



EP. 0039734

## INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

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| <b>(51) International Patent Classification:</b><br>G02B 5/128; C08K 3/00; B05D 1/42   | <b>A1</b> | <b>(11) International Publication Number:</b> WO 81/01471<br><b>(43) International Publication Date:</b> 28 May 1981 (28.05.81)   |
| <b>(21) International Application Number:</b> PCT/US80/01377<br><b>(22) International Filing Date:</b> 14 October 1980 (14.10.80)<br><b>(31) Priority Application Number:</b> 094,696<br><b>(32) Priority Date:</b> 16 November 1979 (16.11.79)<br><b>(33) Priority Country:</b> US<br><b>(71) Applicant:</b> MINNESOTA MINING AND MANUFACTURING COMPANY [US/US]; 3M Center, P.O. Box 33427, St. Paul, MN 55133 (US).<br><b>(72) Inventor:</b> BINGHAM, Wallace, Karl; P.O. Box 33427, St. Paul, MN 55133 (US).<br><b>(74) Agents:</b> TAMTE, Roger, R. et al.; Office of Patent Counsel, Minnesota Mining and Manufacturing Company, P.O. Box 33427, St. Paul, MN 55133 (US).   |           | <b>(81) Designated States:</b> AT (European patent), AU, CH (European patent), DE (European patent), FR (European patent), GB (European patent), JP, LU (European patent), NL (European patent), SE (European patent).<br><br><b>Published</b><br><i>With international search report</i> |
| <b>(54) Title:</b> COATING COMPOSITION FOR FORMING RETROREFLECTIVE TREATMENTS ON FABRICS   |           |   |
|  |           |   |
| <b>(57) Abstract</b><br><br>Coating compositions for coating fabrics to make them brightly retroreflective at nighttime while making a minimal change in their ordinary daylight appearance. The compositions comprise a dilute coating vehicle, preferably an aqueous emulsion of an acrylic-based polymer, and transparent retroreflective microspheres dispersed in the vehicle. The vehicle is diluted with at least 40 volume-percent of a volatile constituent, and the microspheres account for about one-third or less of the volume of the nonvolatile portion of the composition. When the composition is coated onto a fabric (10) the retroreflective microspheres (12) become scattered over the surface of the fabric at low density, often in recesses (13) in the fabric surface, and become adhered to the fabric by a binder material (11) in the coating vehicle. |           |   |

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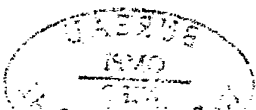
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Background Information

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DescriptionCOATING COMPOSITION FOR FORMING  
RETROREFLECTIVE TREATMENTS ON FABRICSTechnical Field

5           The present invention provides new coating compositions useful for coating fabrics to make them brightly visible under retroreflective viewing conditions without significantly changing their ordinary daylight appearance.

Background Art

10           U.S. Pat. 4,103,060 teaches retroreflective treatments that comprise retroreflective glass microspheres adhered over the surface of a fabric in a low-density, scattered or spaced arrangement. Such treatments are difficult to detect under ordinary daylight viewing conditions;  
15 but the coated garments are brightly visible when viewed in the dark under retroreflective viewing conditions (i.e., with the observer located on substantially the same axis as a light beam shined at the fabric). The low daytime visibility of these treatments gives promise of overcoming  
20 consumer's resistance to prior retroreflective treatments, which were readily visible during ordinary or casual viewing in daylight.

          The specific methods and materials taught in U.S. Pat. 4,103,060 for preparing the described retroreflective  
25 treatments have not become commercial. The principal method was to cascade particles onto the fabric particles under conditions which softened a binder material in the particles and adhered them to the fabric. The method did not seem adapted to existing fabric-treating methods; and with some  
30 tightly woven fabrics the applied particles protruded above the surface of the fabric and had more daytime visibility than desired.

Another method taught in U.S. Pat. 4,103,060 was to print retroreflective coating compositions onto a fabric in a pattern of spaced areas. Useful retroreflective coating compositions based on hemispherically reflectorized glass microspheres had been taught in Palmquist et al, U.S. Pat. 2,963,378, and further teachings appeared in Nellessen, U.S. Pats. 3,099,637; 3,228,897; Nellessen et al, U.S. Pat. 3,420,597; Longlet et al, U.S. Pat. 3,535,019 (compositions particularly intended for coating fabrics); Searight, U.S. Pat. 3,835,087; and Hogseth et al, Canadian Pat. 935,254.

