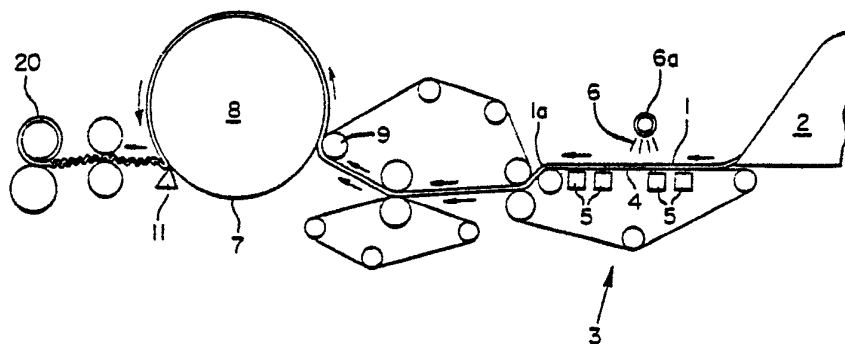




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(54) Title: METHOD FOR PRODUCING A HIGH QUALITY, WATER ABSORBENT, CELLULOSIC SHEET HAVING HIGH SURFACE-PERCEIVED SOFTNESS



(57) Abstract

Method for producing a cellulose sheet (20) capable of being used in high quality grades of tissue and towel, which exhibits outstanding water absorbency properties and a high degree of surface-perceived softness. The method includes the steps of forming a cellulose web (1), treating that web with a chemical bonding inhibitor (6) so that the pH of the web, after treatment, is not more than about 5.0, and then creping and drying the treated web. The sheet (20) produced by this method has an over-all quality factor and surface-perceived softness factor, respectively, of at least about 1.5.

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- 1 -

METHOD FOR PRODUCING A HIGH QUALITY,  
WATER ABSORBENT, CELLULOSIC  
SHEET HAVING HIGH SURFACE-PERCEIVED SOFTNESS

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TECHNICAL FIELD

This invention is directed to a method for producing a highly absorbent cellulosic sheet having excellent over-all quality and a high level of surface-perceived softness, respectively, which is capable of being employed in high softness grades of tissue and towel.

BACKGROUND ART

In the production of standard grades of paper on a conventional paper machine, cellulosic fibers and water are combined in a headbox to form an aqueous fiber slurry having a fiber consistency of from about 0.05% to 0.2% by weight, based on the total weight of that slurry. The wet web from the headbox is formed on a papermaking wire at a consistency of at least about 5% for purposes of mechanically removing water therefrom. This mechanical dewatering step increases the consistency of the web up to a level of about 45-50%. As the water is mechanically displaced, the fibers are moved into close proximity one with the other, and chemical bonds, generally described as "hydrogen bonds", are formed between the adjacent cellulosic fiber surfaces. The formation of these hydrogen bonds serves to strengthen the web, as measured, for example, by a substantial increase in physical properties such as an increase in the breaking length of the web.

The uncontrolled formation of substantial members of hydrogen bonds between the surfaces of adjacent fibers is detrimental to certain paper pro-



- 2 -

perties such as softness and absorbency. Specifically, various types of paper products such as certain grades of tissue and towel in which the sheet must be absorbent and soft to the touch cannot tolerate the unlimited formation of hydrogen bonds. These high softness and absorbency properties are not present in paper products made by the above described conventional papermaking techniques.

In an attempt to produce a sheet having the above described requisite degree of softness and absorbency, certain prior art methods incorporate a chemical "debonding agent" into the aqueous fiber slurry to inhibit the formation of hydrogen bonds. The incorporation of the debonder must, however, be accomplished without destroying the integrity of the web since it is necessary to form a sheet having the requisite strength properties for its intended use as a finished product of commerce, i.e., to produce a sheet having a high, over-all quality.

U. S. Patent 3,812,000 to Salvucci et al.; U. S. 3,844,880 to Meisel et al.; and U. S. 3,903,342 to Roberts et al., respectively, describe the addition of materials such as the above mentioned debonding agents to an aqueous slurry of cellulosic fibers to minimize hydrogen bonding in an attempt to increase softness. Typically, the above chemical debonding agents comprise surfactants such as those described in the Hervey et al. U. S. patents, Nos. 3,554,862; 3,554,863; and 3,395,708. The addition of a chemical debonding agent to the fiber slurry promotes the overall treatment of all fibers without regard to whether the treated fibers are located at the web surface, where hand-feel properties are measured, or in the interstices of the sheet. Therefore, certain patents such as U. S. 3,556,931 to Champaigne; U. S. 2,756,647



- 3 -

to Thompson; and U. S. 4,158,594 to Becker et al., disclose adding a debonding agent to a cellulosic web after web formation, but prior to drying. These latter patents describe softening the web to a certain extent, but do not provide a sheet having the requisite quality, absorbency, and surface-perceived softness level, as hereinafter defined.

#### DISCLOSURE OF INVENTION

10 In order to overcome the previously described problems associated with the prior art resulting from the addition to the headbox of chemical debonding agents, the subject invention contemplates treating the cellulose fibers in the formed web with a chemical bonding inhibitor having an acidic pH. More specifically, the cellulosic web immediately after formation preferably has an initial pH of not less than about 6.0, and preferably not less than about 6.5, and more preferably not less than about 7.0. At least one surface of the web is treated with the chemical bonding inhibitor, and the pH of the treated web immediately after the treatment step is not more than about 5.0, and preferably not more than about 4.5, and more preferably not more than about 4.0, and most preferably not more than about 3.0. The result of treating the web with a chemical bonding inhibitor under the pH conditions described above is an unexpected, dramatic improvement in quality, absorbency, and surface-perceived softness of the sheet products. The treatment of the cellulosic fibers with the acidic chemical bonding inhibitors does not occur until the consistency of the web, as measured immediately after the treatment step, is at least about 5% by weight, up to about 50% by weight.

