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**GB 2346444 A** **GB 2340936 A**  
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(58) Field of Search:  
INT CL **G01N**  
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(54) Title of the Invention: **Apparatus and method for measuring particle size distribution by light scattering**  
Abstract Title: **Measuring particle size distribution by light scattering**

(57) Apparatus 100 for measuring particle size distribution by light scattering comprises a blue LED 102 and a red laser 104. Light from the LED and laser is passed or reflected by dichroic element 116 onto a common path through a sample cell 122. Light scattered from the sample cell is detected by one or more detectors (112B-H). Light transmitted by the sample cell is detected by detectors 112A, 112J. Output signals from the detectors are passed to a processor 114 which calculates particle size distribution. A small percentage of light from LED 102 is reflected by the dichroic element to detector 110. Similarly, a small percentage of light from the laser passes through the dichroic element to the detector. Output signals from the detector are fed back to control units 106, 108 to stabilise the output power of the LED and laser.

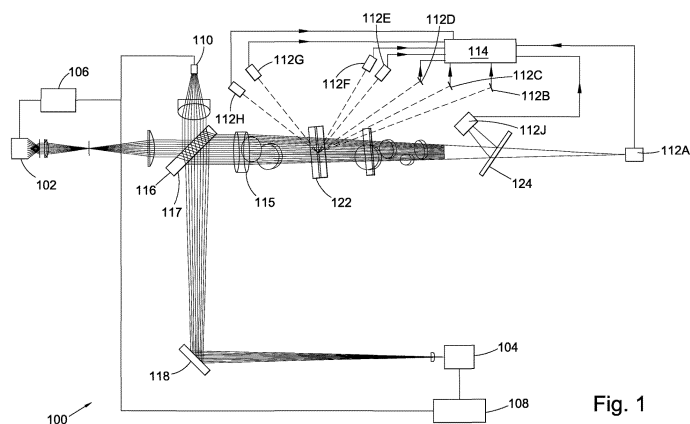