The coating of discrete separated areas has not become commercial either. It was difficult to apply sufficient microspheres to provide desired levels of reflectivity without also causing too great daytime conspicuity. Also, the printing operation left a regular pattern that increased the daytime visibility of the treatment.

#### Disclosure of Invention

The present invention provides new coating compositions that may be coated onto a wide range of fabrics to provide retroreflective treatments that are especially difficult to detect under ordinary daylight viewing conditions. Briefly, a composition of the invention comprises a) a dilute coating vehicle that comprises film-forming binder material and at least 40 volume-percent volatile constituent, and b) transparent microspheres having a refractive index between about 1.85 and 2 hemispherically coated with specular reflective means and dispersed in the coating vehicle in an amount accounting for no more than about one-third of the volume of the nonvolatile portion of the composition.

Compositions of the invention differ from prior-art coating compositions in important ways -- including the concentration of microspheres in the coating composition. The prior-art coating compositions were generally intended

to provide coatings having as dense a concentration of microspheres as possible. For that reason, microspheres constituted a large proportion, i.e., 50 volume-percent or more, of the nonvolatile components of the composition.

Based on the principles and objectives of these prior-art teachings, a reduction of the concentration of microspheres to the amounts used in the present invention would take away utility. But to the contrary, it has now been found that a reduced concentration of microspheres dispersed in a dilute coating vehicle allows preparation of daytime-inconspicuous, nighttime-retroreflective treatments needed to achieve broadly wearable garments.

More specifically, it has been found that when a composition as described is coated onto a fabric -- as by spreading with a knife blade, coating from a rotogravure cylinder, or spraying -- it provides a sparse distribution of microspheres over the surface of the fabric. The binder material in the coating vehicle becomes distributed across the surface of the fabric, and upon drying and/or reaction forms a thin layer holding the microspheres to the fabric. Individual microspheres are partially embedded in the layer and partially protrude above the layer. The microspheres may be randomly oriented, but typically, on the average at least one-third of them are arranged with the hemispherical reflector between the microsphere and the fabric, and with the outwardly facing surface of the microspheres optically exposed.

Some useful characteristics noted in the coating include:

- 1) The microspheres are well distributed across the surface of the fabric. By choice of the volume-percent of microspheres in the composition, the microspheres can be distributed at densities within the parameters taught in the previously noted Bingham and Bailey, U.S. Pat. 4,103,060, i.e., at a density of no more than about 2000, and preferably no more than about 500, per square centi-

meter of the coating.

2) Any concentrations of microspheres are small, with many of the microspheres isolated or separated from other microspheres of the treatment. The maximum surface dimension of discrete retroreflective areas, i.e., concentrations or clusters of microspheres that are seen as one retroreflective area during retroreflective viewing, is no more than about one millimeter.

3) The microspheres tend to nestle in recesses in the surface of the fabric, i.e., in the valleys or interstices between yarns of the fabric. This nestling minimizes removal of the microspheres during ordinary use and reduces daylight visibility of the microspheres. Coating compositions of the invention are useful on a wide variety of fabrics, including tightly woven fabrics such as nylon used in exterior shells for jackets and coats.

Overall, a treatment of the invention is difficult to detect in daylight viewing. The microspheres are very small, often on the order of 50 micrometers in diameter or less, and even clusters of microspheres are not easily visible. Individual microspheres isolated from other microspheres are especially hard to detect. (Visual acuity of the human eye is typically regarded as one minute of arc for subjects illuminated under normal lighting conditions of 100-1000 foot-lamberts; at viewing distances of 15 and 30 inches, one minute of arc covers, respectively, 0.0043 inch (110 micrometers) and 0.0087 inch (221 micrometers)).

In contrast to their daytime inconspicuity, garments treated with coating compositions of the invention are brightly retroreflective at night when illuminated by the headlight beam of a vehicle. Under such retroreflective viewing conditions, each properly oriented microsphere sends back a pinpoint of light. In the aggregate, the myriad pinpoint reflections make the fabric bright and visible.

Brief Description of the Drawing

The drawing is a schematic representation in section of a fabric 10 that has been retroreflectorized with a composition of the invention. The retroreflective treatment comprises a coating or thin layer 11 of binder material in which hemispherically reflectorized microspheres 12 are partially embedded. The microspheres tend to be deposited in recesses or valleys 13 of the fabric, and may be randomly oriented. Typically at least about one-third of the microspheres are oriented with their hemispherical reflective cap 14 disposed between the microsphere and the fabric and with the outwardly facing surface 15 of the microsphere optically exposed so as to receive light rays.

