is integrally but removably fixed on the
21 walls of a sample cell to form part of the walls thereof and be in direct contact with the

1 specimen of particles dispersed in a suspension fluid. More specifically, the sample cell
2 utilizing the spherical or ball lens optical system, comprises a cell block having: an
3 inner, centrally enclosed, hollow chamber; at least three, through-bores placed on the
4 same plane, each of said through-bores beginning at the outer surface of the cell block
5 and ending at and opening into said inner, centrally enclosed, hollow chamber; two of
6 said through-bores juxtaposed, end to end, so that their longitudinal axes are along the
7 same line, but spaced apart the full width of said inner, centrally enclosed, hollow
8 chamber, such that their openings into said inner, centrally enclosed, hollow chamber
9 are positioned exactly across from each other, at opposite walls of said inner, centrally
10 enclosed, hollow, chamber, whereby one of said two, juxtaposed through-bores can act
11 as an incident light through-bore and the other of said two, juxtaposed through-bores
12 can act as an exiting light through-bore; said remaining third through-bore interposed
13 between said two, end to end, juxtaposed through-bores, so that its longitudinal axis is
14 perpendicular to the co-aligned longitudinal axes of the two juxtaposed through bores,
15 and its opening into said inner, centrally enclosed, hollow, chamber is approximately
16 equidistant from the openings of the juxtaposed through-bores, at the opposite walls of
17 said inner, centrally enclosed, hollow chamber, whereby it can act as a measurement
18 through-bore; at least three, spherical or ball lenses, each of said spherical or ball lenses
19 removably and frictionally fixed within each of said through-bores ending at and
20 opening into said inner, centrally enclosed, hollow, micro volume chamber, such that
21 said spherical or ball lenses completely fill and seal said through-bores and and partially
22 form the walls of the inner, centrally enclosed, hollow, chamber; and a sample entry
23 port and a sample exit port, both funnel shaped, with each of their wider ends placed

1 on the outer surface of the cell block and each of their narrow tips leading into and
2 terminating at the inner, centrally enclosed hollow chamber.

3
4 The process of utilizing the sample cell utilizing the spherical or ball lens optical
5 system comprises the following steps: installing the inventive sample cell on the chassis
6 or base of a light-scattering detector; communicatively connecting a fiber optic system
7 via an optical detecting fiber to said measuring through-bore of the inventive sample
8 cell; by using the appropriate hardware, supplying power and generating a focused
9 beam of light; optionally adjusting the temperature of the light beam generating module
10 for the purpose of maintaining the stability of the light beam intensity during the use of
11 the inventive sample cell; simultaneously focusing the light beam both through the entry
12 through-bore and the spherical or ball lens frictionally but removably fixed at said entry
13 through-bore, adjacent to said inner, centrally enclosed, hollow, chamber; via the sample
14 entry port filling the inner, centrally enclosed, hollow, chamber, either manually or via
15 a pump system, with a sample of liquid suspended particles needing to be measured and
16 characterized; focusing the beam of light through the incident light through-bore and
17 through its corresponding spherical or ball lens onto the sample of liquid suspended
18 particles and out the exiting through-bore and its corresponding spherical or ball lens,
19 adjacent to the inner, centrally enclosed hollow chamber. The molecules or particles
20 within the laser beam's path scatter light. The spherical or ball lens located at the
21 opening of the measurement through-bore, between the incident through-bore and the
22 exiting light through-bore spherical ball lenses, collimates the scattered light which is
23 then refocused onto the detecting fiber, *i.e.*, the scattered light intensity is picked up by

1 the optical detector via a fiber optic system, and converted to provide data that generates
2 information regarding the physical characteristics and chemical properties of the liquid-
3 suspended particles filling the chamber.

4 In another embodiment of the inventive optical spherical lens light scattering
5 apparatus, the spherical or ball lens optical system is integrally but removably fixed on the
6 lower end of a light scattering probe to form part of the lower end wall thereof and be in direct
7 contact with the specimen of particles dispersed in a suspension fluid, when the probe is
8 immersed therein. More specifically, the light scattering probe utilizing the spherical or ball
9 lens optical system, comprises a body, preferably cylindrical having: an inner, centrally
10 enclosed, hollow core formed by a single through-bore beginning at the outer surface of the
11 lower end of the body; at least one, spherical or ball lens removably and frictionally fixed
12 within said through-bore ending at said lower end of said body, such that said spherical or ball
13 lens completely fills and seals said lower end of said through-bore and and partially forms the
14 lower wall of the probe, thereby completely sealing the inner, centrally enclosed, hollow, core
15 of the probe to create a cavity therein; an incident light source and at least one optical detecting
16 fiber enclosed within said inner, centrally enclosed hollow core of the probe; and a
17 photodetector.

18 The process of utilizing the probe, having the spherical or ball lens optical system,
19 comprises the following steps: communicatively connecting the at least one optical
20 detecting fiber to said photodetector; supplying power and using the incident light
21 source within the inner chamber of the probe to generate a focused beam of light;
22 focusing the light beam on the spherical or ball lens, frictionally but removably fixed

1 at said lower end of the probe sealing the through-bore, such that the beam penetrates
2 and enters the spherical or ball lens at a zero degree angle relative to the path of said
3 incident light beam; immersing the probe into a sample of liquid suspended particles
4 needing to be measured and characterized; focusing the beam of light through its
5 corresponding spherical or ball lens onto the sample of liquid suspended particles. The
6 molecules or particles within the laser beam's path at the focal point of the spherical or
7 ball lens, and adjacent thereto scatter light. The spherical or ball lens then collimates
8 the scattered light and focuses it back through said spherical or ball lens, onto the
9 detecting fiber, which in turn is sends it to the photodetector and converts to provide
10 data that generates information regarding the physical characteristics and chemical
11 properties of the liquid-suspended particles in the specimen, in which the probe was
12 immersed.

13 **BRIEF DESCRIPTION OF THE DRAWINGS**

14 While the specification shall conclude with claims which will particularly point
15 out and distinctly claim the present invention, it is believed that the present invention
16 will be better understood from the following detailed description taken in conjunction
17 with the accompanying drawings in which like numerals represent identical elements
18 and wherein:

19 **Figure 1** is a plan view generally showing one of the many environments
20 that the inventive optical spherical or ball lens light scattering

1 apparatus can be installed into for use in a flow-through mode,
2 laser, light-scattering analysis;

3 **Figure 2** is a three-dimensional perspective view of the inventive single
4 chamber, light-scattering, sample cell comprising the inventive
5 spherical or ball lens optical system installed in the laser, light-
6 scattering detector of **Figure 1** ;

7 **Figure 3** is a three-dimensional perspective view of the inventive single
8 chamber, light-scattering, sample cell comprising the inventive
9 spherical or ball lens optical system, of **Figure 2**;

10 **Figure 4** is a side plan view of the inventive, single chamber, light-
11 scattering, sample cell of **Figure 3**;

12 **Figure 5** is a top plan view of the inventive, single chamber, light-
13 scattering, sample cell of **Figure 3**;

14 **Figure 6** is a bottom plan view of the inventive, single chamber, light-
15 scattering, sample cell of **Figure 3**;

16 **Figure 7** is a section view of the inventive, single chamber, light-scattering,
17 sample cell of **Figure 6** taken along A-A' and showing an
18 alternate embodiment having at least four through-bores and at
19 least two measuring through-bores arranged along the x-axis;

20 **Figure 8** is a section view of the inventive, single chamber, light-scattering,
21 sample cell of **Figure 6** taken along A-A' and showing a first
22 embodiment having at least three through-bores and at least one
23 measuring through-bore arranged along the x-axis;

1	Figure 9	is a section view of the inventive, single chamber, light-scattering,
2		sample cell of Figure 6 taken along B-B' and showing an
3		embodiment having at least three through-bores and with the
4		incident through-bore and the exit through-bore arranged along the
5		x-axis;
6	Figure 10	is a three-dimensional perspective of an alternate embodiment of
7		the inventive, single chamber, light-scattering sample cell,
8		wherein there is a plurality of measuring through-bores.
9	Figure 11	consists of various views of the inventive probe with the spherical
10		or ball lens optical system within its inner hollow core.
11	Figures 12	
12	through 14	Prior art plan views showing at least three different schematics of
13		the prior art apparatus traditionally used in light scattering studies
14	Figure 15	Plan view of schematic of one of the embodiments of the inventive
15		optical spherical lens light scattering apparatus.

LIST OF ELEMENTS AND THEIR RESPECTIVE
IDENTIFYING NUMERALS

18	<u>NO</u>	<u>ELEMENT</u>
19	10	The inventive optical spherical lens light
20		scattering apparatus
21	12	Incident light source
22	14	Spherical or ball optical lens system

1	20	Cell block
2	30	Inner, centrally enclosed, chamber
3	40	Incident through-bore
4	42	Inner opening of incident through-bore
5	50	Exit through-bore
6	52	Inner opening of exit through-bore
7	60	Measuring through-bore
8	62	Inner opening of measuring through-bore
9	70	Incident Spherical or ball lens
10	72	Lens mount in probe
11	80	Exit spherical or ball lens
12	90	Measuring spherical or ball lens
13	100	Sample entry port
14	110	Sample exit port
15	120	Sample delivery tubing
16	130	Acorn nuts
17	140	Fiber Optic System
18	142	Fibers
19	146	Probe inner hollow core
20	148	Probe Stainless Steel Sleeve
21	150	Photodetector

1	152	Probe body
2	154	Probe upper end
3	156	Probe lower end
4	158	Probe through bore forming inner hollow
5		chamber

6 **DETAILED DESCRIPTION OF THE INVENTION**

7 Referring more specifically to the drawings, **Figures 1-10** on the one hand, and
8 **Figure 11** on the other, depict at least two of the embodiments of the inventive optical
9 spherical lens light scattering apparatus at **10**, and methods for use thereof. They are
10 designed to: (i) minimize, if not totally eliminate, those errors in the results of light
11 scattering technology applications, that arise from the background effects created or
12 contributed by the sample cell; (ii) minimize, if not totally eliminate, those errors in the
13 results of light scattering technology applications, that arise from the background effects
14 created or contributed by the suspension fluid; (iii) minimize, if not totally eliminate,
15 any and all refractive index effects on the light beam generated by the incident light
16 source, as the light beam travels and transitions from one medium and into another; (iv)
17 generate a light beam that will, more likely than not, enter the suspension fluid at a zero
18 degree angle relative to the incident path or axis of said light beam; (v) minimize, if not
19 completely eliminate, the light scattering masking of particles' light scattering results,
20 effectuated by the impurities found in the sample cell and the impurities found in the
21 suspension fluid; (vi) minimize, if not completely eliminate, the light scattering masking

1 of particles' light scattering results, effectuated by the reflection or bouncing back into
2 the suspension fluid, of light not successfully exiting the sample cell; and (vii)
3 minimize, if not completely eliminate, the light scattering masking of particles' light
4 scattering results, effectuated by air bubbles trapped in the sample cell or in the
5 suspension fluid.

