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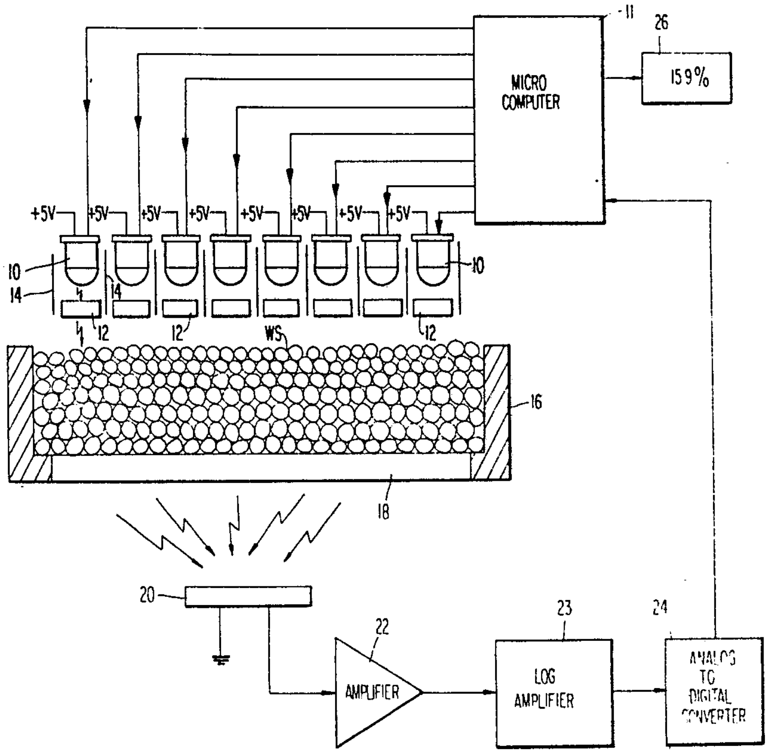
(54) Title: APPARATUS FOR NEAR INFRARED QUANTITATIVE ANALYSIS

(57) Abstract

An apparatus for near infrared quantitative analysis with an infrared emittig diode (IRED) source (10) utilizes narrow bandpass filters (12) and silicon detectors (20) to provide highly accurate and inexpensive measurement. A narrow bandpass filter (12) in the light path of the IRED (10) passes wavelengths outside the half power bandwidth points of the IRED (10). A silicon detector (20) together with a sensitive amplifier (22) is used to accurately measure linearly up to six decades of light sensitivity at desired wavelengths.



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APPARATUS FOR NEAR INFRARED QUANTITATIVE ANALYSIS

Background

Field of the Invention

The invention relates to improvements in apparatus for near infrared (NIR) analysis for measuring chemical constituents in a quantitative fashion. More particularly, the improvements of this invention allow accurate measurements with inexpensive instruments.

Prior Art

There are known in the prior art instruments which measure chemical constituents in a product by means of either reflecting near infrared radiant energy off the product or transmitting infrared energy through the product. These instruments use the phenomenon that certain organic substances absorb energy in the near infrared portion of the spectrum. By measuring the amount of energy absorbed by the product at certain specific wavelengths, precise quantitative measurements of the constituents in a product become available. For example, the measurement of protein content in wheat can be performed in this manner. For general introduction to near infrared quantitative analysis, see the paper presented by Robert D. Rosenthal to the 1977 Annual Meeting of American Association of Cereal Chemists entitled "An Introduction to Near Infrared Quantitative Analysis." Such analysis can be performed on samples within only a few seconds without special sample preparation except for grinding the sample.

Current commercial near infrared instruments use the same generic technology for developing the near infrared energy. The commercial instruments use a tungsten light source that provides radiant energy through the visible, the near infrared as well as the longer infrared portions of the spectrum. This broad spectrum of energy is modified by the use of narrow band optical filters (or gratings or prisms) to allow only a small selected portion of the total spectrum to fall on the object being measured. Thus, the current instruments are not efficient in the use of illuminating energy because only such a very small portion of the total energy provided by the tungsten lamp is used in the measurement. The great majority of the radiated energy from the tungsten lamp is deliberately not used and is dissipated to heat. This causes temperature problems and other penalties in instrument design.