15 Detailed Description

Binder materials that are useful for incorporation into coating compositions of the invention are already well known in the fabric-treating industry. Such binder materials have been used for textile printing (as for ornamental purposes), water-proof coatings, flocking binders, etc. These useful binder materials form durable adherent films when coated on a fabric and exposed to an appropriate environment.

Aqueous emulsions of acrylic-based polymers are preferred binder materials, because of the advantages of water as an inexpensive, convenient, and environmentally safe diluent, and because the acrylic polymers provide a durable well-adhered layer that leaves the fabric supple and flexible. A fabric treated with such binder materials can generally be folded upon itself without fracturing the coated layer of binder material, and the fabric will generally handle and feel approximately the same way after treatment as it did before treatment. These aqueous emulsions of acrylic-based polymers dry upon evaporation of water and coalesce as a continuous film. Typically they

also can be subsequently reacted to a more insoluble infusible state as by heating. Such a reacted binder material is not readily soluble in usual dry cleaning solvents.

Other useful binder materials for use in the  
5 coating vehicle of compositions of the invention are vinylidene chloride-based copolymers, polyesters, and polyurethanes. Although preferred reactive binder materials are one-part compositions which may be activated by application of heat, etc., two-part compositions stored separately  
10 until the time of coating can also be used.

To minimize the effect of the layer of applied binder material on the fabric, the layer is preferably very thin. Such thinness can be achieved by making the coating vehicle dilute; generally the vehicle includes at least 40  
15 volume-percent, and preferably at least 70 volume-percent, of a volatile constituent. Such a volatile constituent may be a volatile liquid in which latex particles are dispersed or a solvent or thinner. Although water is a preferred volatile constituent, organic liquids may also be used.

20 As discussed above, the number of microspheres within the composition is kept low, so as to provide the low densities of microspheres on fabric which provide desired daytime inconspicuity. Different degrees of daytime inconspicuity will be accepted for different kinds of  
25 garments and fashion schemes, and certain fabrics will achieve good daytime inconspicuity even with higher loadings of microspheres. Where full daytime inconspicuity is not needed, the microspheres may comprise up to about one-third of the volume of the nonvolatile ingredients in the  
30 composition; but more typically the microspheres comprise less than about one-fifth of the volume of the nonvolatile ingredients in the composition. The combination of daytime inconspicuity and retroreflectivity that seems preferred today is obtained with loadings of microspheres at levels  
35 less than about 15 volume-percent.

Generally sufficient microspheres are included in



a composition to provide a reflective treatment that exhibits at least 1 candella, and preferably at least 2.5 or 3 candellas, of reflected light per square meter of a treated surface per lux of light that is incident on the surface.

Since the microspheres constitute only a small percentage of the coating composition, they should be well dispersed within the composition immediately prior to application to a fabric. Such a dispersion can be achieved with mechanical stirring techniques. In addition, thixotropic agents such as carboxymethyl cellulose and polyacrylates can be added to the composition.

The microspheres included in the composition are visibly transparent, usually glass, and are preferably colorless in most embodiments of the invention. Where a hemispherical reflector is applied directly to the microsphere, as is most common, the index of refraction of the glass will be in a range between 1.85 and 2.0, and preferably will be slightly over 1.9. Microspheres of lower refractive index can be used by spacing the hemispherical reflector from the microsphere, as by precoating a glass microsphere with a material of lower refractive index. And microspheres of high index of refraction can be used in mixture with lower-index microspheres to obtain useful retroreflection when the microspheres are wet. The microspheres are generally less than 200 micrometers in average diameter, and preferably are less than 100 micrometers in average diameter, to minimize their visibility on treated fabric.

Procedures for hemispherically reflectorizing microspheres are taught in Palmquist et al, U.S. Pat. 2,963,378. Vapor-coated aluminum is the preferred hemispherical reflective coating, but dielectric coatings such as taught in Bingham, U.S. Pat. 3,700,305 (which is incorporated herein by reference) might also be used. Barrier films, such as aluminum phosphate or aluminum oxide films,

may be applied over a metal hemispherical coating in the manner taught in Longlet et al, U.S. Pat. 3,535,019, to increase the shelf-stability of compositions of the invention. Alternatively or in addition, additives such as  
5 6-acetoxy-2,4-dimethyl-m-dioxane or trans-1,2-bis(n-propylsulfonyl)ethylene may be included in the coating vehicle to increase shelf-stability; these additives are understood to inhibit microbial growth.

