

[72] Inventors **Robert W. Lenz**
Amherst;
Leo Barish, Sharon; Virginia L. Lyons,
Arlington, all of Mass.
[21] Appl. No. **718,020**
[22] Filed **Apr. 1, 1968**
[45] Patented **Nov. 16, 1971**
[73] Assignee **Allied Chemical Corporation**
New York, N.Y.

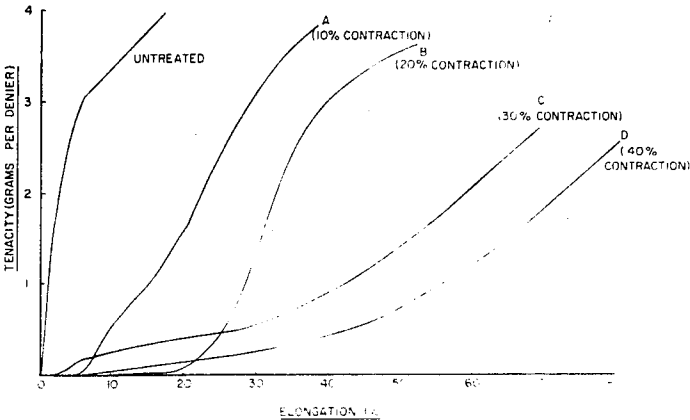
[54] **PROCESS FOR CHEMICALLY TEXTURING**
POLYESTER FIBERS
15 Claims, 4 Drawing Figs.

[52] **U.S. Cl.**..... **8/130.1,**
8/114.5, 8/115.5
[51] **Int. Cl.**..... **D06m 13/08**
[50] **Field of Search**..... **8/130.1,**
115.5, 116.4, 114.5

[56] **References Cited**
UNITED STATES PATENTS
3,374,273 3/1968 Cunningham 260/593
3,129,053 4/1964 Castle..... 8/93
3,084,020 4/1963 Toosli..... 8/130.1
2,870,211 1/1959 Miller..... 260/593
2,897,042 7/1959 Heiks 8/130.1
Primary Examiner—George F. Lesmes
Assistant Examiner—B. Bettis
Attorney—Jay P. Friedenson

ABSTRACT: Process for chemically texturing fibers, particularly polyester fibers, by treatment of the fibers with treating agents in aqueous solution of perfluorinated or perfluorochlorinated ketone hydrates which cause differential swelling to occur between the skin and core of the fibers.

EFFECT OF 6FK 3H₂O TREATMENT ON THE SINGLE LOAD TO RUPTURE
PROPERTIES OF DACRON 70-14-RQ2-56 MULTIFILAMENT POLYESTER FIBER



EFFECT OF $6FK \cdot 3H_2O$ TREATMENT ON THE SINGLE LOAD TO RUPTURE
 PROPERTIES OF DACRON 70-14-R02-56 MULTIFILAMENT POLYESTER YARNS