The instrument laboratory of the U.S. Department of Agriculture in Beltsville, Maryland, several years ago, performed studies on the use of another type of "light source." In those studies infrared emitting diodes (IRED's are solid state devices that yield energy at very narrow regions of the spectrum compared to a conventional tungsten bulb) were used. The U.S.D.A. work was summarized in the technical paper entitled "Fat Measurement of Ground Beef with a Gallium Arsenide Infrared Emitter" (ASAE publication 1-76 "Quality Detection in Foods"). In the U.S.D.A.'s technical paper there is mentioned the possibility of narrowing the region of infrared energy by putting narrow bandpass filters in front of the IRED. This, however, was directed to an effort for measurement of wavelengths between 920 and 970 nanometers (nm).

In 1978 U.S.D.A. research discovered that precise measurement of the constituents in grain (e.g., protein) can be made at wavelengths slightly longer than discussed in the 1976 paper. The desired wavelengths for protein measurement in grain are between 1000 and 1060nm.

Previously protein measurement in grain required measurement at wavelengths above 1200nm. These longer wavelengths forced the use of poor performing PbS light detectors. Thus, the U.S.D.A. discovery that precise measurement of protein in grain can be wavelengths shorter than 1200nm appears to be of great value because it allows the use of lower cost, and more stable, silicon detectors.

The U.S.D.A. began to search for IREDs with center wavelengths between 1000 and 1060nm. Unfortunately, only very expensive indium gallium agenide ARED's are available in this wavelength. Conventional IREDs of gallium arsenide provide peak energy well below 1000nm; normally about 940nm. Therefore, although the conventional IREDs are of low cost (about a third of 1% of the indium gallium arsenide IRED's) they are not considered useful for protein measurement because the half power bandwidth was limited to between 910 and 970nm. Half power bandwidth refers to the wavelength that has one-half of the output power as the center wavelength. Normally, IREDs are never used outside the half power bandwidth points.

Summary of the Invention

This invention utilizes a combination of the properties of conventional IREDs and of silicon detectors to allow the conventional low cost IREDs to be used outside their half power bandwidth up to (at least) 1060nm. Conventional IREDs that have a peak radiation at 940nm only emit about 1 to 10% of their power at 1000nm as the wavelength gets longer, toward 1060nm, the amount of energy radiated falls even lower.

Silicon detectors are well known for having the unique property of being sensitive and linear over seven decades of light level. This invention, involving the combining of a conventional IRED and silicon detector, allow six decades of light sensitivity at wavelengths between 1000 and 1060nm to be measured linearly.

In specific application, a narrow bandpass optical filter is positioned in the light path of a conventional IRED and the energy transmitted through the narrow bandpass is measured with a silicon detector which together with sensitive amplifiers (e.g., logarithmic amplifiers) allows the IRED to provide a specific measurement wavelength of up to 1060nm.

In some cases, many wavelengths are simultaneously needed. Thus, the preferred embodiment of the invention includes multiple numbers of IREDs each with its own specific bandpass filter. The IREDs are rippled, i.e., turned on one at a time consecutively, thereby allowing a single silcon detector to measure many wavelengths. The IRED has low cost and extremely low power dissipation property. Thus, this invention having for example 12 IREDs, the total power dissipation is still less than 1% of what would be used with a normal tungsten lamp of the prior art.

Another embodiment of the invention involves mounting a number of bandpass filters on a flat spinning wheel and using a single IRED to accomplish the functional equivalent of the multiple IRED arrangement described.

Brief Description of the Drawings

Figure 1 is a curve of a typical, spectral radial flux from a commercial infrared emitting diode showing the five wavelengths required for wheat protein measurement;

Figure 2 illustrates a preferred embodiment of an apparatus of this invention.

Detailed Description of the Preferred Embodiment

The preferred embodiment will be described with regard to making accurate protein measurements of wheat by means of the magnitude of near infrared light absorbed passing through the wheat.