Fig. 1

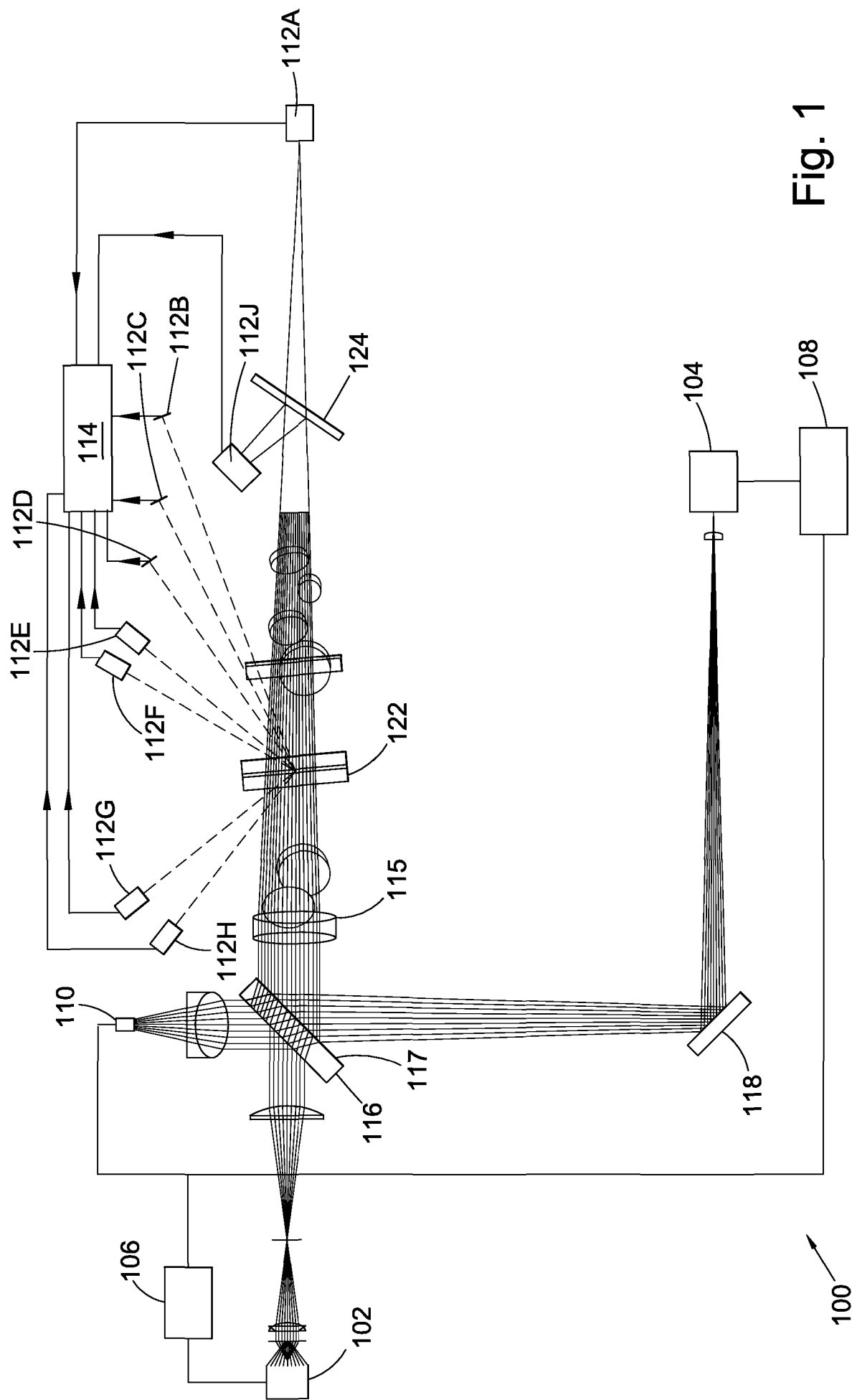


Fig. 1

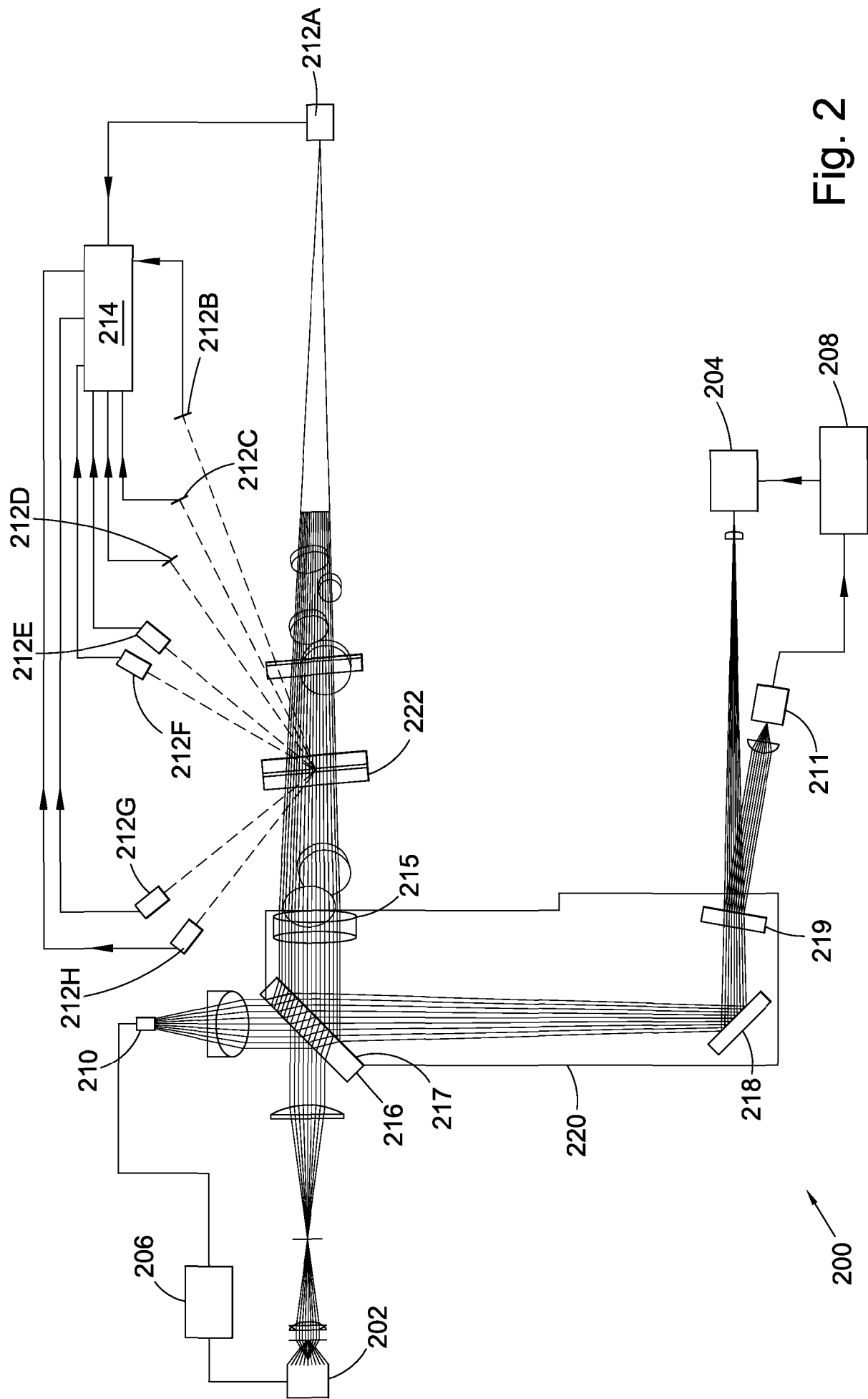


Fig. 2

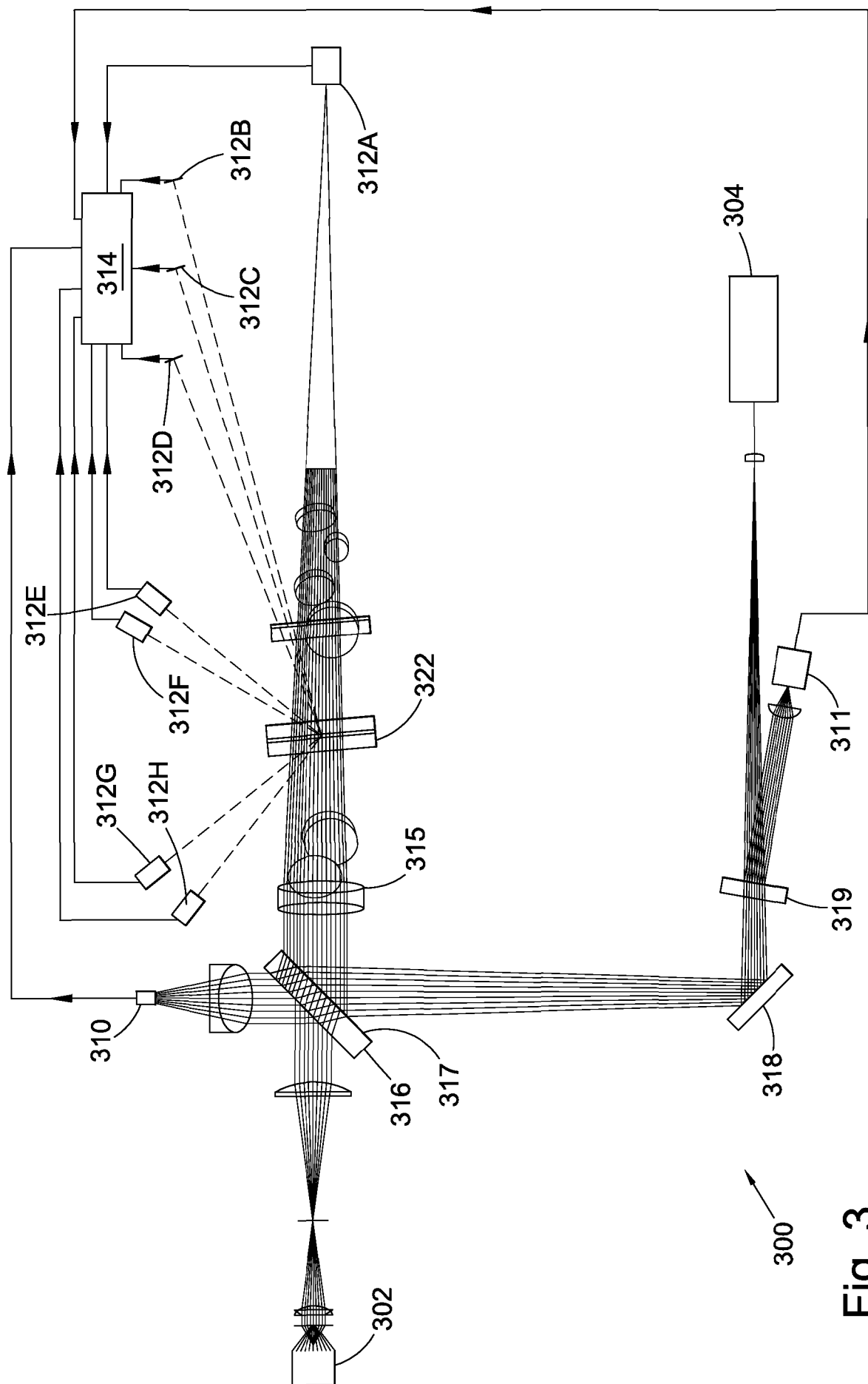


Fig. 3

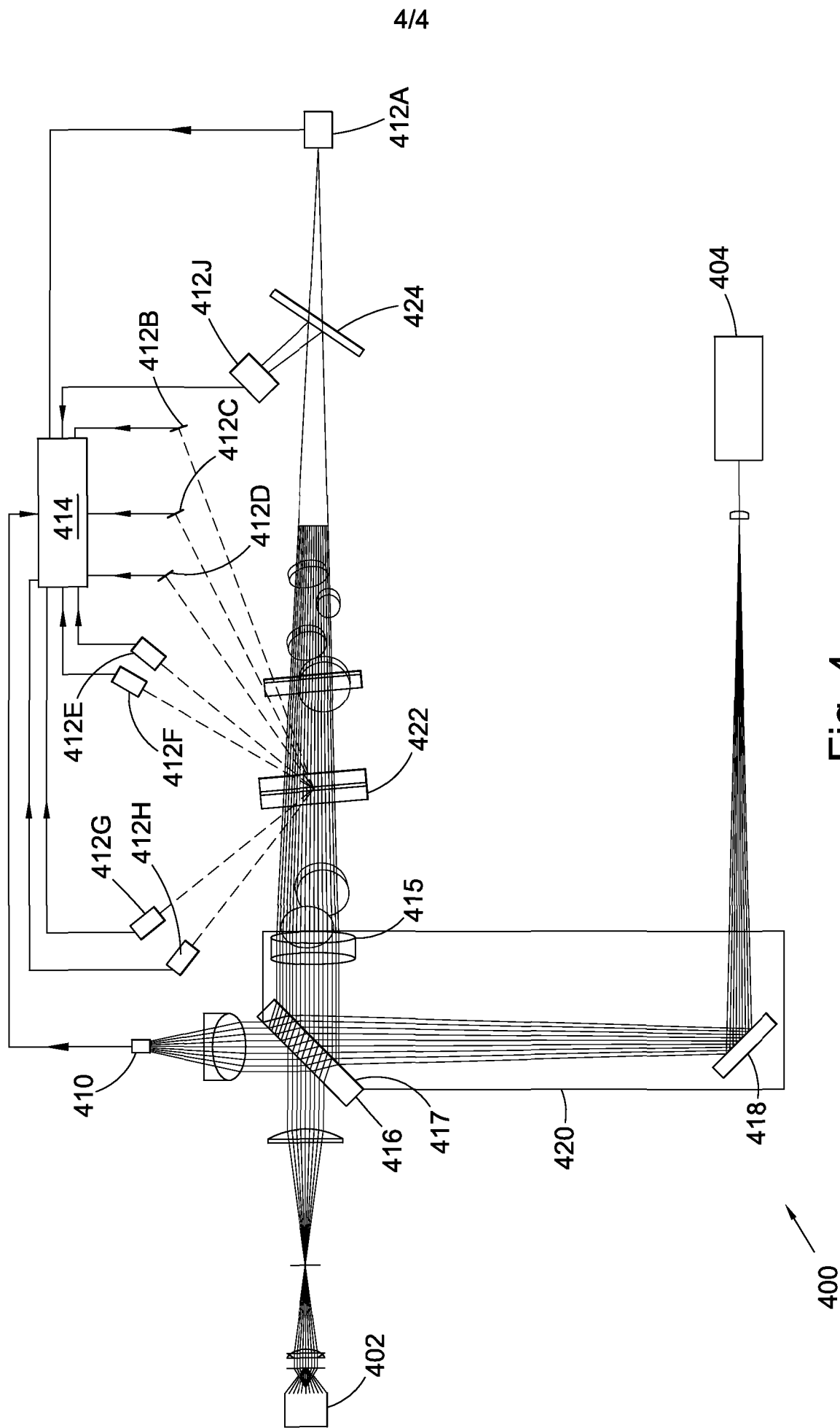


Fig. 4

## **APPARATUS AND METHOD FOR MEASURING PARTICLE SIZE DISTRIBUTION BY LIGHT SCATTERING**

The invention relates to apparatus and methods for measuring particle size distribution  
5 by light scattering.