6 The inventive optical spherical lens light scattering apparatus at 10 uses certain
7 properties of light to determine, among other things, the size of particles, particle size
8 distributions, and molecular weights. The term "particles" as being used herein, denotes
9 objects whose size can range from macromolecule size at one extreme end of the
10 spectrum, to large mammalian cell or even phytoplankton size at the other end.
11 Macromolecule size in turn, can vary from one nanometer (one billionth (10^{-9}) of a
12 meter) in length to tens of micrometers.

13 The properties of light that the inventive optical spherical lens light scattering
14 apparatus at 10 uses to determine particle size, are the light's wavelength and its
15 frequency. Furthermore, the particles' properties that the inventive optical spherical lens
16 light scattering apparatus at 10 uses are their ability to scatter light that is shone upon
17 them. Specifically when a beam of radiant energy is incident upon a particle, a portion
18 of this energy will be scattered in all directions. The frequency or intensity of this
19 scattered light or radiant energy will depend on the wavelength of the incident radiant
20 energy, upon the difference in the refractive index of the particles with respect to the
21 medium in which they are suspended, upon the size and shape of the particles and upon

1 the angle at which the scattered energy is observed. In other words, one of the things
2 that the shift in the light frequency of scattered light is related to is the size of the
3 particles causing the shift. As was set forth herein above, the smaller particles move
4 faster. They have a higher average velocity, which in turn causes a greater shift in the
5 light frequency than the shift caused by the larger particles. It is this difference in the
6 frequency of scattered light among the particles of different sizes that is used to
7 determine the size of the particles present.

8 The inventive optical spherical lens light scattering apparatus at **10** can be used
9 both in static light scattering and dynamic light scattering. If it is used in static light
10 scattering, it will measure the intensity of the scattered light, $I(q)$, as a function of the
11 scattering angle. These measurements can be used for the determination of the
12 equilibrium structure factor, the particle form factor, the molecular weight and the
13 thermodynamic quantities such as the radius of gyration and the second virial
14 coefficients in polymer solutions. If it is used in dynamic light scattering, it will
15 measure the decay of the intensity fluctuations via the intensity auto-correlation
16 function $G(q,t)$, which provides a convenient way to measure diffusion coefficients of
17 macromolecules in dilute solutions and the dynamics of polymers in solutions of varying
18 concentrations.

19 A schematic diagram of the inventive optical spherical lens light scattering apparatus
20 at **10** and method for use thereof is shown at **Figure 15**. It comprises: an incident light source
21 **12**, a spherical or ball optical lens system **14** and a photodetector **150**. The spherical or ball
22 lens optical system **14** comprises at least one spherical or ball lens **70** having, as a result of its

1 spherical geometry, and as compared to a plano-convex lens or a convex lens, a relatively short
2 focal point. When the incident light source **12** is used to generate a beam of light, and the beam
3 of light is then focused through the spherical or ball lens **70**, it lands at a point that is much
4 closer to the lens **70**, as compared to the focal point it would normally land on, when focused
5 through a plano-convex, or a convex lens. It is the proximity of the spherical or ball lens' **70**
6 focal point to the spherical or ball lens **70** itself, that minimizes, if not eliminates all light
7 scattering masking of particles' light scattering results.

8 The incident light source **12** can be any number of lasers because of their properties of
9 coherency, high monochromaticity, and ability to reach extremely high powers. They include
10 but are not limited to gas lasers such as helium-neon lasers which emit light having
11 wavelengths of 543 nm and 633 nm; argon-ion lasers capable of emitting light having
12 wavelengths of 458 nm, 488nm, and 514.5 nm; Helium-Silver lasers capable of emitting light
13 having a wavelength of 224; and neon-copper lasers capable of emitting light having a
14 wavelength of 248 nm.

15 The spherical or ball lens **70** that is interposed between the incident light source **12**, i.e.
16 the laser, and the specimen is preferably a spherical or ball lens. It is capable of taking the
17 collimated or parallel beam of light generated by the laser **12** and traveling parallel to the lens
18 axis and passing through the lens and converging it (or focusing it) to a spot on the axis of the
19 lens at a very short distance behind the lens (known as the focal length). As a result, the light
20 scattering volume is very close to the spherical or ball lens **70**.

21

22 The photodetector **150** can be a photomultiplier, an avalanche photodiode, or a charge-
23 coupled device (CCD) which is an image sensor consisting of an integrated circuit containing

1 an array of linked, or coupled, light-sensitive capacitors.

2 The inventive method for use of the inventive optical spherical lens light
3 scattering apparatus **10** comprises at least the following steps: (i) using an incident light
4 source **12** to generate a beam of light or a beam of energy having a certain wavelength
5 and frequency; (ii) focusing the beam of light or beam of energy through a spherical or
6 ball lens **70**; (iii) placing a specimen of particles, dispersed in a suspension fluid, at the
7 focal point of the spherical or ball lens **70** or the scattering volume of the spherical or
8 ball optical lens system, so that the focused beam of light or focused beam of energy hits
9 the suspended particles at exactly the focal point of the spherical or ball lens **70** and the
10 suspended particles scatter the beam of light; and (iv) directing that scattered light
11 produced at a ninety degree angle to the axis of the lens through a spherical or ball lens
12 **90** onto a photodetector **150**.

13 Referring more specifically to the drawings, **Figures 1-10** generally depicts one
14 embodiment of the inventive optical spherical lens light scattering apparatus set forth
15 herein above, *i.e.*, an inventive, single chamber, light-scattering sample cell at **10**, as
16 used in a flow-through mode, laser, dynamic light-scattering analysis system. It is
17 designed to achieve all of the objects set forth herein above, as well as: (i) allow the use
18 of micro-volumes of samples of liquid-suspended particles, during the flow-through,
19 laser, dynamic, light-scattering analysis of the particles; (ii) eliminate the need for large
20 volume samples of liquid-suspended particles, during the flow-through, laser, dynamic,
21 light-scattering analysis; (iii) permit the use of truly micro-volume samples of liquid-

1 suspended, hazardous particles during both batch mode, laser, dynamic, light-scattering
2 analysis; (iv) provide a laser, dynamic, light- scattering flow cell and method for use in
3 both batch and flow-through modes of laser, dynamic, light-scattering analyses using
4 truly micro-volume samples of liquid-suspended particles; (v) provide a laser, dynamic,
5 light-scattering flow cell and method for use in the flow-through mode of the laser,
6 dynamic, light-scattering analysis, which flow cell and method permit a high pressure
7 seal, without the use of additional seals, gaskets, windows or other sealing compounds
8 (e.g. epoxy, silicone or similar); (vi) provide a laser, dynamic, light- scattering flow cell
9 and method for use in the flow-through mode of dynamic, light-scattering analysis,
10 which flow cell and method do away with the need to find a broad range of chemically
11 compatible, broad range temperature, insensitive seals, gaskets or sealing compounds;
12 and (vii) eliminate the need for refractive index matching or refractive index correction
13 during the flow-through mode of dynamic, light-scattering analysis.

14 The inventive single chamber, light-scattering sample cell at **10** enables, for the
15 first time in the art, the use of extremely small volumes, *i.e.*, micro-volumes, of 2 micro
16 liters to 8 micro-liters of sample of liquid-suspended particles during flow-through,
17 laser, dynamic, light-scattering analysis of said particles, by permitting the focusing of
18 a laser beam within 0.5 mm of the lens surface of the flow-cell. This, in turn, allows for
19 the gathering of a tremendous amount of information about said particles; information
20 that heretofore could not be collected in a flow-through mode of laser, dynamic, light-
21 scattering analysis due to the long focusing distances of the laser beam from the lens.

1 The inventive micro-volume, single chamber, laser, dynamic, light-scattering
2 sample flow cell **10**, as can be seen from **Figure 2** is removably inserted and fixed
3 directly on the chassis, or base, or housing of a laser, dynamic, light-scattering
4 photodetector **150**. The photodetector can be used in both a flow-through mode and a
5 batch mode, laser light-scattering analysis system.

6 The inventive micro-volume, single chamber, dynamic, light-scattering sample
7 flow cell **10** comprises a cell block **20**. As can be seen from **figures 7, 8 and 9**, the cell
8 block **20** in turn, comprises an inner, centrally enclosed, hollow, micro-volume chamber
9 **30**, at least three, through-bores **40, 50 and 60**, at least three, spherical or ball lenses **70,**
10 **80 and 90**, a sample entry port **100** and a sample exit port **110**.

11 It is important that the cell block **20** be made of a material that permits a high
12 pressure seal for all of the flow-cell's components, without the use of additional seals,
13 gaskets, windows or other sealing compounds (e.g. epoxy, silicone or similar) and
14 which does away with the need to find a broad range of chemically compatible, broad
15 range temperature, insensitive seals, gaskets or sealing compounds. Furthermore, it has
16 to be compatible with both the solvents in which the specimen particles are suspended,
17 as well as be compatible with the specimen particles themselves. Finally, it must not
18 break down, either physically or chemically, when coming in contact with the solvents
19 and the specimen particles suspended therein, in a wide range of temperatures. One
20 material that has the ability to provide all of the foregoing is Polyetheretherketone
21 (PEEK).

1 In the preferred embodiment, the cell block is made of only PEEK. Not only is
2 it compatible with a very wide range of solvent liquids and sample particles, but is also
3 neutral in that it does not leach contaminants into the sample of liquid-suspended
4 particles; even when it is exposed to temperatures ranging from 0 degrees C to 160
5 degrees C.

6 Sealing in flow-through cells is a well documented problem. Sealing is
7 necessary to continue to maintain the high pressure necessary for the analysis. Many
8 times however, the materials used during the sealing process are incompatible with the
9 chemical solvents used in the analysis of the specimen particles. Therefore, finding a
10 broad range of chemically compatible, broad range temperature insensitive seal, gaskets
11 or sealing compounds has always been a challenge. Use of PEEK in the cell block
12 provides for a high pressure seal without the use of additional seals, gaskets, windows
13 or other sealing compounds (e.g. epoxy, silicone or similar), thereby eliminating such
14 challenge.

15 The cell block **20** comprises an inner, centrally enclosed, hollow, micro-volume
16 chamber **30**. The cell block **20** further comprises at least three, through-bores, an
17 incident through-bore **40**, an exit through-bore **50**, and at least one measuring through-
18 bore **60**. The three through-bores are placed on the same horizontal plane, within the
19 cell block **20**. Each of said through-bores begins at the outer surface of the cell block
20 **20**, and terminates at an opening into said inner, centrally enclosed, hollow, micro-
21 volume chamber **30**. The incident through-bore **40** and the exit through-bore **50** are

1 juxtaposed, end to end, so that their longitudinal axes are along the same line. However,
2 they do not abut. Rather, they are spaced apart the full width of the inner, centrally
3 enclosed, hollow, micro-volume chamber **30**, such that their terminating openings **42**
4 and **52** into said inner, centrally enclosed, hollow micro-volume chamber respectively,
5 are positioned exactly across from each other, at opposite walls of the inner, centrally
6 enclosed, hollow, micro-volume chamber **30**. Thus, when a laser beam is focused
7 through the incident through-bore **40**, it enters into the inner, centrally enclosed, hollow,
8 micro-volume chamber **30**, and emerges out the exit through-bore **50**.