A variety of other additives may be included in  
10 the composition to achieve special results. For example, anti-foaming agents may be included in the coating vehicle to minimize foaming of the vehicle as it is being formulated or when it is applied to a fabric. Other additives which may be included in the composition are organofunc-  
15 tional silanes and transparent pigments and dyes, which are included as adhesion aids or coupling agents and as camouflaging or coloring compounds.

The preferred technique for applying a coating composition of the invention to a fabric is to spread the  
20 composition across the fabric with dam or barrier means such as a knife blade. The knife blade may be lightly pressed against the surface of the fabric or may be elevated above the fabric a slight amount and moved across the fabric. The coating composition moves ahead of the knife  
25 blade, with only a thin layer of coating vehicle being applied over the surface of the fabric, and with most of the microspheres tending to be deposited in recesses in the fabric. Because of the low concentration of nonvolatile binder material in the coating vehicle, the vehicle is  
30 reduced upon drying to a very thin film. Some penetration of the binder material into the fabric occurs as represented by penetration of the binder material 11 in the attached drawing. Instead of spreading composition across a fabric with a knife blade, the composition may be applied by other  
35 techniques such as rotogravure rolls, sprays or spray and knife-blade combinations, or screen printing.

The invention will be further illustrated with the following examples. A coating composition was prepared from the following ingredients.

|    |   | Parts by<br><u>Weight</u> |
|----|---|---------------------------|
| 5  | Aqueous emulsion comprising 54 weight-percent water and 46 weight-percent of a copolymer of ethyl acrylate and methylol acrylamide (Rhoplex HA-8 from Rohm & Haas Company)            | 26.6                      |
| 10 | Aqueous emulsion containing 80 weight-percent water and 20 weight-percent copolymer of ethyl methacrylate and acrylic acid used as thickener (Acrysol ASE95 from Rohm & Haas Company) | 10.7                      |
| 15 | Anti-foaming agent (Nopco DF160L from Diamond Shamrock Company; diluted with 50 percent water)  | 0.9                       |
|    | Ammonium nitrate catalyst enhancing cure of acrylic-based polymer (diluted with 75 percent water)   | 0.4                       |
| 20 | Silane coupling agent (Dow Corning Z6040)   | 0.9                       |
|    | Nitrogenous thermosetting resin catalyst agent (Arotex M-3 from American Cyanamid Company)  | 0.9                       |
| 25 | Hemispherically aluminized glass microspheres of approximately 1.92 refractive index averaging 45 micrometers in diameter and ranging between 21 and 63 micrometers in diameter       | 6.4                       |
|    | Water   | 53.2                      |

The described ingredients were mixed together and adjusted to a pH of 8 with ammonium hydroxide to prepare a coating composition having a viscosity of about 1700 centipoises.

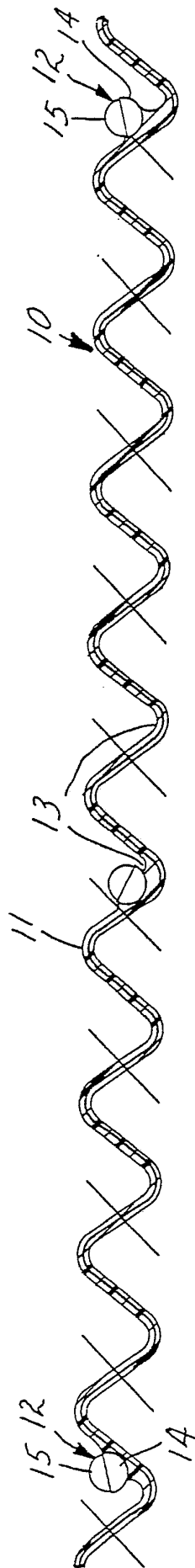
The microspheres constituted 1.84 volume-percent of the total coating composition and 11.58 percent of the non-volatile components of the composition.

5 The composition was coated onto a tightly-woven nylon oxford fabric using a knife blade riding directly against the fabric. The composition was dried by forced air heated to 150°F (66°C) and subsequently was heated for 10 minutes at 305°F (150°C) to cure the binder material. The fabric was found to be coated with a continuous layer  
10 of binder material with microspheres distributed over the surface of the fabric at a density of about 1450 microspheres per square centimeter. Almost all the microspheres rested in recesses in the surface of the fabric. When the fabric was viewed in ordinary daylight the microspheres  
15 could not be detected except upon very close inspection with the eye. At nighttime the fabric was brightly retro-reflective, providing 2.5 candella of reflected light per square meter of treated surface per lux of incident light.