Methods and apparatus for measuring particle size distribution for a sample by  
monitoring light scattered by the sample are known. In some such techniques, light  
scattered at two different wavelengths is monitored to extend the range of particles  
10 sizes that can be measured and/or to improve resolution. For example, in the method  
and apparatus described in European Patent 0 992 785 light from a blue laser diode or  
blue LED is used to make scattering measurements in addition to taking scattering  
measurements using a laser having an output of longer wavelength, for example a  
helium-neon laser, to enhance detection and resolution for sub-micron particle sizes.

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In order to obtain scattering measurements at two different wavelengths, the optical  
output beams from two suitable light sources are generally multiplexed onto a  
common path through a sample containing particles, or at least some arrangement is  
provided so that each beam may impinge on the sample at respective time along the  
20 same path. A beam-splitter or dichroic mirror can be used to achieve this function.  
Means are normally provided for detecting the beams prior to interaction with the  
sample so that fluctuations in the output powers of the light sources can be corrected  
by adjusting the light sources, or so that such fluctuations can be taken into account  
when calculating particle size distribution.

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A first aspect of the present invention provides apparatus for measuring particle size  
distribution for a sample by light scattering, the apparatus comprising light-generating  
means for generating first and second beams of light having first and second  
wavelengths respectively, a dichroic element arranged to direct most of the power of  
30 the first and second beams onto a common path by transmission and reflection of the  
first and second beams respectively at the dichroic element, and wherein the apparatus  
further comprises a first detector arranged to detect a portion of the first beam  
reflected by the dichroic element.

In apparatus of the invention, the power of the first beam may be monitored by detecting the portion of the first beam which is reflected by the dichroic mirror to the first detector. This obviates the need for more complex arrangements for monitoring the power of the first beam as incident on the sample. Apparatus of the invention is therefore simpler and cheaper than apparatus of the prior art having the same functionality, and utilises light energy that would otherwise be wasted. By reducing the number of optical elements compared to apparatus of the prior art, stray reflections and unwanted scattering within the apparatus is reduced, thus improving the signal-to-noise ratio for light scattered by the sample and subsequently detected within the apparatus. This is particularly important in the detection of light backscattered by the sample.

The apparatus may comprise a control system for controlling the power of the first beam, the control system being arranged to receive an output signal from the first detector and to adjust the power of the first beam in response thereto. For example if the first beam is provided by a laser, the control system may be arranged to increase the pumping rate of the laser if power detected by the first detector decreases, and to reduce the pumping rate if the power detected by the first detector increases, in order to stabilise the power of the first beam.

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The apparatus may comprise a computation unit arranged to receive a signal from the first detector and to calculate particle size distribution of the sample based in part on this signal. In this case the computation unit is arranged to take into account fluctuations in the power of the first beam in calculating the particle size distribution for particles in the sample.

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The first detector may be arranged to detect a portion of the second beam transmitted by the dichroic element, in addition to being arranged to detect a portion of the first beam reflected by the dichroic element. This allows the powers of both the first and second beams to be monitored and used either to stabilise their respective powers or to be taken into account to determine particle size distribution for the sample. Alternatively, in order to additionally monitor the power of the second beam, the apparatus may further comprise a second detector and an optical element arranged to transmit substantially all the power of the second beam to the dichroic element and to reflect a portion of the power of the second beam to the second detector.

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The first wavelength may be shorter than the second wavelength, for example the first beam may be generated by a blue laser diode or blue LED, and the second beam may be generated by a 633 nm helium-neon laser or a red LED.

- 5 The dichroic element may be glass element having substantially plane parallel sides, one of which carries a dichroic coating. Preferably, the rate of change of reflectivity of the dielectric coating as a function of wavelength at the first wavelength is substantially zero so that the performance of the dichroic element is insensitive to variations in the first wavelength that may occur when the light-generating means is first switched on. For a typical standard dichroic coating, the transmission for red light is around 0.2%, however more preferably the transmission of the dichroic coating at the second wavelength is between 2% and 10%.

15 The dichroic element may be comprised in a dust-free housing together with one or more other optical elements for delivery of light to a sample within the apparatus. This reduces unwanted scattering between the light source and the sample.

A second aspect of the invention provides a method of measuring particle size distribution for a sample comprising the steps of:

- 20 (i) generating first and second beams of light having first and second wavelengths respectively; and  
(ii) using a dichroic element to direct most of the power of the first and second beams onto a common path by transmission and reflection of the first and second beams respectively at the dichroic element;  
25 wherein the method comprises the step of using a first detector to detect a portion of the first beam reflected by the dichroic element and generate a corresponding output signal.