9 Said measuring through-bore **60**, in turn is interposed between said end to end,
10 juxtaposed incident through-bore **40** and exit through-bore **50**, so that the through-bore
11 **60**'s longitudinal axis is perpendicular to the co-aligned longitudinal axes of the two
12 juxtaposed through-bores **40** and **50**, and its terminating opening **62**, into said inner,
13 centrally enclosed, hollow, micro-volume chamber is approximately equidistant from
14 the terminating openings **42** and **52** of the juxtaposed through-bores **40** and **50**
15 respectively, at the opposite walls of said inner, centrally enclosed, hollow micro-
16 volume chamber.

17 The cell block **20**, as set forth above, further comprises at least three spherical
18 or ball lenses **70**, **80** and **90** respectively. While a wide variety of spherical or ball
19 lenses can be used, in the preferred embodiments the ball lenses are either rubies or
20 sapphires due to their strength and purity. Their diameters range from 0.5 mm to 10
21 mm. Each of said spherical ball lenses **70**, **80** and **90** respectively, is removably and

1 frictionally fixed at each of the through- bores' terminating openings **42, 52 and 62**
2 located on the walls of said inner, centrally enclosed, hollow, micro-volume chamber
3 **30**, The PEEK material of the cell block **20**, has enough elasticity to give a bit when the
4 spherical or ball lenses are pressed into the through-bores' terminating openings, but
5 enough strength and resiliency to then wrap around said spherical or ball lenses, and seal
6 them into place, without leaks or compromising the integrity of the inner, centrally
7 enclosed, hollow, micro-volume chamber. Thus, as can be seen from **Figures 7, 8 and**
8 **9**, said spherical ball lenses, not only act as lenses for the laser beam, but also partially
9 form and seal the wall of the inner, centrally enclosed, hollow, micro-volume chamber.

10 The cell block **20**, further comprises a sample entry port **100** and a sample exit
11 port **110**. As can be seen from **figures 7, 8 and 9**, their shape is generally funnel shaped.
12 Each of their wider ends is placed on the outer surface of the cell block and each of
13 their narrow tips leads into and terminates at the inner, centrally enclosed, hollow,
14 micro-volume chamber **30**. During use, the sample entry port **100** and the sample exit
15 port **110** are penetrated by acorn nuts **130**. The PEEK material of the cell block **20**, has
16 enough elasticity to give a bit when the acorn nuts are pressed to penetrate said sample
17 entry port **100** and said sample exit port **110**, but enough strength and resiliency to then
18 wrap around the acorn nuts and seal them into place, without leaks or compromising
19 the integrity of the inner, centrally enclosed, hollow, micro-volume chamber.

20 The acorn nut **130**, which is inserted into the sample entry port **100**, encloses a
21 sample delivery tube **120** which is utilized to deliver a micro-volume of flowing sample

1 of liquid suspended particles into the inner, centrally enclosed, hollow, micro-volume
2 chamber **30**. The internal volume of the chamber **30** ranges from 2 micro-liters to 8
3 micro-liters. Consequently, the amount of flowing sample of liquid-suspended particles
4 that can be delivered into the chamber will also range between 2 and 8 micro-liters.
5 Once the dynamic light scattering measurements of the liquid-suspended particles have
6 been made, the specimen is removed from the chamber via a tubing bearing acorn nut
7 **130** that has been inserted into the sample exit port **110**.

8 The process of utilizing the inventive micro-volume, single chamber, dynamic,
9 light-scattering sample flow cell **10** comprises the following steps:

10 installing the inventive flow cell **10** on the chassis or base, or housing of a
11 dynamic, light-scattering detector **150**;

12 communicatingly connecting a fiber optic system **140** via an optical detecting
13 fiber **142** to said measuring through-bore **60** of the inventive flow cell **10**;

14 by using the appropriate hardware such as a laser generating module, supplying
15 power and generating a focused laser beam;

16 optionally adjusting the temperature of the laser generating module for the
17 purpose of maintaining the stability of the laser beam intensity during the use of the
18 inventive flow cell **10**;

19 simultaneously focusing the laser beam both through the incident through-bore
20 **40** and the spherical or ball lens **70**, frictionally but removably fixed at the terminating
21 opening of said incident through-bore **40**, adjacent to said inner, centrally enclosed,
22 hollow, micro-volume chamber **30**;

1 via the sample entry port **100**, filling the inner, centrally enclosed, hollow,
2 micro-volume chamber **30**, either manually or via a pump system, with a flowing sample
3 of liquid- suspended particles needing to be measured and characterized;

4 focusing the laser beam through the incident through-bore **40** and through its
5 corresponding spherical or ball lens **70** onto the sample of liquid-suspended particles
6 and out the exiting through-bore **50** and its corresponding spherical or ball lens **80**,
7 adjacent to the inner, centrally enclosed, hollow micro-volume chamber **30**. The
8 molecules or particles within the laser beam's path scatter light from the laser beam.
9 The spherical or ball lens **90**, located at the terminating opening **62** of the measurement
10 through-bore **60**, between the incident through-bore **40** and the exiting through-bore **50**,
11 collimates the scattered light which is then refocused onto the detecting fiber **142**, *i.e.*,
12 the scattered light intensity is picked up by the optical detector via a fiber optic system,
13 and converted to provide data that generates information regarding the physical
14 characteristics and chemical properties of the liquid-suspended particles filling the
15 chamber.

16 Referring more specifically to the drawings, **Figures 11** generally depicts another
17 embodiment of the inventive optical spherical lens light scattering apparatus, *i.e.*, an
18 inventive, single chamber, light-scattering probe at **10**, as used for example in batch
19 mode, laser light-scattering analysis systems. It is designed to achieve all of the objects
20 set forth herein above.

1 The inventive single chamber, light-scattering probe at **10** enables, for the first
2 time in the art, the use of probe that permits the focusing of a laser beam within a very
3 short distance from the lens surface.. This, in turn, allows for the gathering of a
4 tremendous amount of information. that heretofore could not be collected due to the
5 long focusing distances of the laser beam from the lens.

6 The inventive single chamber, laser light-scattering probe **10**, as can be seen
7 from **Figure 11** comprises a body **152**, preferably cylindrical, having an upper end **154** and
8 a lower end **156**; at least one partial through-bore **146** beginning at the outer surface of the
9 upper end **154** of the body and forming an inner, hollow core or chamber **158**; at least one,
10 spherical or ball lens **70** removably and frictionally mounted on said upper end **154** and
11 sealably fixed within said through-bore **158**, such that said spherical or ball lens **70** completely
12 fills and seals the upper end **154** of the hollow core or chamber **156** and and partially forms
13 the upper surface of the upper end **154** of the probe; an incident light source **12** and at least one
14 optical detecting fiber **142** supported and enclosed within said inner, centrally enclosed hollow
15 core or chamber **158** of the probe; and a photodetector **150** connected to said one optical
16 detecting fiber **142**.

17 It is important that the probe be made, at least partially and preferable at its upper
18 end **154** of a material that permits an uncompromisable seal for all of the probe's
19 components, without the use of additional seals, gaskets, windows or other sealing
20 compounds (e.g. epoxy, silicone or similar) and which does away with the need to find
21 a broad range of chemically compatible, broad range temperature, insensitive seals,
22 gaskets or sealing compounds. Furthermore, it has to be compatible with both the

1 solvents in which the specimen particles are suspended, as well as be compatible with
2 the specimen particles themselves. Finally, it must not break down, either physically or
3 chemically, when coming in contact with the solvents and the specimen particles
4 suspended therein, in a wide range of temperatures. One material that has the ability to
5 provide all of the foregoing is Polyetheretherketone (PEEK).

6 In the preferred embodiment, the probe is at least partially made of PEEK. Not
7 only is it compatible with a very wide range of solvent liquids and sample particles, but
8 is also neutral in that it does not leach contaminants into the sample of liquid-suspended
9 particles; even when it is exposed to temperatures ranging from 0 degrees C to 160
10 degrees C. Use of PEEK in the probe allows for a secure seal between the spherical or
11 ball lens and the upper end of the probe, without the use of additional seals, gaskets,
12 windows or other sealing compounds (e.g. epoxy, silicone or similar).

13 The process of utilizing the probe, having the spherical or ball lens optical system,
14 comprises the following steps: communicatingly connecting the at least one optical
15 detecting fiber to said photodetector; supplying power and using the incident light
16 source within the inner chamber of the probe to generate a focused beam of light;
17 focusing the light beam on the spherical or ball lens, frictionally but removably fixed
18 at said lower end of the probe sealing the through-bore, such that the beam penetrates
19 and enters the spherical or ball lens at a zero degree angle relative to the path of said
20 incident light beam; immersing the probe into a sample of liquid suspended particles
21 needing to be measured and characterized; focusing the beam of light through its

1 corresponding spherical or ball lens onto the sample of liquid suspended particles. The
2 molecules or particles within the laser beam's path at the focal point of the spherical or
3 ball lens, and adjacent thereto scatter light. The spherical or ball lens then collimates
4 the scattered light and focuses it back through said spherical or ball lens, onto the
5 detecting fiber, which in turn is sends it to the photodetector which in turn converts to
6 provide data that generates information regarding the physical characteristics and
7 chemical properties of the liquid-suspended particles in the specimen, in which the
8 probe was immersed.

9 As a result of the components of the inventive optical spherical or ball lens light
10 scattering apparatus and its various embodiments disclosed herein above and the way
11 they cooperatingly function, it is clear that they achieve all of the objectives set forth
12 herein above including: (i) minimizing, if not totally eliminating, those errors in the
13 results of light scattering technology applications, that arise from the background effects
14 created or contributed by the sample cell; (ii) minimizing, if not totally eliminating,
15 those errors in the results of light scattering technology applications, that arise from the
16 background effects created or contributed by the suspension fluid; (iii) minimizing, if
17 not totally eliminating, any and all refractive index effects on the light beam generated
18 by the incident light source, as the light beam travels and transitions from one medium
19 and into another; (iv) generating a light beam that will, more likely than not, enter the
20 suspension fluid at a zero degree angle relative to the incident path or axis of said light
21 beam; (v) minimizing, if not completely eliminating, the light scattering masking of
22 particles' light scattering results, effectuated by the impurities found in the sample cell

1 and the impurities found in the suspension fluid; (vi) minimizing, if not completely
2 eliminating, the light scattering masking of particles' light scattering results, effectuated
3 by the reflection or bouncing back into the suspension fluid, of light not successfully
4 exiting the sample cell; and (vii) minimizing, if not completely eliminating, the light
5 scattering masking of particles' light scattering results, effectuated by air bubbles
6 trapped in the sample cell or in the suspension fluid.