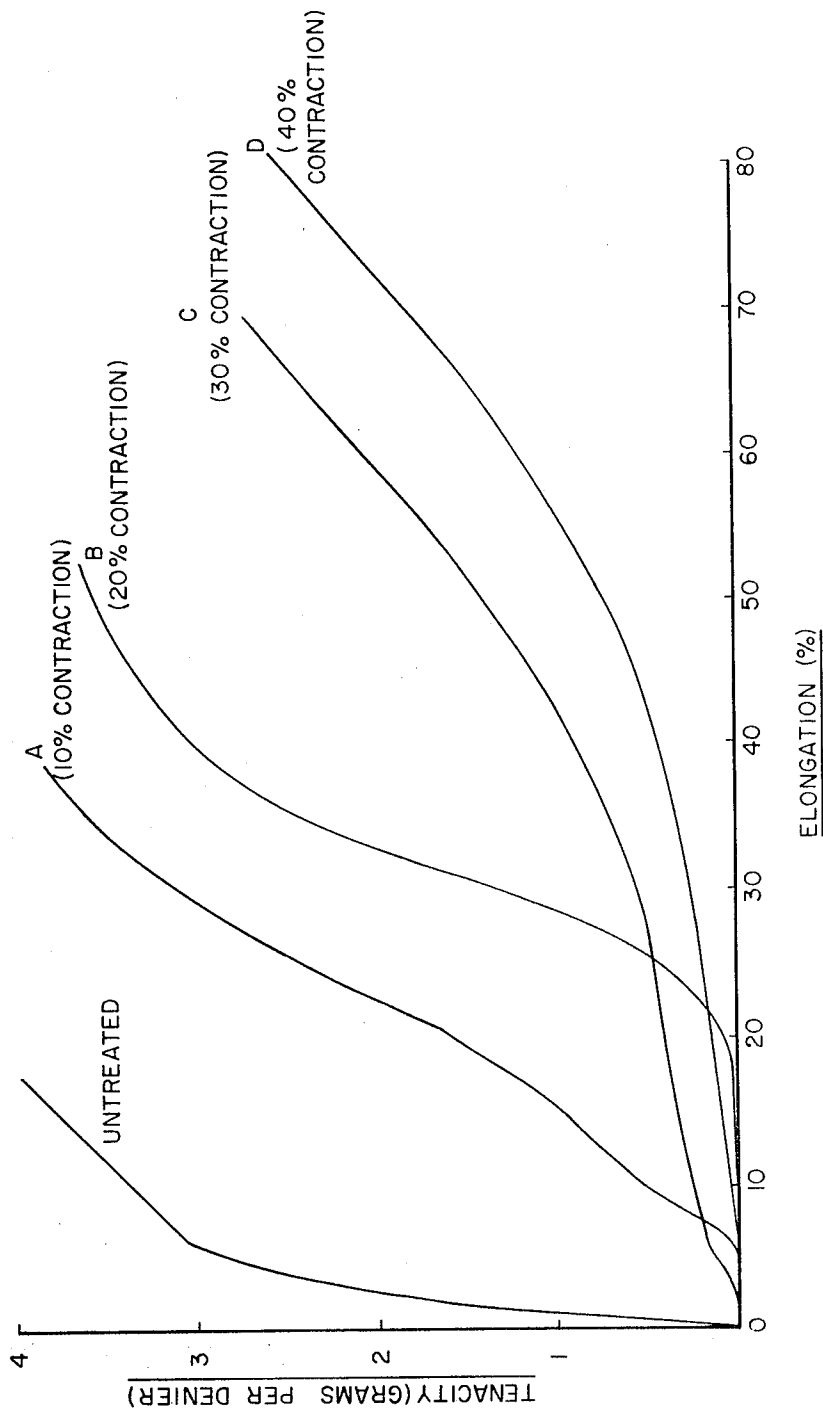


FIG.1

INVENTORS:
 ROBERT W. LENZ
 LEO BARISH
 VIRGINIA L. LYONS
 BY *Philip R. Riddick*
 ATTORNEY

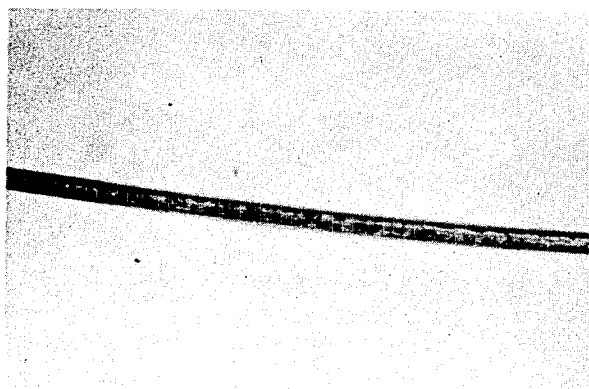


FIG. 2

DACRON FILAMENT IN 10:1 6FK · 3H₂O:WATER, 7 MINUTES

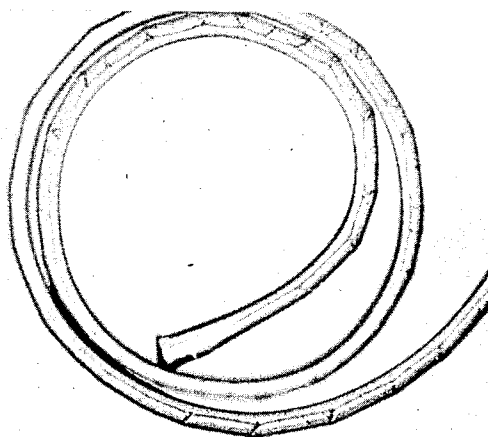


FIG. 3

DACRON FILAMENT IN 10:1 6FK · 3H₂O:WATER, 18 MINUTES

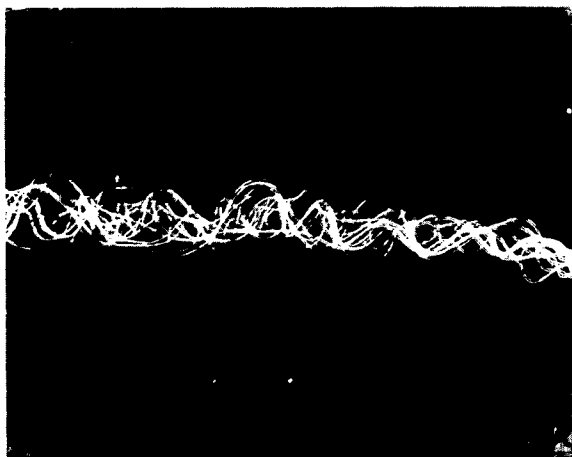


FIG. 4

70/14 DACRON YARN CRIMPED IN 10:1 6FK · 3H₂O SOLUTION

INVENTORS
ROBERT W. LENZ
LEO BARISH
BY VIRGINIA L. LYONS

Philip R. Davidson
ATTORNEY

PROCESS FOR CHEMICALLY TEXTURING POLYESTER FIBERS

BACKGROUND OF THE INVENTION

Fabrics made from synthetic continuous filament yarns are normally smooth, dense lustrous, and look and feel cold and metallic, making them unacceptable for many textile purposes. However, because of their relatively low cost the use of continuous filament yarns in fabrics is attractive. Thus synthetic continuous filament yarns are often textured to give them softer characteristics more like natural fibers. Such texturing is done by inserting into the filaments of the yarn a complexity of random permanent bends, loops or crimps. Use of such yarns produces fabrics which are less smooth and uniform than those made with untextured yarns but which are soft, lofty, dull, stretchy, warm and generally approach the look and feel of fabrics made from natural fibers.

Many techniques have been devised for the purposes of texturing yarn filaments or fibers in order to achieve the above results. However, such techniques have not been notably successful for texturing polyester fibers. For example, the so-called stuffer box method, which can be used to texture nylon and acrylic fibers, is not successful with polyester fibers.

One of the most successful methods of texturing polyester fibers is the so-called false twist method, which is applicable only with difficulty. In this method a continuous filament is twisted and the twist is set by the use of heat or steam. The twist is then removed from the filament, for example while winding the filament on a spool. When yarn and fabric are made from such treated filaments the filaments tend to return to their twisted configuration, thus giving the fabric the desired texture characteristics. However, in addition to being difficult to practice on polyesters, this method has the disadvantage that it imparts stretch but not bulk to the yarn. Also, false-twisted yarns contain a residual torque and must be used in plys to prevent distortion of the fabric.