Research at the U.S. Department of Agriculture (USDA) has shown that the protein content of wheat can be measured by transmitting selective wavelengths of near infrared light through the wheat and determining the magnitude of light absorbed at each of selected wavelengths. The USDA research demonstrated that accurate protein measurement can be made using the following algorithm:

$$\text{Percent Protein} = k_o + k_1 \left\{ \frac{\log \frac{1}{T_1} - 2 \log \frac{1}{T_2} + \log \frac{1}{T_3}}{\log \frac{1}{T_4} - 2 \log \frac{1}{T_2} + \log \frac{1}{T_5}} \right\}$$

where k_o & k_1 are proportionality constants

T_1 = percent light transmitted through the wheat sample at 1007.5 nanometers (nm)

T_2 = percent light transmitted through the wheat sample at 1022 nm

T_3 = percent light transmitted at 1036.5 nm

T_4 = percent light transmitted at 997 nm

T_5 = percent light transmitted at 1057 nm

Figure 1 shows the spectrum distribution of the light emitted by typical commercial infrared emitting diodes (IRED). Also shown in Figure 1 are the five wavelengths required for wheat protein measurement. Most of the required wavelengths are in the region where the IRED emits very little optical energy. However, a finite amount of usable energy is still emitted at such wavelengths even though the wavelengths are outside the half power bandwidth points of the IRED as shown in Figure 1.

Figure 2 illustrates the preferred embodiment in schematic form.

In Figure 2 eight commercial IREDs are sequentially pulsed on and off by signals from a microprocessor 11. The microprocessor is programmed to allow only one of the IREDs to be turned on at a time and automatically sequences through all eight IREDs. In addition the time the IRED is on is controlled by the microprocessor. The IREDs are pulsed so that they are driven with much more current than in a DC state. This is necessary because of the small amount of optical energy that is radiated at the wavelengths of interest.

The infrared light emitted from each IRED 10 is transmitted through an accompanying narrow bandpass optical filter 12. The optical filter 12 absorbs all wavelengths of light except those near its center wavelength. These eight filters all have different center wavelengths. Five are used for wheat protein measurement wavelengths of Figure 1 and the remaining three wavelengths are used to measure additional parameters such as the moisture content of wheat. Light baffles 14 between each set of IREDs and narrow bandpass filters prevent the light from one IRED being transmitted through an adjacent narrow bandpass filter.

Each of the specific wavelengths are transmitted through a wheat sample WS that is held in an opaque cup 16. The cup has a glass bottom 18 so that the light energy can pass through and impact on a silicon photodetector 20. This silicon detector is especially designed to measure extremely low light levels. The light impacting on the silicon detector is converted by the detector into an electrical current which is amplified by a low noise amplifier 22. The output of this amplifier is fed to and further amplified by a log amplifier 23. The amplified signal is then converted to a digital signal for inputting into a microcomputer by means a 12 bit analog-to-digital converter 24.

The microprocessor 11 records in its memory the amount of transmitted energy obtained from each of the IREDs. It then uses the equation set forth above to calculate the amount by weight of protein in the sample of wheat. The result is displayed on a digital meter 26 built into the instrument.

The combination of the microprocessor and the IRED array allows large numbers of measurements to be made and simultaneously averaged in less than a second to thereby reduce error as compared to using a single or small number of like measurements.

In one specific example in an instrument constructed in accordance with this invention, the IREDs 10 are General Electric Company part No. 1N6264. The microprocessor 11 is an Intel 8085 system. The silicon detector 20 is a Silicon Detector Corporation part SD-444-11-11-251. The amplifier 22 is an Analog Devices Corp. AD517, the log amplifier 23 is an Analog Devices Corp. AD755N, and the analog-to-digital converter 24 is an Analog Devices Corp. AD574. Although the preceding discussion described a system using a silicon detector, other types of detectors with similar operating characteristics can be used, e.g., cadmium sulfide, or germanium.

Claims

1. An apparatus for near infrared quantitative analysis of the type particularly useful for but not limited to measurements of quantitative analysis of chemical constituents within grain and being of the type having an infrared emitting source, an infrared detector positioned to receive infrared energy transmitted by the source toward a sample, and a filter selected to allow passage of a preselected wavelength band through it positioned between the source and the detector with the improvements comprising and characterized by the source being a pulsed infrared emitting diode (IRED) of a specific one-half power bandwidth, and the filter being a stationary bandpass filter selected to allow passage of a preselected wavelength band through it, which selected band is outside the one-half power bandwidth of the IRED.

2. An apparatus as defined in Claim 1, wherein the detector is a silicon detector and further comprising a sensitive amplifier connected to the silicon detector.

3. An apparatus as defined in Claims 1 or 2, wherein there are a plurality of IREDs and a plurality of station bandpass filters, one for each IRED.