7 While particular embodiments of the invention have been illustrated and
8 described in detail herein, they are provided by way of illustration only and should not
9 be construed to limit the invention. Since certain changes may be made without
10 departing from the scope of the present invention, it is intended that all matter contained
11 in the above description, or shown in the accompanying drawings be interpreted as
12 illustrative and not in a literal sense. Practitioners of the art will realize that the
13 sequence of steps and the embodiments depicted in the figures can be altered without
14 departing from the scope of the present invention and that the illustrations contained
15 herein are singular examples of a multitude of possible depictions of the present
16 invention.

17 We claim:

- 1 1. An apparatus for light scattering analysis of particles dispersed in a suspension
2 fluid comprising at least one ball lens in direct contact with the suspension fluid.
- 3 2. The apparatus of **claim 1**, wherein said one ball lens is interposed between an
4 incident light source and a photodetector.
- 5 3. The apparatus of **claim 2**, wherein said incident light source comprises a laser.
- 6 4. The apparatus of **claim 2**, wherein said photodetector is selected from the group of
7 photodetectors consisting of photomultipliers, avalanche photodiodes, and charge-
8 coupled devices.
- 9 5. The apparatus of **claim 3**, wherein said photodetector is selected from the group of
10 photodetectors consisting of photomultipliers and avalanche photodiodes.
- 11 6. A method for light scattering analysis of particles dispersed in a suspension fluid
12 comprising the step of focusing an incident beam of energy through a ball lens
13 contacting said suspension fluid and into said suspension fluid.
- 14 7. A method for light scattering analysis of particles dispersed in a suspension fluid
15 comprising the steps of: (i) using an incident light source to generate a beam of energy
16 having a certain wavelength and frequency; (ii) focusing said beam of energy through
17 a ball lens; (iii) placing a specimen of particles, dispersed in a suspension fluid, at the
18 focal point of said ball lens so that said ball lens is in direct contact with the suspension
19 fluid and the focused beam of energy hits the suspended particles at exactly the focal

1 point of the ball lens to cause the suspended particles to scatter the beam of energy; and
2 (iv) directing that scattered light produced by the suspended particles through a ball
3 lens onto a photodetector.

4 8. A sample cell for use in light scattering analysis of particles dispersed in a
5 suspension fluid comprising:

6 a cell block having an upper end and a lower end, and an inner, centrally
7 enclosed chamber;

8 at least three through-bores placed on the same horizontal plane, within
9 said cell block,

10 one through-bore being an incident through-bore, another being an
11 exit through-bore, and the third one being a measuring through-
12 bore,

13 said incident through-bore and said exit through-bore being
14 juxtaposed end to end, but spaced apart the full width of the inner,
15 centrally enclosed chamber, so that their terminating openings
16 into said inner, centrally enclosed chamber, are positioned exactly
17 across from and are co-axial to each other, at opposite walls of the
18 inner, centrally enclosed chamber, whereby when a laser beam is
19 focused through said incident through-bore, it enters into said
20 inner, centrally enclosed chamber, and emerges out said exit
21 through-bore, and

22 Said measuring through-bore is interposed between said end to
23 end, juxtaposed incident through-bore and exit through-bore, such

1 that said measuring through-bore's longitudinal axis is
2 perpendicular to the co-aligned longitudinal axes of the two
3 juxtaposed through-bores, and its terminating opening into said
4 inner, centrally enclosed chamber is approximately equidistant
5 from the terminating openings of the juxtaposed through-bores;
6 at least three ball lenses, removably and frictionally fixed within each of
7 said through-bores such that they partially form and seal the wall of the
8 inner, centrally enclosed chamber; and
9 means for introducing into said inner, centrally enclosed chamber a
10 specimen of said particles dispersed in said suspension fluid, as well as
11 means for removing said specimen therefrom.

12

13 9. The sample cell of **claim 8**, wherein said ball lenses are selected from the group
14 of ball lenses consisting of ruby ball lenses, sapphire ball lenses and glass lenses.

15 10. The sample cell of **claim 8**, wherein said ball lenses have a diameter range from
16 0.5 mm to 10 mm.

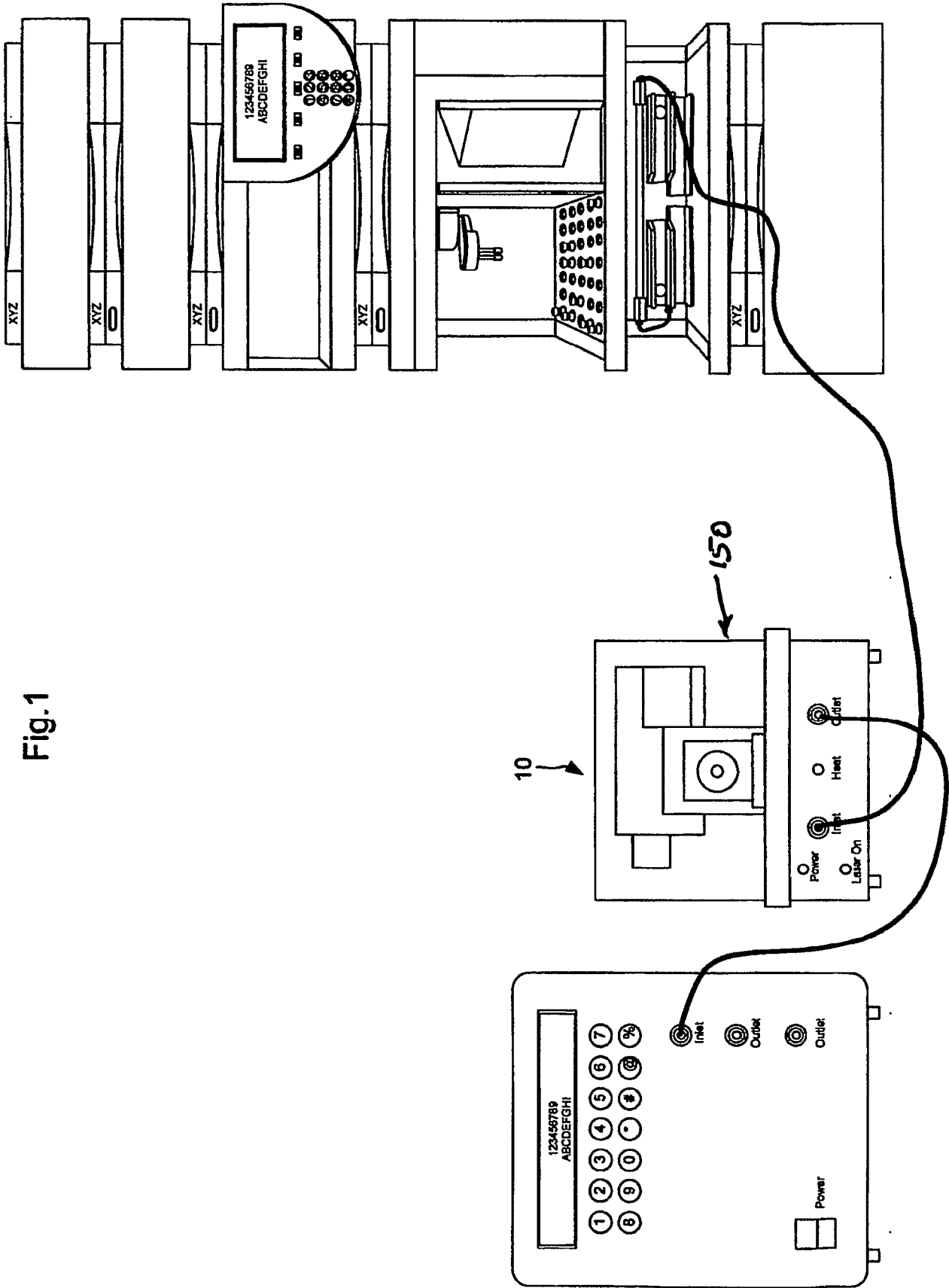
17 11. The sample cell of **claim 8**, wherein said cell block consists of PEEK.

18 12. The sample cell of **claim 9**, wherein said cell block consists of PEEK.

19 13. The sample cell of **claim 10**, wherein said cell block consists of PEEK.

- 1 14. A method for light scattering analysis of particles dispersed in a suspension fluid
2 comprising the steps of:
- 3 equipping a sample cell with a cell block having an inner, centrally
4 enclosed chamber, at least three through-bores placed on the same
5 horizontal plane, within said cell block, one through-bore being an
6 incident through-bore, another being an exit through-bore, and the third
7 one being a measuring through-bore, said incident through-bore and said
8 exit through-bore being juxtaposed end to end, but spaced apart the full
9 width of the inner, centrally enclosed chamber, so that their terminating
10 openings into said inner, centrally enclosed chamber, are positioned
11 exactly across from and are co-axial to each other, at opposite walls of the
12 inner, centrally enclosed chamber, whereby when a laser beam is focused
13 through said incident through-bore, it enters into said inner, centrally
14 enclosed chamber, and emerges out said exit through-bore, and said
15 measuring through-bore is interposed between said end to end,
16 juxtaposed incident through-bore and exit through-bore, such that said
17 measuring through-bore's longitudinal axis is perpendicular to the co-
18 aligned longitudinal axes of the two juxtaposed through-bores, and its
19 terminating opening into said inner, centrally enclosed chamber is
20 approximately equidistant from the terminating openings of the
21 juxtaposed through-bores; at least three ball lenses, removably and
22 frictionally fixed within each of said through-bores such that they partially

- 1 form and seal the wall of the inner, centrally enclosed chamber and come in
2 contact with said suspension fluid, and means for introducing into and
3 removing from said an inner, centrally enclosed chamber a sample a
4 specimen of particles dispersed in said suspension fluid;
5 supplying power and generating a focused laser beam;
6 focusing the laser beam both through the incident through-bore and its
7 corresponding ball lens;
8 filling said inner, centrally enclosed, chamber, either manually or via a
9 pump system, with a specimen of fluid- suspended particles needing to
10 be measured and characterized, such that said incident ball lens, said exit
11 ball lens and said measuring ball lens are in direct contact with said
12 specimen;
13 focusing the laser beam through the incident through-bore and through its
14 corresponding ball lens onto the specimen of fluid-suspended particles
15 and out the exiting through-bore and its corresponding spherical or ball
16 lens adjacent to the inner, centrally enclosed, hollow micro-volume
17 chamber to allow the particles within the laser beam's path to scatter the
18 light from the laser beam; and
19 collimating the scattered light for transmittal to a photodetector.
- 20 15. A probe for use in light scattering analysis of particles dispersed in a suspension
21 fluid comprising a ball lens that is in direct contact with said suspension fluid.