Another aspect of the invention provides apparatus for measuring particle-size distribution of a sample by light-scattering, the apparatus comprising light-generating means for generating first and second beams of light having first and second wavelengths respectively, the first wavelength being shorter than the second wavelength, means for directing respective portions of the two beams along a common path to a converging optic arranged to provide converging light at the first and second wavelengths to a sample cell, and a focal plane detector arranged to detect light of the

30  
35



second wavelength transmitted by the sample cell, and wherein the apparatus further comprises an optical component disposed between the sample cell and the focal plane detector and arranged to reflect light of the first wavelength to an optical detector.

5    Embodiments of the invention are described below with reference to the accompanying drawings in which Figures 1 to 4 show respective apparatus for measuring particle size distribution.

Referring to Figure 1, apparatus 100 for measuring particle size distribution comprises  
10    a blue LED 102, a 633 nm helium neon (HeNe) laser 104, control units 106, 108, a dichroic element 116 having a dichroic coating 117 on one side thereof, a sample cell 122 containing a sample of particles the size distribution of which is to be measured, an optical detector 110, a detection arrangement having detectors 112A-J and a computation unit 114.

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The apparatus 100 is typically operated using the blue LED 102 and HeNe laser 104 separately to obtain light scattering measurements from which the particle size distribution for the sample in the sample cell 122 may be deduced by the computation unit 114. Blue light from the blue LED 102 is largely passed by the dichroic element  
20    116 and passes to the sample cell 122 via a focussing optic 115 where it is scattered and subsequently detected by one or more of detectors 112B-H. A few percent of the light from the blue LED is reflected by the dichroic element 116 and passes to the detector 110. Red light output by the HeNe laser 104 is reflected by a mirror 118 and is incident on the dichroic element 116. A few percent of the red light passes through  
25    the dichroic element 116 to the detector 110; the remainder is reflected by the dichroic element 116 towards the sample cell 122 where it is scattered and detected by one or more of the detectors 112B-H. Blue light from the LED 102 and red light from the HeNe laser 104 thus follow a common path from the dichroic element 116 to the sample cell 122. Signals from the individual detectors 112B-H of the detection  
30    arrangement (resulting from the detection of scattered light) are passed to computation unit 114 arranged to calculate particle size distribution for particle in the sample cell 122 in response thereto. Blue light transmitted by the sample cell 122 is detected by detector 112J. Red light transmitted by the sample cell 122 is reflected by a reflective element 124 and detected by a detector 112A. Output signals from the detectors

112A, 112J are also passed to computation unit 114 and used in the calculation of particle size distribution for sample in the sample cell 122.

Output from the detector 110 is passed to control units 106 and 108 which operate to  
5 stabilise the output powers of the LED 102 and HeNe laser 104. (In alternative  
embodiment, only the output power of the LED 102 is controlled.) If the power  
detected by the detector 110 decreases, the relevant control unit 106 or 108 operates to  
increase the output power of the LED 102 or laser 104. Similarly if the power  
detected by the detector 110 increases, the relevant control unit 106 or 108 operates to  
10 reduce the output power of the LED 102 or HeNe laser 104. By using the blue light  
reflected by the dichroic element 116 and the red light passed by the dichroic element  
116 to monitor the output powers the LED 102 and laser 104, the need for more  
complex arrangements to monitor beam power incident on the sample is avoided.  
Also, such light energy is not simply wasted, as in the prior art, and the number of  
15 optical elements in the apparatus 100 is reduced, reducing unwanted scattering and  
stray reflections and improving the signal-to-noise ratio in light scattered by the  
sample and detected by the detectors 112B-H.

The computation unit 114 is programmed to include steady-state output power values  
20 for the LED 102 and HeNe laser 108 which are fixed by the control units 106, 108.