4. An apparatus as defined in Claims 1-3, wherein each of the IREDs are turned on one at a time consecutively and are detected by a single silicon detector.

5. An apparatus as defined in Claims 3 and 4 wherein a microprocessor is used to control the IREDs and provide a large number of averaged measurements in less than a second.

6. An apparatus as defined in Claim 5 wherein the microprocessor utilizes the amplified output of the silicon detector to compute with a preselected algorithm and includes means to display the results of the computation.

7. An apparatus as defined in Claims 1-6 wherein the preselected wavelength bands of the bandpass filters include those wavelengths set out in Figure 1.

8. An apparatus as claimed in Claims 2-7 further comprising an amplifier connected to the silicon detector feeding a logarithmic amplifier whose output in turn is converted to digital signals used to input a microprocessor.

AMENDED CLAIMS

(received by the International Bureau on 11 February 1981 (11.02.81))

1. An apparatus for near infrared quantitative analysis, particularly for measurements of chemical constituents in grain, and including an infrared emitting source, an infrared detector positioned to receive infrared energy transmitted by the source through a sample, and a filter selected to allow passage of a preselected wavelength band through it positioned between the source and the detector, characterized by:

a) a plurality of infrared emitting sources, each comprising a pulsed infrared emitting diode (IRED) having a specific one-half power bandwidth, and

b) an equal plurality of stationary bandpass filters individually associated with the plurality of IREDs, each filter having a different preselected wavelength band lying outside the one-half power bandwidth of the IREDs.

2. An apparatus as defined in Claim 1, wherein the detector is a silicon detector and further comprising a sensitive amplifier connected to the silicon detector.

3. An apparatus as defined in Claims 1 or 2, wherein each of the IREDs is turned on one at a time consecutively and is detected by a single silicon detector.

4. An apparatus as defined in any of the preceding Claims, wherein a microprocessor is used to control the IREDs and provide a large number of averaged measurements in less than a second.

5. An apparatus as defined in Claim 4, wherein the microprocessor utilizes the amplified output of the silicon detector to compute with a preselected algorithm and includes means to display the results of the computation.

6. An apparatus as defined in any of the preceding Claims wherein the preselected wavelength bands of the bandpass filters include those wavelengths set out in Figure 1.

7. An apparatus as claimed in Claim 1, further comprising an amplifier connected to a silicon detector feeding a logarithmic amplifier whose output in turn is converted to digital signals used to input a microprocessor.

EDITORIAL NOTE

The applicant failed to renumber the amended claims in accordance with Section 205 of the Administrative Instructions.

In the absence of any specific indication from the applicant as to the correspondence between original and amended claims, these claims are published as filed and as amended.