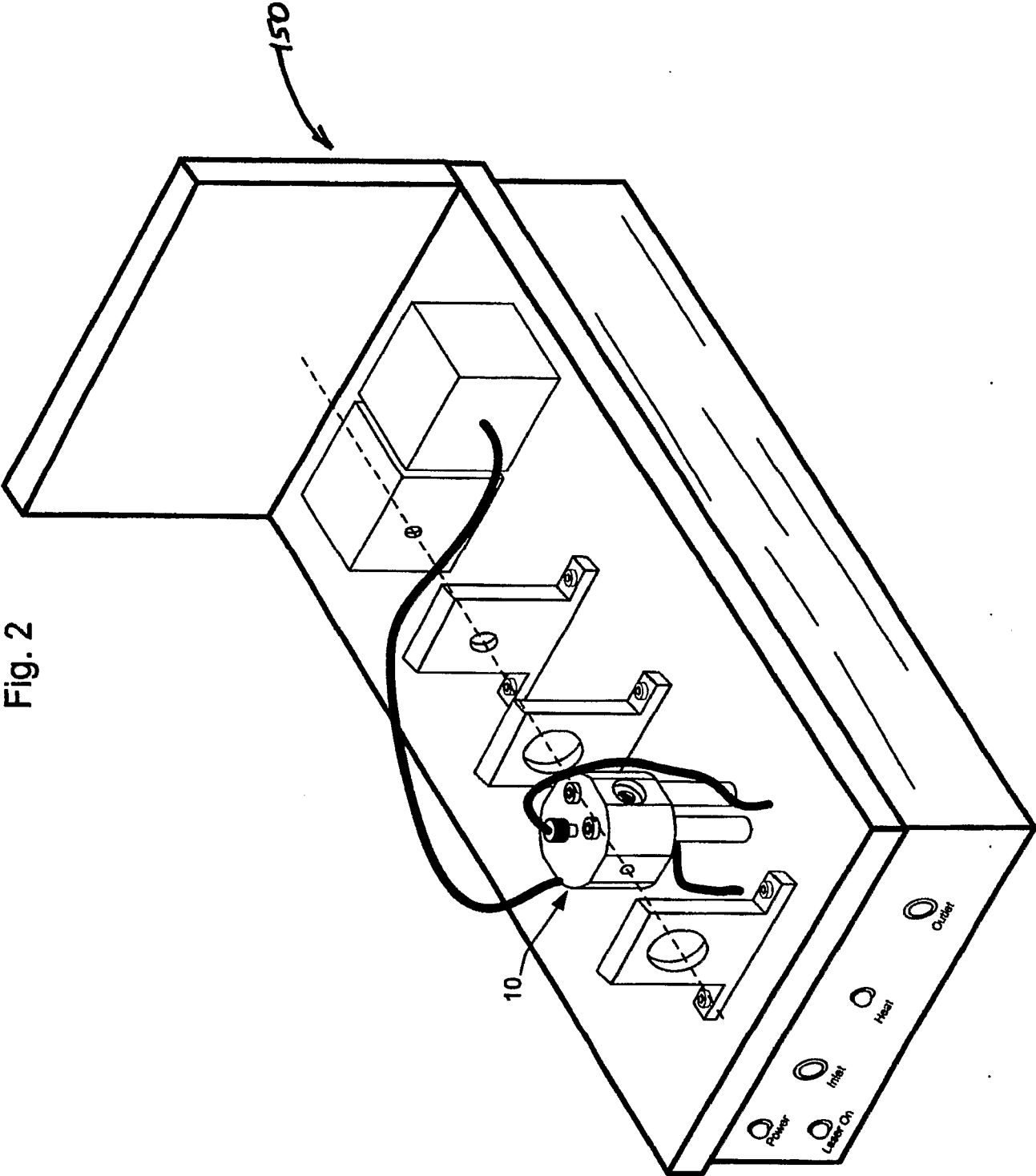


Fig. 2

Fig. 3

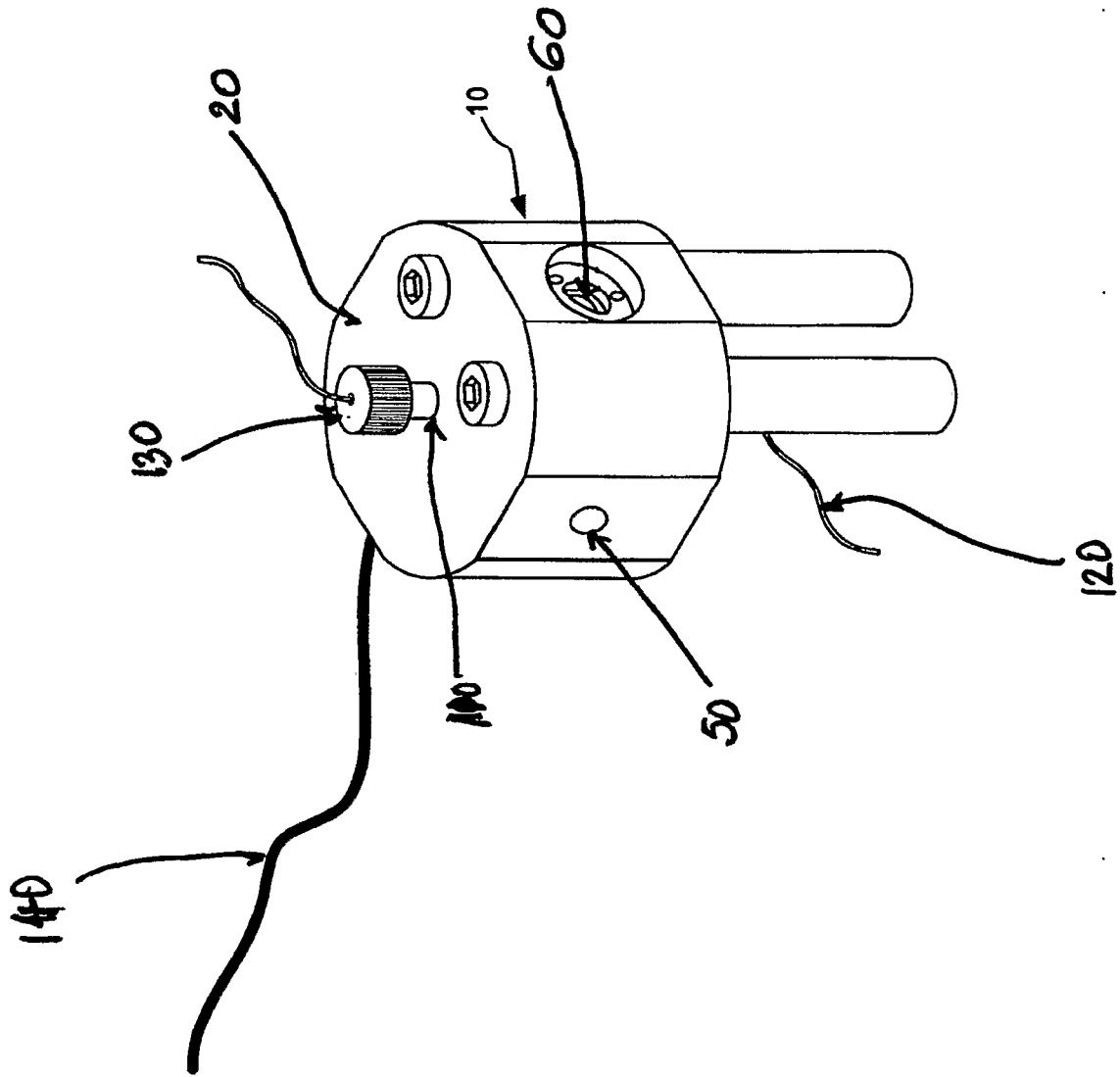


Fig. 4

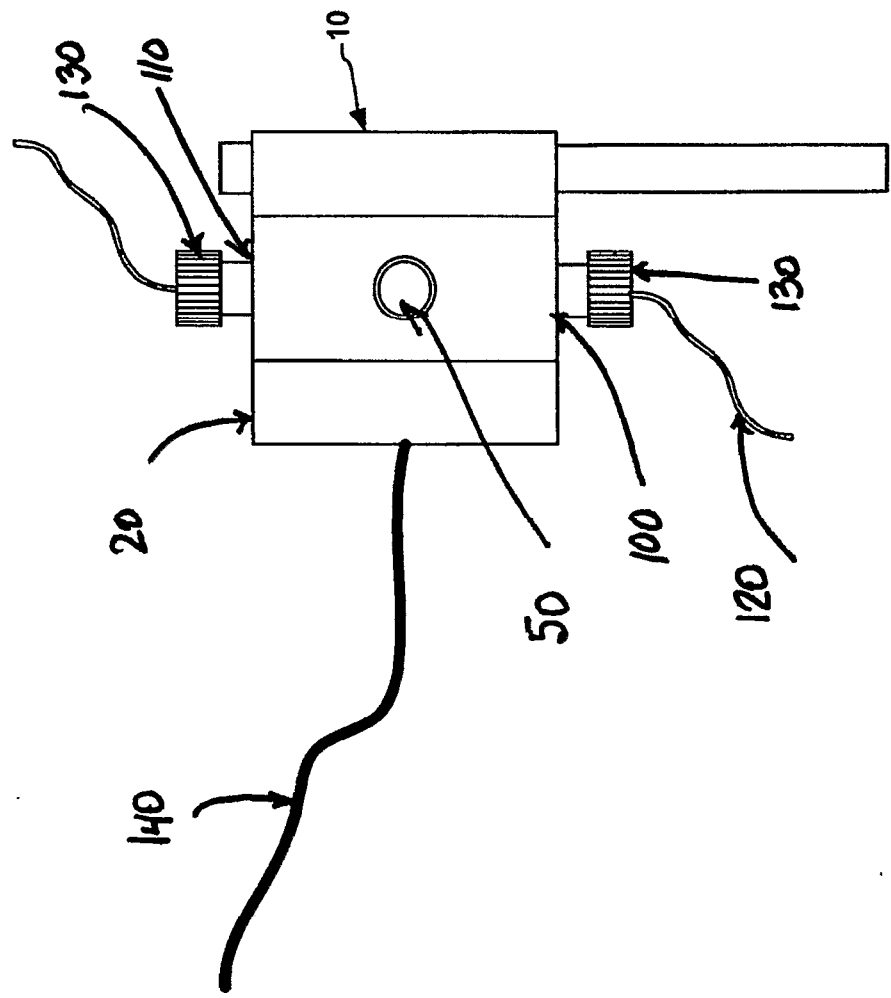


Fig. 5

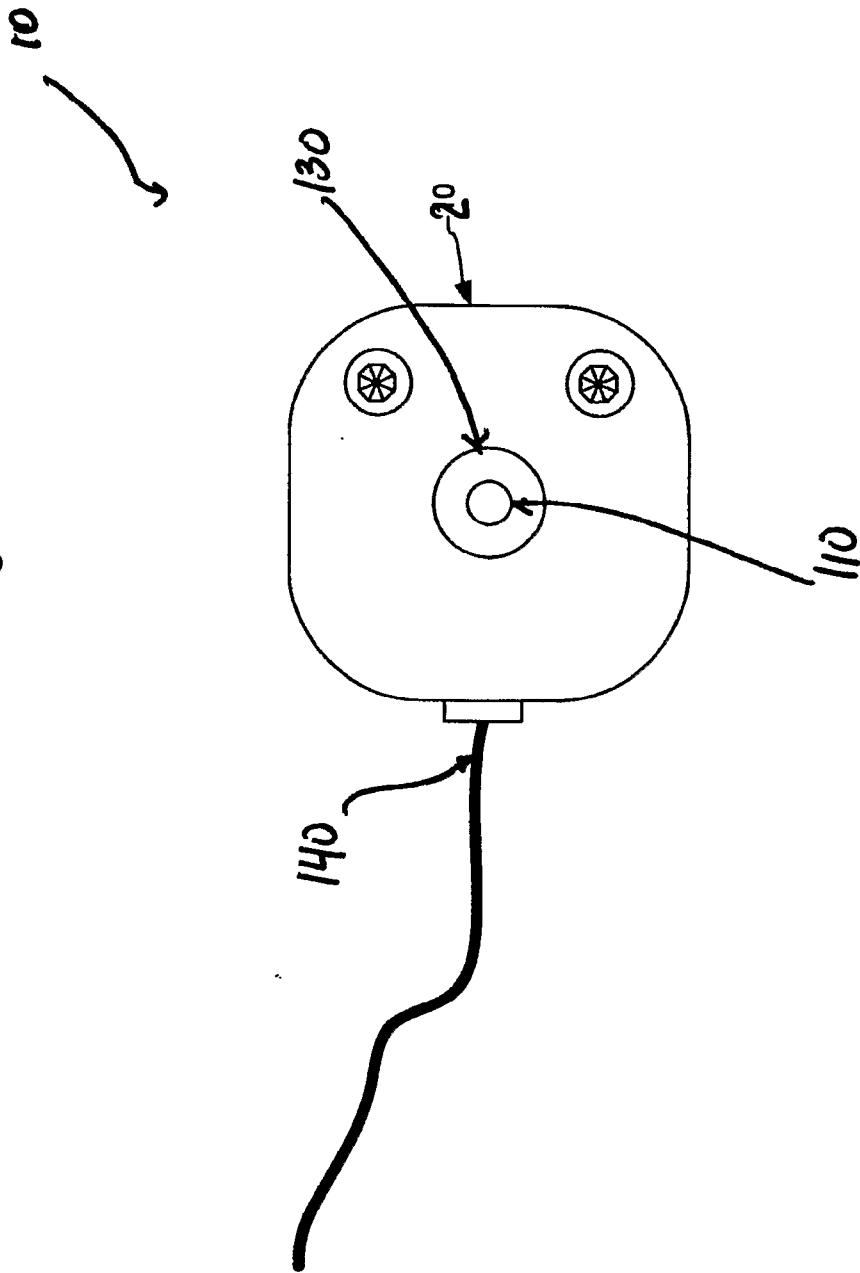


Fig. 6