Figure 2 shows a second example apparatus 200 of the invention for measuring  
particle size distribution. Parts of the apparatus 200 corresponding to parts of the  
apparatus 100 of Figure 1 are labelled using reference signs differing by 100 from  
25 reference signs used to label the corresponding parts in Figure 1. Light from a blue  
LED 202 and a HeNe laser 204 may be coupled onto a common path through a sample  
cell 222 containing a sample of particles the size distribution of which is to be  
measured. Dichroic element 216 carries a standard dichroic coating 217 which  
transmits 0.2% of the power incident from the HeNe laser 204 to detector 210. The  
apparatus 200 includes a reflective element 219 which reflects a few percent of the  
30 output power of the HeNe laser 204 to a second detector 211. A few percent of the  
output power of the blue LED 202 is reflected by dichroic element 216 to a first  
detector 210. The output powers of the LED 202 and the HeNe laser 204 are  
stabilised by control units 206, 208 in response to output signals from the first 210  
35 and second 211 detectors respectively. Both blue and red light transmitted by the

sample cell 222 is detected by detector 212A. Light of both wavelengths scattered by the sample in the sample cell 222 is detected by one or more of detectors 212B-H. Output signals from detectors 212A-H are passed to computation unit 214 which is programmed to include the steady-state values of the output powers of the LED 102 and HeNe laser 104 as fixed by control units 204, 206.

Dichroic element 216 is integrated into a dust-free housing 220 together with mirror 218, reflective element 219 and focussing optic 215. The dust-free housing 220 reduces or eliminates scattering of light between the LED 202 and HeNe laser 204 and the sample cell 222.

Figure 3 shows a third example apparatus 300 of the invention. Parts of the apparatus 200 corresponding to parts of the apparatus 100 of Figure 1 are labelled using reference signs differing by 200 from reference signs used to label the corresponding parts in Figure 1. The apparatus 300 is similar to the apparatus 200 of Figure 2 in that dichroic coating 317 is a standard dichroic coating and in that first 310 and second 311 detectors are provided to monitor the output powers of the LED 302 and HeNe laser 311. Output from the first 310 and second 311 detectors is passed to the computation unit 314 together with output from detectors 312A-H to allow computation of particle size distribution for the sample within the sample cell 322. Detector 312A detects both blue and red light transmitted by sample cell 322.

Figure 4 shows a fourth example apparatus 400 of the invention. Parts of the apparatus 400 corresponding to parts of the apparatus 100 of Figure 1 are labelled using reference signs differing by 300 from reference signs used to label the corresponding parts in Figure 1. The apparatus comprises a dichroic element 416 having a custom dichroic coating 417 which passes 5% of the light from HeNe laser 404. A single detector 410 is used to monitor the output powers of blue LED 402 and HeNe laser 404. Output signals from the detector 410 are passed to a computation unit 414 together with output signals from detectors 412A-J to allow computation of particle size distribution of a sample within sample cell 422. Detector 412J detects blue light transmitted by sample cell 422. Red light transmitted by sample cell 422 reflected to detector 412A.

Dichroic element 416 is comprised in a dust-free housing 420 together with mirror 418 and focussing optic 415 to reduce unwanted scattering by dust between LED 402 and sample cell 422, and between laser 404 and sample cell 422.

**CLAIMS**

1. Apparatus for measuring particle size distribution of a sample by light scattering, the apparatus comprising light-generating means for generating first and second beams of light having first and second wavelengths respectively, a  
5       dichroic element arranged to direct most of the power of the first and second beams onto a common path by transmission and reflection of the first and second beams respectively at the dichroic element, and wherein the apparatus further comprises a first detector arranged to detect a portion of the first beam reflected  
10       by the dichroic element.
2. Apparatus according to claim 1 further comprising a control system for controlling the power of the first beam and wherein said control system is arranged to receive an output signal from the first detector and to adjust the  
15       power of the first beam in response thereto.
3. Apparatus according to claim 1 comprising a computation unit arranged to receive a signal from the first detector and to calculate particle size distribution of the sample based in part on the signal.  
20
4. Apparatus according to claim 1 wherein the first detector is arranged to detect a portion of the second beam transmitted by the dichroic element.
5. Apparatus according to claim 1 further comprising a second detector and an  
25       optical element arranged to transmit substantially all the power of the second beam to the dichroic element and to reflect a portion of the power of the second beam to the second detector.
6. Apparatus according to claim 4 or claim 5 further comprising a control system  
30       for controlling the power of the first and second beams and wherein said control system is arranged to receive an output signal from the first detector, or as the case may be output signals from the first and second detectors, and to adjust the powers of the first and second beams in response thereto.