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FIG 1

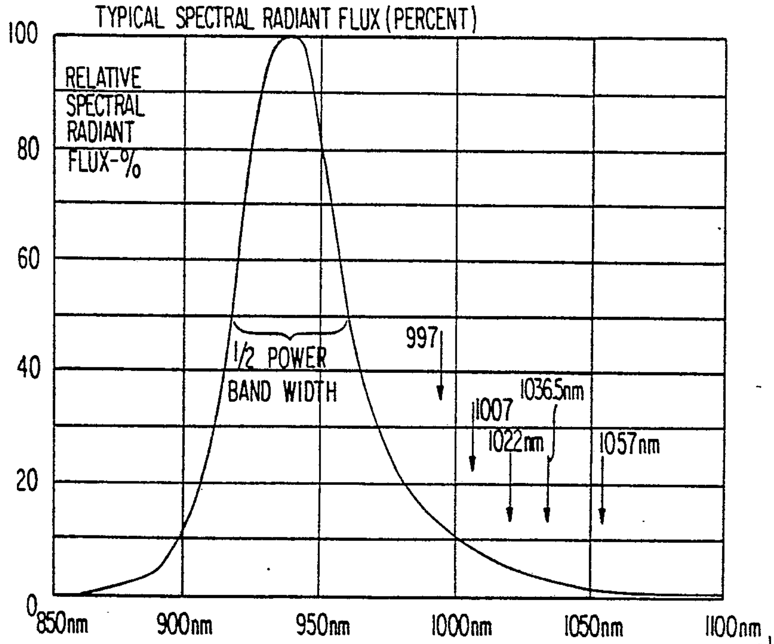
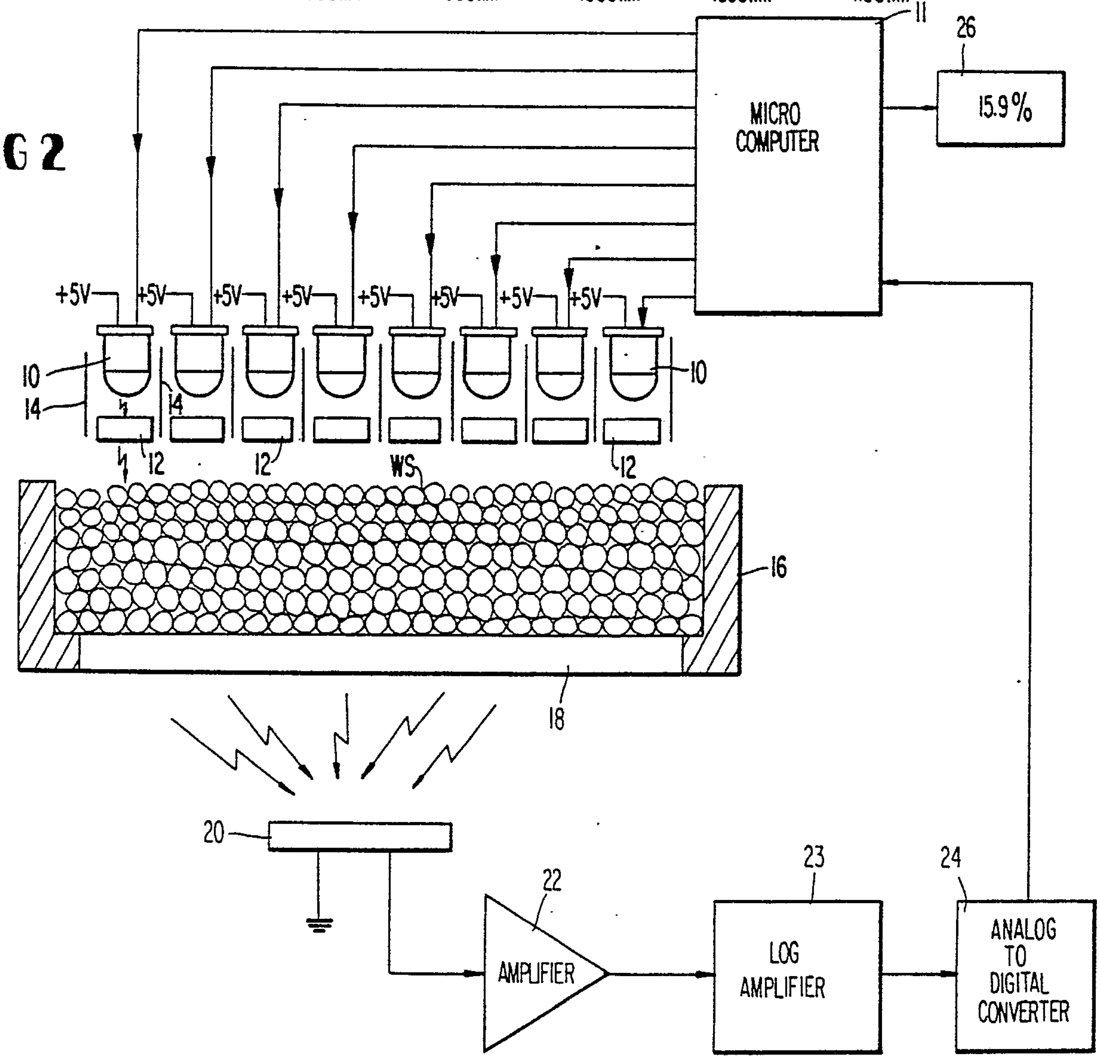

TYPICAL SPECTRAL RADIANT FLUX (PERCENT)
RELATIVE SPECTRAL RADIANT FLUX-%
100
80
60
40
20
0
850nm
900nm
950nm
1000nm
1050nm
1100nm
1/2 POWER BAND WIDTH
997
1007
1022nm
1036.5nm
1057nm