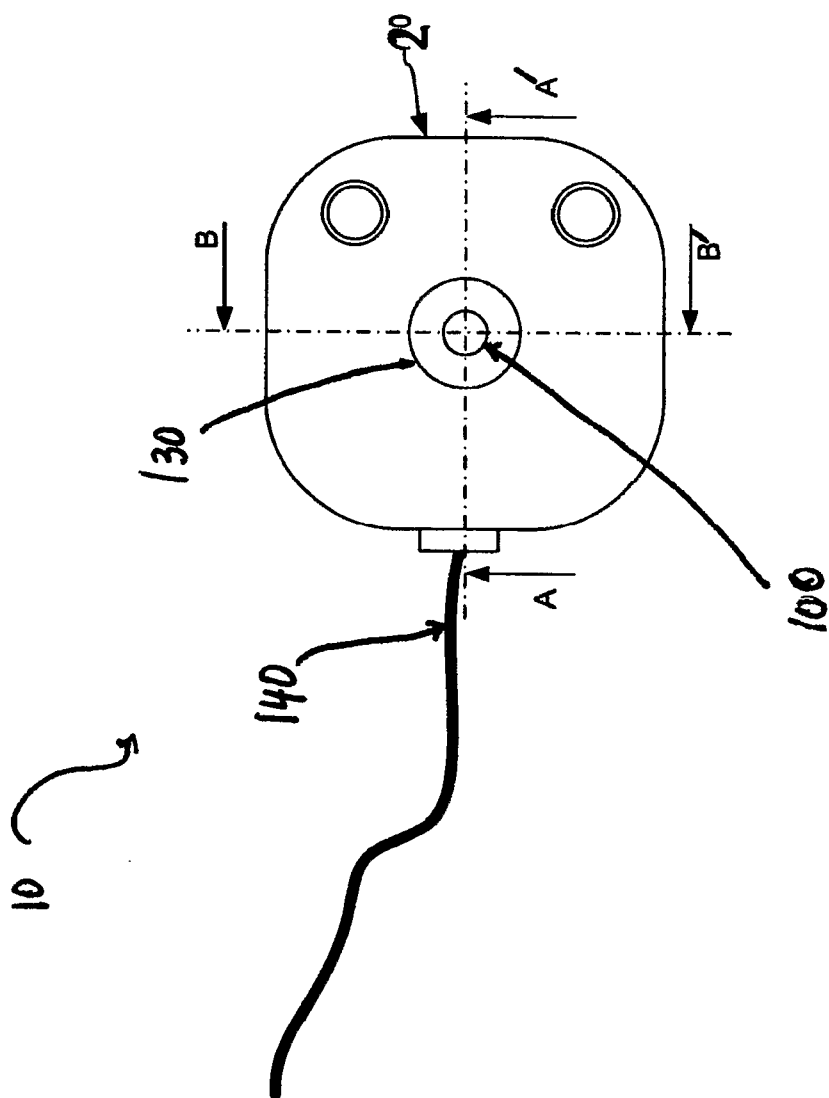


Fig. 7

SECTION A-A

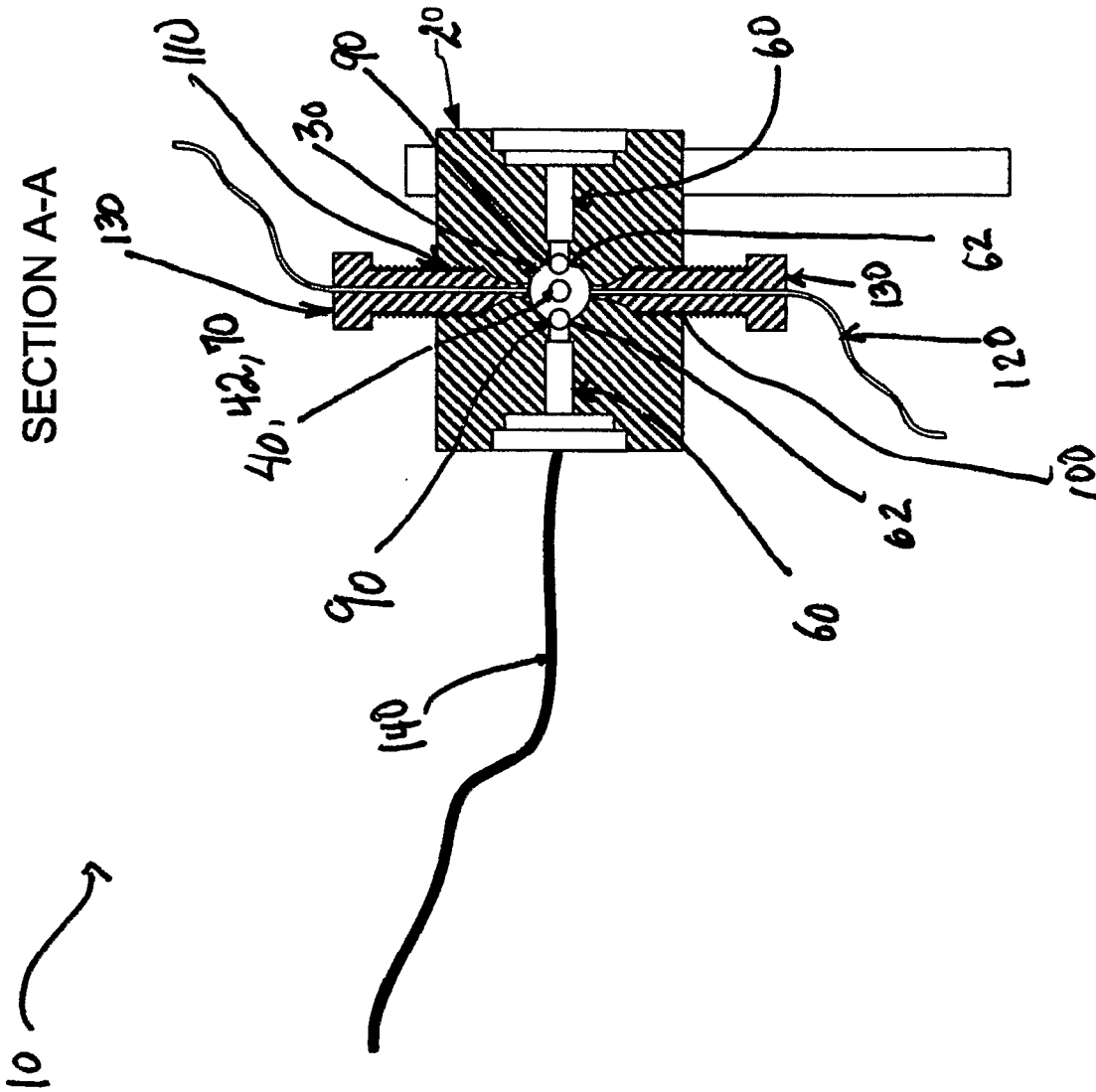


Fig. 8

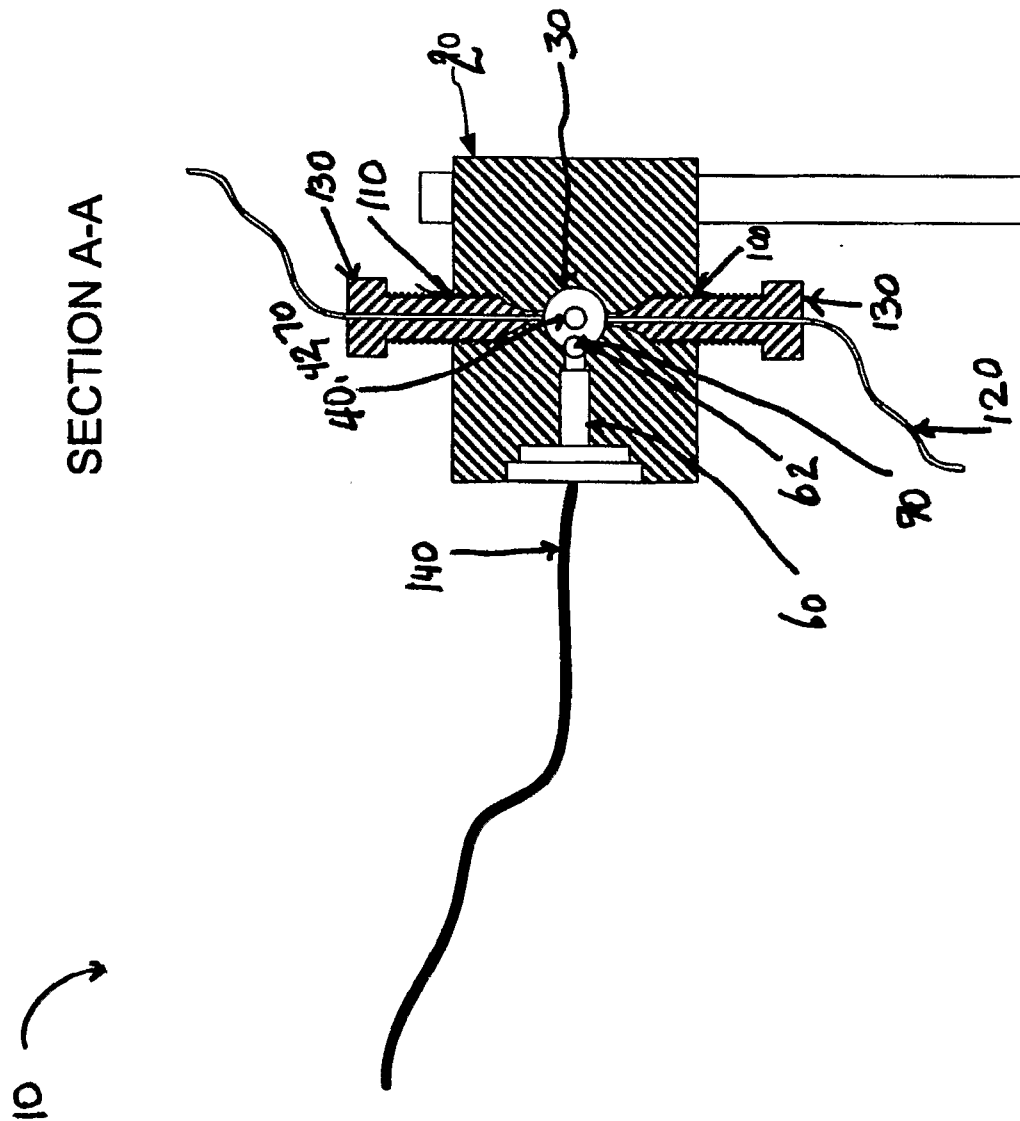


Fig. 9

SECTION B-B

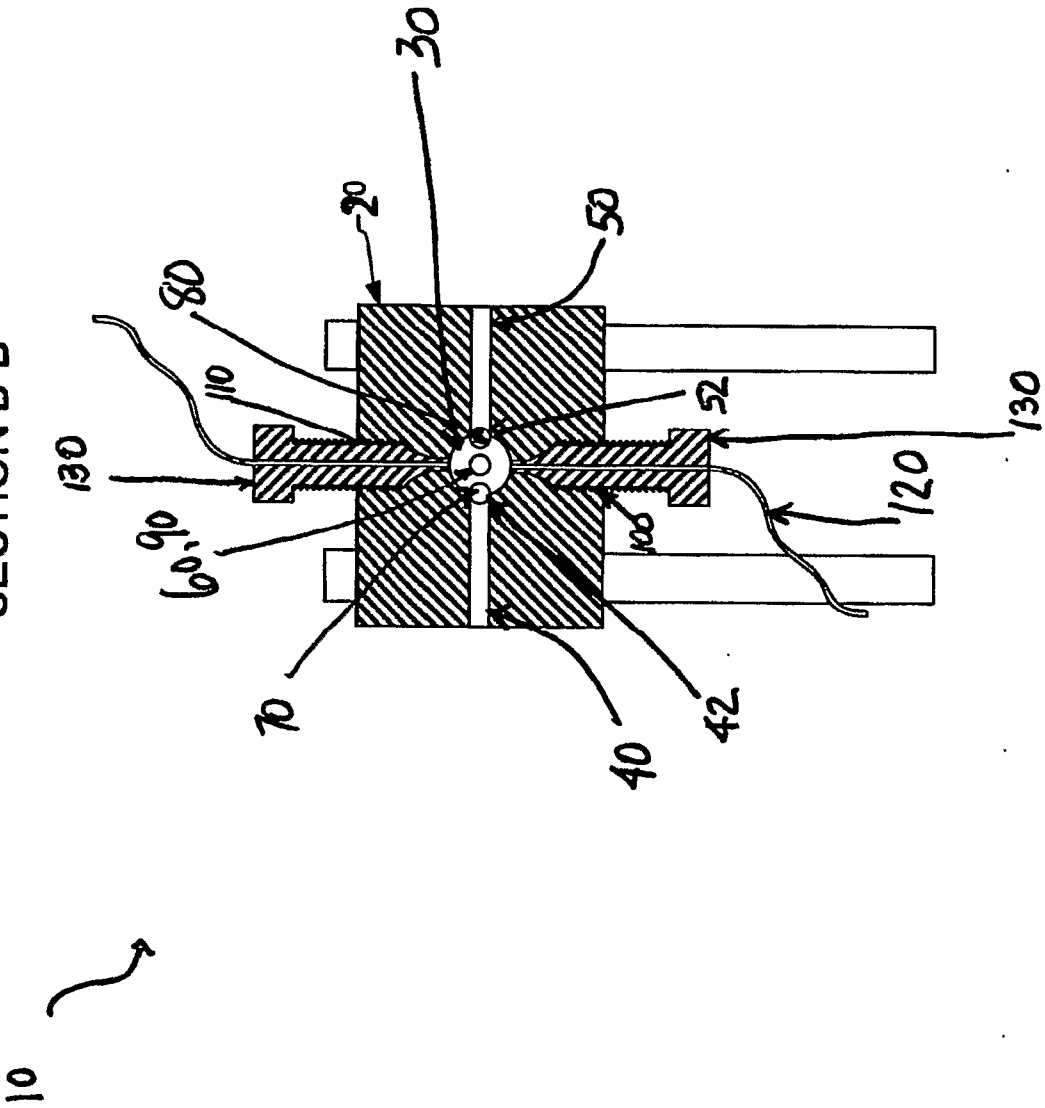