For the above reasons it is highly desirable to develop new and improved processes for texturing polyester fibers.

SUMMARY OF THE INVENTION

It is an object of this invention to provide a new and improved method of texturing fibers, particularly polyester fibers.

It is also an object of this invention to provide a chemical method for texturing fibers, particularly polyester fibers.

It is a further object of this invention to provide a method for texturing polyester fibers which imparts bulk to yarn and which results in fibers which do not possess residual torque.

Other and further objects of this invention will be obvious to those skilled in the art from a reading of the following specification and claims.

The process of this invention comprises inducing fiber curling by treating the fibers, particularly polyester fibers, with treating agents which are solvents having a diffusion rate such that the solvent causes the skin or outer annular portion of the fiber to swell differentially with respect to the core of the fiber. Although this invention is particularly described with regard to its application to polyester fibers, it can be used on any natural or synthetic fiber which exhibits the above-mentioned differential swelling. The process of this invention more particularly comprises inducing such fiber curling in polyester fibers by treating them with an aqueous solution of a treating agent selected from the group consisting of perfluorinated ketone hydrates, perfluorochlorinated ketone hydrates and phenol. Perfluorinated acetone and perfluorochlorinated acetone hydrates have been found to be particularly useful in this process. Even more particularly, hexafluoroacetone trihydrate ($\text{CF}_3\text{COCF}_3 \cdot \text{H}_2\text{O}$), pentafluoromonochloroacetone trihydrate ($\text{CF}_3\text{CoCF}_2\text{Cl} \cdot 3\text{H}_2\text{O}$), and tetrafluorodichloroacetone dihydrate ($\text{CClF}_2\text{COCOCF}_2 \cdot 2\frac{1}{2}\text{H}_2\text{O}$) have proved to be particularly useful textile-treating agents, as has phenol.