CLAIMS

1. Coating composition useful for retroreflect-  
torizing fabrics comprising a) a coating vehicle that  
includes film-forming binder material and a volatile con-  
5 stituent, and b) transparent microspheres having a refrac-  
tive index between about 1.85 and 2 hemispherically coated  
with specular reflective means and dispersed in the coating  
vehicle; characterized in that the volatile constituent  
comprises at least 40 volume-percent of the coating vehicle,  
10 and the transparent microspheres account for about one-third  
or less of the volume of the nonvolatile portion of the  
composition.
2. Composition of claim 1 in which the binder  
material comprises an aqueous emulsion of acrylic-based  
15 polymer.
3. Composition of claim 1 or 2 in which the  
microspheres account for about one-fifth or less of the  
volume of the nonvolatile portion of the composition.
4. Composition of claim 1, 2 or 3 in which the  
20 vehicle includes at least 70 volume-percent of a volatile  
constituent.
5. A method of forming a retroreflective treat-  
ment on fabric comprising spreading a composition of any  
of claims 1-4 across the fabric with barrier means.

1 / 1



# INTERNATIONAL SEARCH REPORT

International Application No

PCT/US80/01377

| <b>I. CLASSIFICATION OF SUBJECT MATTER</b> (If several classification symbols apply, indicate all) <sup>3</sup><br>According to International Patent Classification (IPC) or to both National Classification and IPC<br>Int. Cl. <sup>3</sup> G02B 5/128; C08K 3/00; B05D 1/42<br>US. Cl. 427/163; 260/29.6R  |   |                                     |  |   |  |   |  |     |   |   |     |   |   |     |   |  |     |
|---|---|-------------------------------------|--|---|--|---|--|-----|---|---|-----|---|---|-----|---|--|-----|
| <b>II. FIELDS SEARCHED</b><br><div style="text-align: right; font-size: small;">Minimum Documentation Searched <sup>4</sup></div> <table border="1" style="width: 100%; border-collapse: collapse;"> <tr> <th style="width: 20%; text-align: left; padding: 5px;">Classification System</th> <th style="text-align: left; padding: 5px;">Classification Symbols</th> </tr> <tr> <td style="padding: 5px;">U. S.</td> <td style="padding: 5px;">427/163; 260/29.6R</td> </tr> </table> <div style="text-align: center; font-size: x-small; margin-top: 10px;">           Documentation Searched other than Minimum Documentation<br/>           to the Extent that such Documents are Included in the Fields Searched <sup>5</sup> </div>  |   |                                     | Classification System  | Classification Symbols  | U. S.  | 427/163; 260/29.6R  |  |     |   |   |     |   |   |     |   |  |     |
| Classification System   | Classification Symbols  |                                     |  |   |  |   |  |     |   |   |     |   |   |     |   |  |     |
| U. S.   | 427/163; 260/29.6R  |                                     |  |   |  |   |  |     |   |   |     |   |   |     |   |  |     |
| <b>III. DOCUMENTS CONSIDERED TO BE RELEVANT</b> <sup>14</sup> <table border="1" style="width: 100%; border-collapse: collapse;"> <tr> <th style="width: 10%; text-align: left; padding: 5px;">Category *</th> <th style="text-align: left; padding: 5px;">Citation of Document, <sup>16</sup> with indication, where appropriate, of the relevant passages <sup>17</sup></th> <th style="width: 10%; text-align: left; padding: 5px;">Relevant to Claim No. <sup>18</sup></th> </tr> <tr> <td style="text-align: center; vertical-align: top; padding: 5px;">A</td> <td style="padding: 5px;">US, A, 3,535,019 Published 20 October 1970<br/>Longlet et al.</td> <td style="text-align: center; vertical-align: top; padding: 5px;">1-5</td> </tr> <tr> <td style="text-align: center; vertical-align: top; padding: 5px;">A</td> <td style="padding: 5px;">US, A, 3,700,305 Published 24 October 1972<br/>Bingham</td> <td style="text-align: center; vertical-align: top; padding: 5px;">1-5</td> </tr> <tr> <td style="text-align: center; vertical-align: top; padding: 5px;">A</td> <td style="padding: 5px;">US, A, 4,103,060 Published 25 July 1978<br/>Bingham et al.</td> <td style="text-align: center; vertical-align: top; padding: 5px;">1-5</td> </tr> <tr> <td style="text-align: center; vertical-align: top; padding: 5px;">A</td> <td style="padding: 5px;">CA, A, 935,254 Published 16 October 1973<br/>Hogseth et al.