Fig. 10

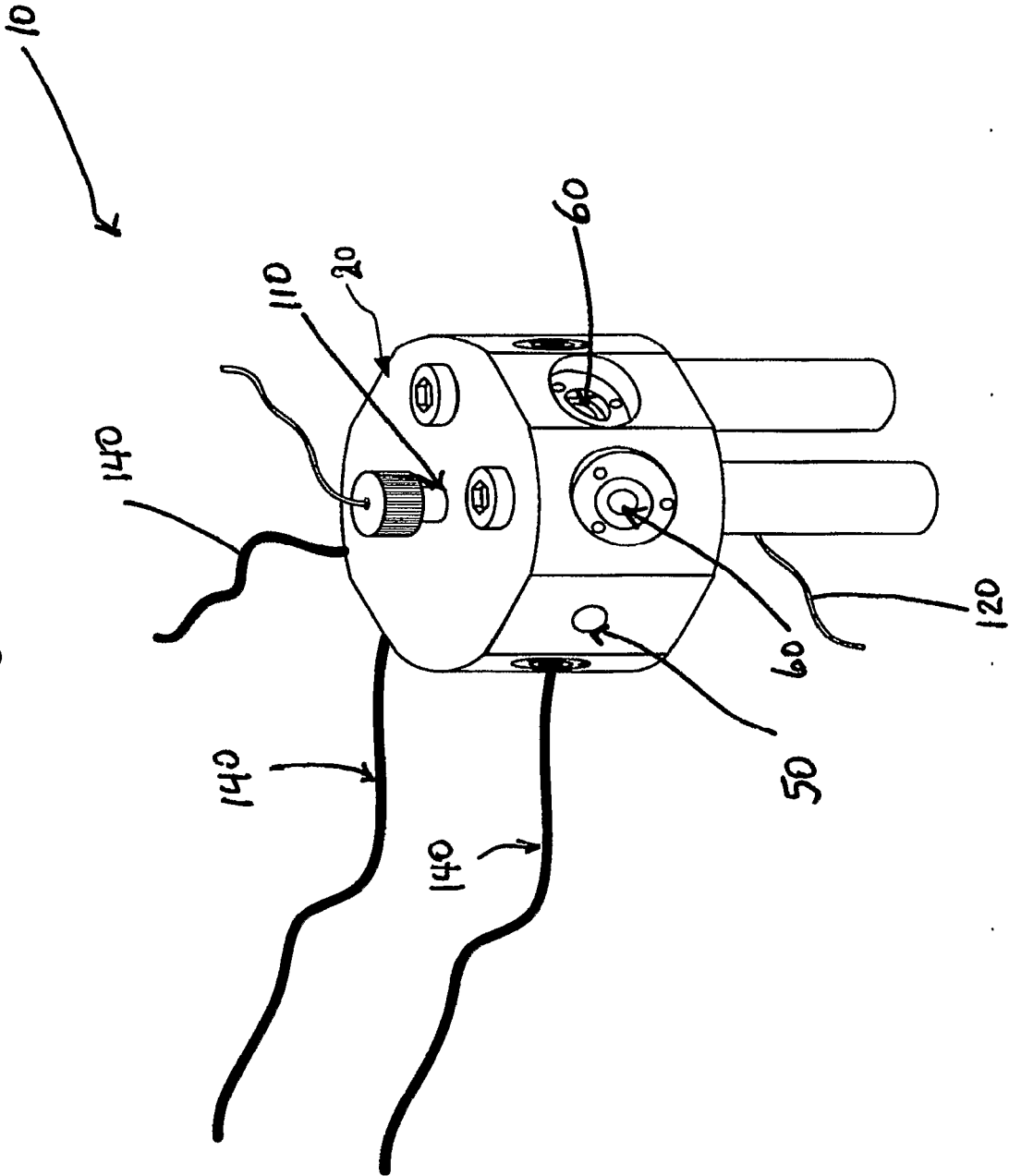
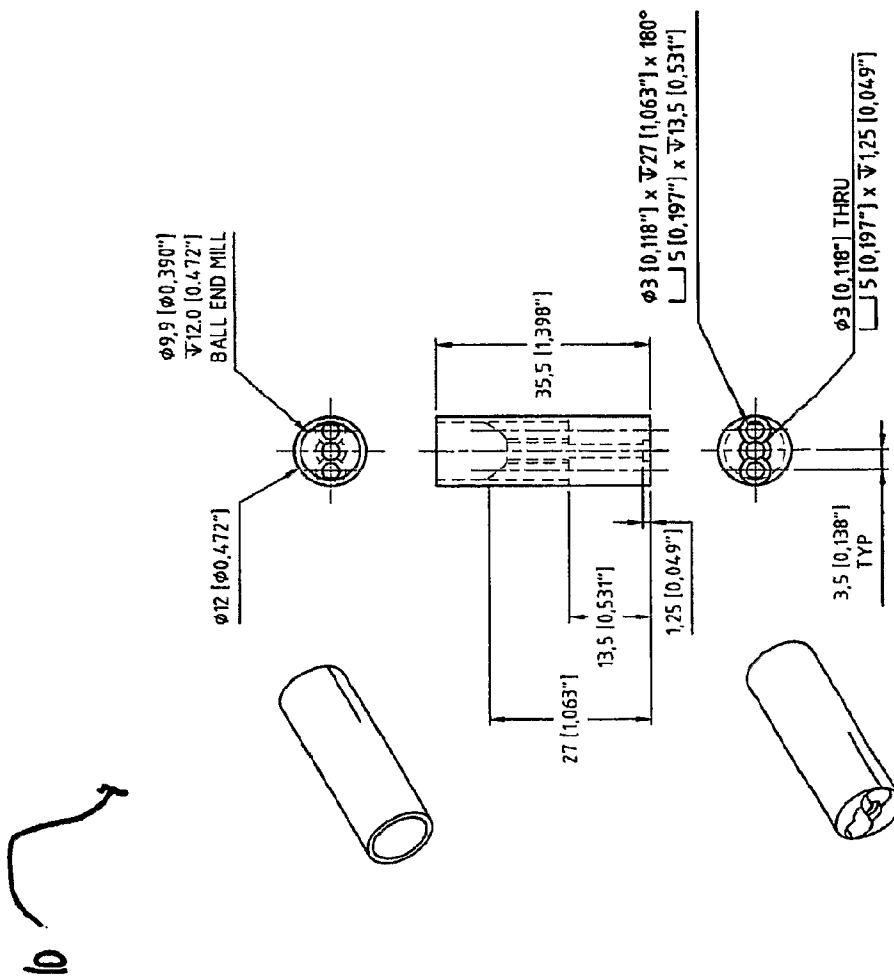
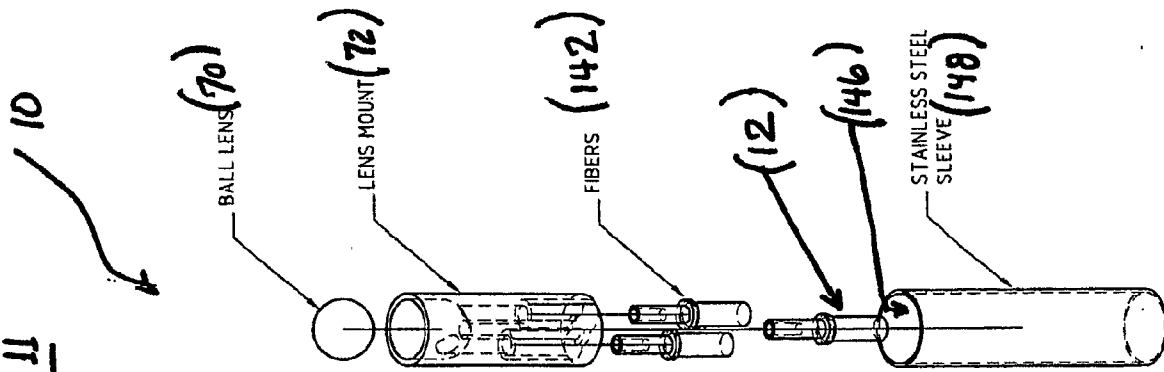
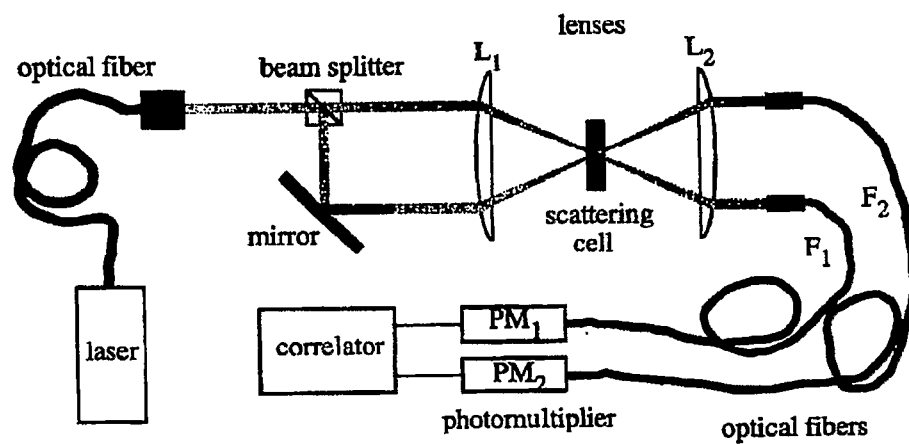
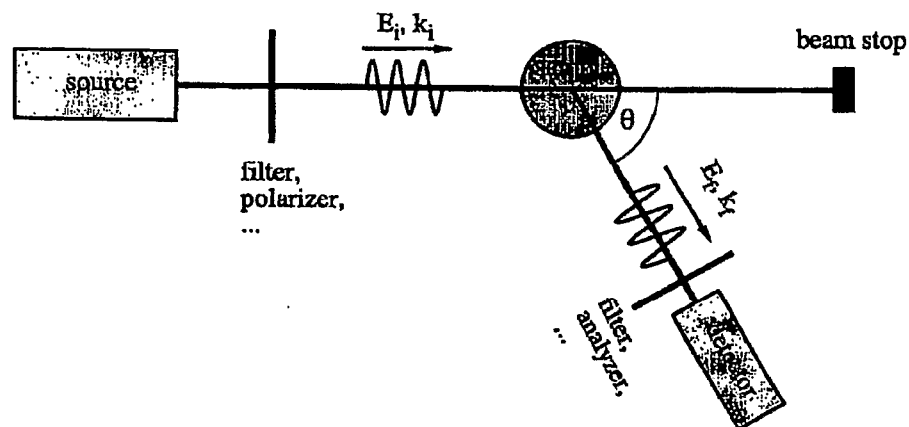