Phenol is, of course, a known and widely available compound.

Hexafluoroacetone trihydrate and pentafluoromonochloroacetone trihydrate are known materials which are and the preparation of which are described in detail in copending, commonly assigned U.S. application No. 591,034, filed Oct. 31, 1966, by W. J. Cunningham and C. Woolf, the disclosure of which application is incorporated herein by reference. Briefly, hexafluoroacetone trihydrate and pentafluoromonochloroacetone trihydrate can be made by heating the corresponding monohydrate compound to volatilize therefrom constituents boiling below about 105°C . at atmospheric pressure.

Tetrafluorodichloroacetone dihydrate is a known material which is and the preparation of which is described in commonly assigned U.S. Pat. No. 2,870,211, the disclosure of which is incorporated herein by reference. Briefly, tetrafluorodichloroacetone dihydrate can be made by heating a liquor containing tetrafluorodichloroacetone and water to distill therefrom volatile constituents boiling below about 106°C ., heating the remaining solution to vaporize same, and condensing the vapors.

In treatment according to this invention, the treating solution can be applied to the fibers by dipping, spraying, vapor phase swelling or any other contact technique. When it is desired to cease treatment, the treating solution can be removed from the fibers by quenching and washing with means which will stop the differential swelling, such as water.

Applicants have found that the critical factors in such treatment are the rate of diffusion of the treating agent into the fiber, the concentration of treating agent in the aqueous solution, the temperature of the solution, the length of time during which the fibers are exposed to the treating solution and the amount of stress-free contraction allowed to the fibers during treatment.

Applicants have found that the concentration of treating agent in the aqueous solution should be sufficient to cause the treated fibers to curl within a reasonable time, for example 30 minutes or less, but should also be insufficient to dissolve the fiber or to swell the fibers at too fast a rate to allow control of the extent of curling and uncurling. When ketones are used, concentration of ketone in solution will generally be between about 95 percent and about 75 percent, preferably between about 95 percent and about 80 percent, by volume. When aqueous phenol solutions are used as textile treating solutions the concentration of phenol in water should be between about 0-8 percent and about 85 to 100 percent by volume, since between 8 and 85 percent phenol concentration water and phenol form a two-phase system which is unsuitable for use as a treating solution.

Treating agents having a diffusion rate into the fibers of less than about 0.1 micron per second have been found to be acceptable, with a diffusion rate of less than about 0.05 microns per second being preferable and a rate of less than about 0.035 microns per second being particularly suitable.

Applicants have also found that, if treatment of the fibers is continued beyond the point at which they curl, after a relatively short time the fibers begin to uncurl and finally straighten out and the purpose of treatment is defeated. Thus treatment should be continued only until the desired degree of curling is achieved. In general, curling time should be less than about 30 minutes.

It was found that curling time was a function of treating agent concentration and also of temperature. It is difficult to generalize as to relationship between concentration and curling and uncurling times; however, it has been observed that as temperature increases the length of time between curling and uncurling decreases.

Applicants have further found that, in order to control curling so as to achieve the desired degree of curling, the fibers should be restrained in such a manner that they can undergo stress-free contraction in an amount in the range from about 10 to about 40 percent of their original length, preferably from about 15 to about 25 percent. Such restraint can be achieved, for example, by loosely winding the fibers to be treated on a spool, allowing the desired about 10 to about 40 percent slack in the winding.

Applicants have still further found that, after treating the fibers according to the process of this invention, further curling can be achieved by exposing the fibers to steam.

Although one skilled in the art can easily determine the optimum concentration of treating agent in the treating solution, the optimum treatment time, and the optimum amount of stress-free contraction by utilizing the above disclosure, specific examples of ranges of these values for several suitable treating agents are given below.

BRIEF DESCRIPTION OF THE DRAWINGS

FIG. 1 is a graph illustrating the improved properties of Dacron polyester fiber yarn (du Pont 70-14-R02-56 multifilament yarn) when treated with a 10:1 solution of hexafluoroacetone trihydrate in water. It will be apparent that considerable crimp has been effected by the treatment of the yarn. The yarn was treated for a period of about 9 minutes at room temperature.