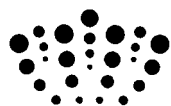
7. Apparatus according to claim 4 or claim 5 comprising a computation unit arranged to receive an output signal from the first detector, or as the case may be output signals from the first and second detectors, and to calculate particle size distribution of the sample based in part on said output signal or output signals.  
5
8. Apparatus according to any preceding claim wherein the first wavelength is shorter than the second wavelength.
- 10 9. Apparatus according to claim 8 wherein the light-generating means comprises a blue laser diode or blue LED for generating the first beam, and a helium-neon laser or a red LED for generating the second beam.
- 15 10. Apparatus according to claim 8 or claim 9 wherein the dichroic element is an glass optical element having substantially plane parallel sides one of which carries a dichroic coating.
- 20 11. Apparatus according to claim 10 wherein the rate of change of reflectivity of the dichroic coating as a function of wavelength at the first wavelength is substantially zero.
12. Apparatus according to claim 10 or claim 11 wherein the transmission  $T_{\lambda 2}$  of the dichroic coating at the second wavelength is in the range  $2\% \leq T_{\lambda 2} \leq 10\%$ .
- 25 13. Apparatus according to any preceding claim wherein the dichroic element is comprised in a dust-free housing together with one or more other optical elements for delivery of light to a sample within the apparatus.
- 30 14. A method of measuring particle size distribution for a sample comprising the steps of:
  - (i) generating first and second beams of light having first and second wavelengths respectively; and
  - (ii) using a dichroic element to direct most of the power of the first and second beams onto a common path by transmission and reflection of the first and second beams respectively at the dichroic element;  
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wherein the method comprises the step of using a first detector to detect a portion of the first beam reflected by the dichroic element and generate a corresponding output signal.

- 5     15. A method according to claim 14 further comprising the step of providing the output signal of the first detector to a control system arranged to adjust the power of the first beam in response to the output signal.
- 10     16. A method according to claim 14 comprising the step of providing the output signal of the first detector to a computation unit arranged to calculate particle size distribution for the sample based in part on the output signal.
- 15     17. A method according to claim 14 comprising the step of using the first detector to detect a portion of the second beam transmitted by the dichroic element.
- 20     18. A method according to claim 14 comprising the step of using an optical element in the second beam to transmit most of the power of the second beam to the dichroic element and to reflect a portion of the power of the second beam to a second detector.
- 25     19. A method according to claim 17 or claim 18 comprising the step of providing an output signal from the first detector, or as the case may be output signals from the first and second detectors, to a control system arranged to control the power of the first and second beams in response thereto.
- 30     20. A method according to claim 17 or claim 18 comprising the step of providing the output signal of the first detector, or as the case may be the output signals of the first and second detectors, to a computation unit arranged to calculate particle size distribution for the sample based in part on said output signal or signals.
21. Apparatus for measuring particle size distribution, the apparatus being substantially as described above with reference to any one of Figures 1 to 4.

22. A method of measuring particle size distribution, the method being substantially as described above with reference to any one of Figures 1 to 4.





**Application No:** GB1208181.6

**Examiner:** Simon Colcombe

**Claims searched:** 1-20

**Date of search:** 29 May 2012

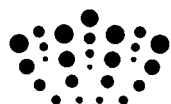
## Patents Act 1977: Search Report under Section 17

### Documents considered to be relevant:

Category	Relevant to claims	Identity of document and passage or figure of particular relevance
Y	1-20	GB2340936 A (MALVERN INSTRUMENTS) Figures 2-5 and related description, in particular
Y	1-20	EP0485817 A1 (HORIBA) Figure 5 and related description, in particular
Y	1-20	EP1884762 A2 (SHIMADZU) Figures 1 and 3 and related description, in particular
Y	1-20	WO00/77489 A1 (GOLDBECK) Figure 2 and related description
Y	1-20	GB2346444 A (HARLEY SCIENTIFIC) Figure 2 and related description
Y	1-20	US4957363 A (TAKEDA) Figures 1, 4 and 7
A	-	US2006/052944 A1 (NAGURA)
A	-	US5416580 A (TRAINER)
A	-	US4361403 A (LOOS)

### Categories:

X	Document indicating lack of novelty or inventive step	A	Document indicating technological background and/or state of the art.
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&	Member of the same patent family	E	Patent document published on or after, but with priority date earlier than, the filing date of this application.



**Field of Search:**

Search of GB, EP, WO & US patent documents classified in the following areas of the UKC<sup>X</sup> :

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Worldwide search of patent documents classified in the following areas of the IPC

G01N
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The following online and other databases have been used in the preparation of this search report

EPODOC, WPI
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**International Classification:**

Subclass	Subgroup	Valid From
G01N	0015/02	01/01/2006
G01N	0021/47	01/01/2006
G01N	0021/49	01/01/2006