</td> <td style="text-align: center; vertical-align: top; padding: 5px;">1-5</td> </tr> </table> |   |                                     | Category *   | Citation of Document, <sup>16</sup> with indication, where appropriate, of the relevant passages <sup>17</sup>  | Relevant to Claim No. <sup>18</sup>                          | A   | US, A, 3,535,019 Published 20 October 1970<br>Longlet et al. | 1-5 | A | US, A, 3,700,305 Published 24 October 1972<br>Bingham | 1-5 | A | US, A, 4,103,060 Published 25 July 1978<br>Bingham et al. | 1-5 | A | CA, A, 935,254 Published 16 October 1973<br>Hogseth et al. | 1-5 |
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| A   | US, A, 3,535,019 Published 20 October 1970<br>Longlet et al.  | 1-5                                 |  |   |  |   |  |     |   |   |     |   |   |     |   |  |     |
| A   | US, A, 3,700,305 Published 24 October 1972<br>Bingham   | 1-5                                 |  |   |  |   |  |     |   |   |     |   |   |     |   |  |     |
| A   | US, A, 4,103,060 Published 25 July 1978<br>Bingham et al.   | 1-5                                 |  |   |  |   |  |     |   |   |     |   |   |     |   |  |     |
| A   | CA, A, 935,254 Published 16 October 1973<br>Hogseth et al.  | 1-5                                 |  |   |  |   |  |     |   |   |     |   |   |     |   |  |     |
| <div style="font-size: x-small;">             * Special categories of cited documents: <sup>15</sup><br/>             "A" document defining the general state of the art<br/>             "E" earlier document but published on or after the international filing date<br/>             "L" document cited for special reason other than those referred to in the other categories<br/>             "O" document referring to an oral disclosure, use, exhibition or other means<br/>             "P" document published prior to the international filing date but on or after the priority date claimed<br/>             "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<br/>             "X" document of particular relevance           </div>  |   |                                     |  |   |  |   |  |     |   |   |     |   |   |     |   |  |     |
| <b>IV. CERTIFICATION</b> <table border="1" style="width: 100%; border-collapse: collapse;"> <tr> <td style="width: 50%; padding: 5px;">           Date of the Actual Completion of the International Search <sup>1</sup><br/><br/>           11 FEBRUARY 1981         </td> <td style="width: 50%; padding: 5px;">           Date of Mailing of this International Search Report <sup>2</sup><br/><br/> <div style="text-align: center; font-size: large; font-weight: bold;">10 MAR 1981</div> </td> </tr> <tr> <td style="padding: 5px;">           International Searching Authority <sup>1</sup><br/><br/>           ISA/US         </td> <td style="padding: 5px;">           Signature of Authorized Officer <sup>19</sup><br/> <div style="text-align: center;">              J. R. Hoffman :wag           </div> </td> </tr> </table>   |   |                                     | Date of the Actual Completion of the International Search <sup>1</sup><br><br>11 FEBRUARY 1981 | Date of Mailing of this International Search Report <sup>2</sup><br><br><div style="text-align: center; font-size: large; font-weight: bold;">10 MAR 1981</div> | International Searching Authority <sup>1</sup><br><br>ISA/US | Signature of Authorized Officer <sup>19</sup><br><div style="text-align: center;">              J. R. Hoffman :wag           </div> |  |     |   |   |     |   |   |     |   |  |     |
| Date of the Actual Completion of the International Search <sup>1</sup><br><br>11 FEBRUARY 1981  | Date of Mailing of this International Search Report <sup>2</sup><br><br><div style="text-align: center; font-size: large; font-weight: bold;">10 MAR 1981</div> |                                     |  |   |  |   |  |     |   |   |     |   |   |     |   |  |     |
| International Searching Authority <sup>1</sup><br><br>ISA/US  | Signature of Authorized Officer <sup>19</sup><br><div style="text-align: center;">              J. R. Hoffman :wag           </div>                             |                                     |  |   |  |   |  |     |   |   |     |   |   |     |   |  |     |