FIGS. 2 and 3 illustrate the effect of hexafluoroacetone trihydrate treatment of a single filament of Dacron polyester fiber.

FIG. 4 illustrates the effect of hexafluoroacetone trihydrate treatment of a Dacron Polyester yarn (du Pont's 70/14).

DESCRIPTION OF THE PREFERRED EMBODIMENT

EXAMPLE 1

When hexafluoroacetone trihydrate was used as the treating agent on Dacron polyester fibers, applicants found that it should be diluted with water in the range of from about 8:1 to about 20:1 parts of treating agent per part of water. Above about 20:1 concentration curling and solution tended to occur at the same time, while below about 5:1 concentration no tendency to curl was observed after an exposure time as long as 130 minutes. The preferred range of concentration was found to be from about 12:1 to about 8:1 parts of treating agent per part of water.

Table 1 below gives curling and uncurling times for Dacron polyester fibers (du Pont's 70/14 Rotaset, Type 56) treated with various concentrations of hexafluoroacetone trihydrate in water.

TABLE 1

[Coiling time as a function of dilution of 6FK.3H₂O with water]

Volume dilution 6FK.3H ₂ O:H ₂ O	Curling time, minutes	Uncurling time, minutes	Comments
Undiluted.....	1½		Curling and solution occur at almost same time.
20:1.....	3¾	5¾	Begins to form gelatinous layer on fiber surface at 4½ min.
15:1.....	2½	15	
10:1.....	5	8	
8:1.....	30	39	
5:1.....			No curling in.

Table 2 below indicates the changes in fiber curling and uncurling times with increase in temperature for a 10:1 solution of hexafluoroacetone trihydrate in water using the same type of polyester fiber described in the previous paragraph.

Table 2

Temperature °C.	Curling Time	Uncurling Time
22	5 min.	8 min.
42	1	1½
52	¾	—(undetermined)
65	½	½

EXAMPLE 2

When pentafluoromonochloroacetone trihydrate was used as the treating agent on the Dacron polyester fibers described in example 1 applicants found that the concentration of treating agent in water should be in the range of from about 5:1 to about 20:1 parts of treating agent per part of water. The preferred range of concentration was found to be from about 20:1 to about 7:1. It was found that exposure time for curling should be within the range of about 3 minutes for a 20 to 1 dilution to about 5½ minutes for a 7:1 dilution.

EXAMPLE 3

When tetrafluorodichloroacetone dihydrate was used as treating agent on the Dacron polyester fibers described in example 1, applicants found that its concentration should be within the range from about 10:1 to about 3:1 parts of treating agent per part of water. The preferred range of concentration was found to be from about 5:1 to about 3:1. It was found that at 5 to 1 dilution curling time was about 3 minutes and at 3 to 1 dilution curling time was about 15½ minutes and curling time about 22 minutes.

EXAMPLE 4

When phenol was used as the treating agent in aqueous solution applied to the Dacron polyester fibers described in example 1 applicants found that the curling and uncurling times for various concentrations were as follows:

TABLE 3

Concentration of Phenol in aqueous Solution	Curling Time	Uncurling Time
8%	3 min.	6½ min.
85	2	3½
93.5	1	2½

Hexafluoroacetone trihydrate and phenol were found to be the preferred treating agents, since they exhibited little tendency to fibrillate the treated yarns even at the extremes of concentration.

Table 4 below indicates that texturing by treatment with ketone hydrates is applicable to many types of polyester fibers.

TABLE 4

[Curling behavior of polyester yarns in 6FK.3H₂O at 10:1 dilution with water]

Yarn type	Curling time, minutes	Uncurling time, minutes	Comments
Du Pont:			
70-14-R02-56.....	6	12½	
70-14-R02-55, bright.....	30-44	49	
70-34-R02-57.....	10-12	16	
70-34-R02-55.....	4¼-8	11½	Fibrillated after 10 minutes.
70-34-R02-56.....	17½-21	27	
70-44-0-62 dull.....	1¼	4½	
70-34-0-56 SD.....	1¾	2¾	
Fiber industries:			
70-15-0-6, bright.....	3¾	611	
70-36-0-25 SD.....	1½	5½	
70-15-1/4Z 6 SD.....	4	17	Loose curl.
Kodel, 1.5 denier staple.....	6¾	13½	
Beaunit, 220-50 Type 7, bright.....			No curling.