Figure 11



FOOELS PROBE LAYOUT
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Figure 12(Prior Art)Figure 13 (Prior Art)

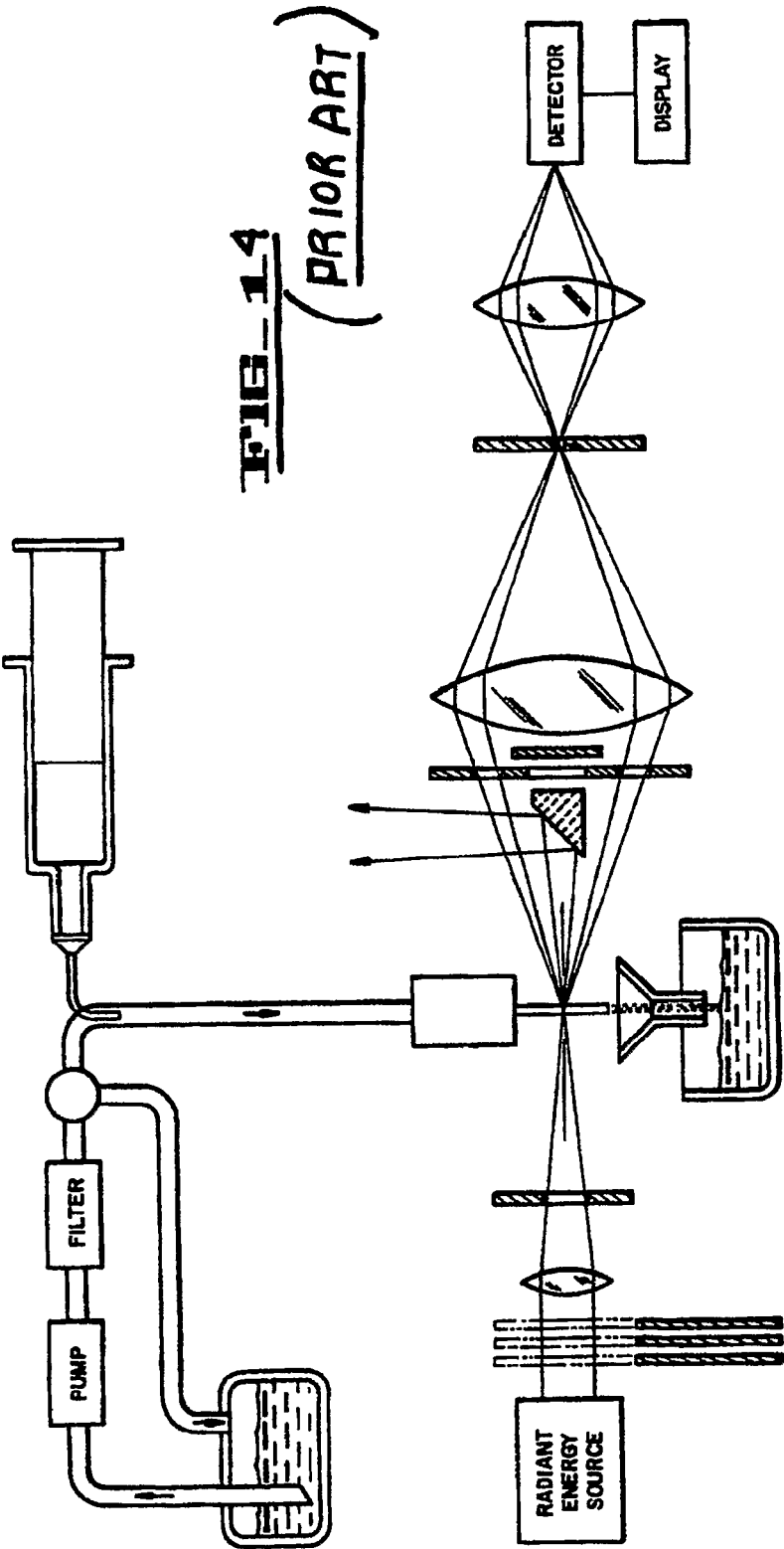


FIG-14
(PRIOR ART)