FIG 2


MICRO COMPUTER
11
26
15.9%
+5V
10
14
12
WS
16
18
20
22
AMPLIFIER
23
LOG AMPLIFIER
24
ANALOG TO DIGITAL CONVERTER

INTERNATIONAL SEARCH REPORT

International Application No PCT/US80/01166

I. CLASSIFICATION OF SUBJECT MATTER (if several classification symbols apply, indicate all) [3]

According to International Patent Classification (IPC) or to both National Classification and IPC

Int. Cl.[3] G06F 15/20; G01N 21/00; G01J3/48
U.S. Cl. 364/497, 498; 250/338; 356/416

II. FIELDS SEARCHED

Minimum Documentation Searched [4]

Classification System	Classification Symbols
U.S.	364/496, 497,498,499,526 250/338,341,343,345 356/416,418

Documentation Searched other than Minimum Documentation to the Extent that such Documents are Included in the Fields Searched [5]

III. DOCUMENTS CONSIDERED TO BE RELEVANT [14]

Category *	Citation of Document, [16] with indication, where appropriate, of the relevant passages [17]	Relevant to Claim No. [18]
X	US, A, 3,776,642 Published 04 December 1973, Anson et al.	1-3
X	US, A, 3,823,173 Published 06 August 1974, Knepler.	1-3
A	US, A, 4,040,747 Published 09 August 1977, Webster.	1-3
A	US, A, 3,765,775 Published 16 October 1973, Ganssle et al.	1-3
A	US, A, 4,035,643 Published 12 July 1977, Barrett	1-3
A,P	US, A, 4,207,466 Published 10 June 1980, Drage et al.	1-3
A,P	US, A, 4,190,851 Published 26 February 1980, Finnila et al.	2
A	R. Rosenthal, 'An Introduction to Near Infrared Quantitative Analysis', Published 1978, by Neotec Instruments, Inc. See pages 1-15.	1

* Special categories of cited documents: [15]

"A" document defining the general state of the art
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"O" document referring to an oral disclosure, use, exhibition or other means
"P" document published prior to the international filing date but on or after the priority date claimed
"T" later document published on or after the international filing date or priority date and not in conflict with the application, but cited to understand the principle or theory underlying the invention
"X" document of particular relevance

IV. CERTIFICATION

Date of the Actual Completion of the International Search [2]	Date of Mailing of this International Search Report [2]
10 December 1980	22 JAN 1981
International Searching Authority [1]	**Signature of Authorized Officer [20]**
ISA/US	Gary Ch[illegible]

Form PCT/ISA/210 (second sheet) (October 1977)

International Application No. PCT/US80/01166

III. DOCUMENTS CONSIDERED TO BE RELEVANT (CONTINUED FROM THE SECOND SHEET)		
Category *	Citation of Document, [16] with indication, where appropriate, of the relevant passages [17]	Relevant to Claim No [18]
A	D·Massie, 'Fat Measurement of Ground Beef with a Gallium Arsenide Infrared Emitter', Published 1976, by ASAE Publication.	1-3

Form PCT/ISA/210 (extra sheet) (June 1980)

International Application No. PCT/US80/01166

FURTHER INFORMATION CONTINUED FROM THE SECOND SHEET

V. [X] OBSERVATIONS WHERE CERTAIN CLAIMS WERE FOUND UNSEARCHABLE [10]

This international search report has not been established in respect of certain claims under Article 17(2) (a) for the following reasons:

1. [ ] Claim numbers ...., because they relate to subject matter [12] not required to be searched by this Authority, namely:

2. [X] Claim numbers 4-8 because they relate to parts of the international application that do not comply with the prescribed requirements to such an extent that no meaningful international search can be carried out [13], specifically:

These claims are improper multiple dependent claims.

VI. [ ] OBSERVATIONS WHERE UNITY OF INVENTION IS LACKING [11]

This International Searching Authority found multiple inventions in this international application as follows:

1. [ ] As all required additional search fees were timely paid by the applicant, this international search report covers all searchable claims of the international application.
2. [ ] As only some of the required additional search fees were timely paid by the applicant, this international search report covers only those claims of the international application for which fees were paid, specifically claims:
3. [ ] No required additional search fees were timely paid by the applicant. Consequently, this international search report is restricted to the invention first mentioned in the claims; it is covered by claim numbers:

Remark on Protest

[ ] The additional search fees were accompanied by applicant's protest.

[ ] No protest accompanied the payment of additional search fees.