¹ In 20 minutes, but curled in 1¾ minutes and uncurled in 2¾ minutes in 20:1 solution.

While this invention has been described with particular reference to a specific embodiment it is to be understood that it is not to be limited thereto but is to be construed broadly and restricted solely by the scope of the appended claims.

We claim:

1. The process of curling polyester fibers which comprises contacting the fibers with an aqueous solution consisting essentially of from about 75 to 95 percent by volume of a treating agent selected from the group consisting of a perfluorinated ketone hydrate and a perfluorochlorinated ketone hydrate having a diffusion rate into the fibers of less than about 0.1 microns per second, at a temperature and for a period of time sufficient to cause differential swelling of the outer annular portions of the fibers with respect to the cores of the fibers, but not substantially longer than necessary to effect the desired degree of curling, while maintaining said fibers in a sufficiently slack condition to permit a stress-free contraction of from about 10 to 40 percent of the original fiber length.

2. The process of claim 1 wherein the aqueous solution consists essentially of hexafluoroacetone trihydrate diluted with water in the range of from about 8:1 to about 2::1 parts of hexafluoroacetone trihydrate per part of water.

3. The process of claim 2 wherein the aqueous solution contains from about 8:1 to about 12:1 parts of hexafluoroacetone trihydrate per part of water.

4. The process of claim 2 wherein the fibers are maintained in a sufficiently slack condition to permit a stress-free contraction of from about 15 to about 25 percent of the original fiber length.

5. The process of claim 2 wherein the curled fibers are subsequently treated by exposure to steam to effect further curling.

6. The process of claim 2 wherein the contact time is less than about 30 minutes.

7. The process of claim 1 wherein the aqueous solution consists essentially of pentafluoromonochloroacetone trihydrate diluted with water in the range of from about 5:1 to about 20:1

parts of pentafluoromonochloroacetone trihydrate per part of water.

8. The process of claim 7 wherein the aqueous solution contains from about 7:1 to about 20:1 parts of pentafluoromonochloroacetone trihydrate per part of water.

9. The process of claim 7 wherein the fibers are maintained in a sufficiently slack condition to permit a stress-free contraction of from about 15 to about 25 percent of the original fiber length.

10. The process of claim 7 wherein the curled fibers are subsequently treated by exposure to steam to effect further curling.

11. The process of claim 7 wherein the contact time is less than about 30 minutes.

12. The process of claim 1 wherein the aqueous solution consists essentially of tetrafluorodichloroacetone dihydrate diluted with water in the range of from about 3:1 to about 10:1 parts of tetrafluorodichloroacetone dihydrate per part of water.

13. The process of claim 12 wherein the aqueous solution consists essentially of tetrafluorodichloroacetone dihydrate diluted with water in the range of from about 3:1 to about 5:1 parts of tetrafluorodichloroacetone dihydrate per part of water.

14. The process of claim 12 wherein the fibers are maintained in a sufficiently slack condition to permit a stress-free contraction of from about 15 to about 25 percent of the original fiber length.

15. The process of claim 12 wherein the curled fibers are subsequently treated by exposure to steam to effect further curling.

* * * * *

35

40

45

50

55

60

65

70

75

UNITED STATES PATENT OFFICE
CERTIFICATE OF CORRECTION

Patent No. 3,620,666 Dated November 16, 1971

Inventor(s) Robert W. Lenz, Leo Barish, Virginia L. Lyons

It is certified that error appears in the above-identified patent and that said Letters Patent are hereby corrected as shown below:

Column 1, line 69, that part of the formula which reads
" $\cdot\text{H}_2\text{O}$ " should read -- $\cdot 3\text{H}_2\text{O}$ --.

Column 1, line 70, that part of the formula which reads
" CoCF_2Cl " should read -- COCF_2Cl --.

Column 4, line 17, the word "he" should be --the--.

Claim 2, line 4, "2.:1" should be -- 20:1 --.

Signed and sealed this 26th day of December 1972.

(SEAL)
Attest:

EDWARD M. FLETCHER, JR.
Attesting Officer

ROBERT GOTTSCHALK
Commissioner of Patents