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- 4 -

BRIEF DESCRIPTION OF THE DRAWING

FIGURE 1 is a schematic representation of a preferred method within the scope of this invention.

5 MODES FOR CARRYING OUT THE INVENTION

In accordance with the present invention, a method is provided for producing a highly absorbent, cellulosic sheet which exhibits excellent over-all quality and a high degree of surface-perceived soft-  
10 ness. A schematic drawing depicting a process configuration is set forth in FIGURE 1.

In the method of the present invention, an aqueous furnish including cellulose papermaking fibers is initially formed. The cellulosic fibers have under-  
15 gone some degree of lignin modification, such as at least partial chemical treatment, to produce materials such as chemimechanical pulp, semichemical pulp, chemical pulp, or mixtures thereof. Suitable materials from which the above cellulose fibers can be derived include  
20 the usual species of coniferous and deciduous pulpwood, the cellulosic components being preferably produced from coniferous pulpwood because of its greater fiber length.

The aqueous furnish is transported to a head-  
25 box 2 at a level sufficient to permit the formation of a substantially dry sheet upon completion of the hereinafter described dewatering and thermal drying steps, respectively, without requiring further drying thereof subsequent to creping. As a practical matter, however,  
30 the consistency of the aqueous furnish used in forming the subject wet web is desirably maintained at a level of from about 0.05% by weight, and more preferably from about 0.1% by weight, based on the total weight of cellulosic fibers in the aqueous furnish, up to a pre-  
35 ferred consistency of about 1.0% by weight, and more

- 5 -

preferably up to about 0.75% by weight.

A wet web 1 is then formed by deposition of the aqueous furnish onto a web forming means 3, typically a conventional papermaking system including a foraminous conveying means 4 such as a Fourdrinier wire, Stevens former, or the like.

Dewatering of the wet web is then provided prior to the thermal drying operation typically employing a nonthermal dewatering means 5. The nonthermal dewatering step is usually accomplished by various means for imparting mechanical compaction to the web 1 such as vacuum boxes, slot boxes, coating press rolls, or combinations thereof. For purposes of illustration of the method of this invention, the wet web 1 is dewatered by subjecting same to a series of vacuum boxes and/or slot boxes, as shown in FIGURE 1. Thereafter, the web is further dewatered by subjecting same to the compressive forces exerted by nonthermal dewatering means such as, for example, a pair of rolls, followed by a pressure roll coating with a thermal drying means.

The wet web 1 is carried by the foraminous conveying means 4 through the nonthermal dewatering means 5, where it is dewatered to a consistency of at least about 5%, preferably at least 10%, and more preferably at least 15%, up to a consistency of preferably about 50%, and more preferably up to about 45%, and most preferably up to about 35%. The cellulosic web formed, as described above, preferably has an initial pH of at least about 6 in order to minimize corrosion problems which can occur with respect to the foraminous conveying means, as well as other portions of the papermaking equipment if the pH per se is too low. More preferably, the initial pH of the web is maintained at a level, as previously described. The

- 6 -

wet web 1 prior to the thermal drying step is treated with an acidic chemical material 6, which inhibits the formation of papermaking bonds between adjacent cellulosic fibers. By treating the web 1 in this manner, a sheet 20 having the hereinafter defined, unexpected properties can be produced. The pH of web immediately after the treatment step, denoted "1a", is not more than about 5.0, and is preferably not more than about 4.5, and is more preferably not more than about 4.0, and is most preferably not more than about 3.0.

The consistency of the web immediately after the treatment step is preferably within the previously defined consistency parameters. Preferably, this chemical bonding inhibitor per se is a "chemical debonding agent". These materials are well-known in the prior art, and are preferably substantially cationic in nature. Examples of suitable chemical bonding inhibitor materials include Quaker 2001 (Quaker Chemical), Ceranine HCS (Sandoz), Leomin KP (Hoechst AG), and Amasoft-PM (American Color and Chemical).

The bonding inhibitor 6 employed for treatment of the web is provided at a treatment level which is sufficient to minimize the formation of the above described hydrogen bonds, but less than an amount which would cause significant runnability and sheet strength problems in the final commercial product. The amount of acid chemical bonding inhibitor 6 employed, on a 100% active basis, is preferably from about 0.5 pound per ton (1 kg/tonne) of cellulose pulp, up to about 15 pounds per ton (30 kg/tonne) of cellulose pulp. However, a more preferred addition of from about 1 pound (2 kg), up to about 10 pounds (20 kg), of chemical bonding inhibitor per ton (tonne) of cellulose pulp can be employed.

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- 7 -

Treatment of the wet web with the bonding inhibitor material can be accomplished by various means. For instance, the treatment step can comprise spraying, applying with a direct contact applicator means or by employing an applicator felt. However, the preferred method of application is by spraying the web such as by employing spray header 6a, at various points prior to thermal drying means 8 (see FIGURE 1). The adjustment of the pH of the web is localized at a given point of treatment, as opposed to having the web adjusted to the desired acidic pH level in the headbox. In a preferred method, the pH of the chemical bonding inhibitor 6 is adjusted to a pH level, prior to treatment therewith, which will facilitate the requisite minimum pH of the treated web at a level within the previously set forth pH limits. In a further preferred method, the pH of the web is first adjusted to a prescribed level and thereafter the sheet is treated with the chemical bonding inhibitor.

The acidity level of the bonding inhibitor material 6 can be adjusted employing numerous materials capable of performing that function. However, organic acids such as formic acid, acetic acid, propionic acid and benzoic acid, and inorganic acids such as hydrochloric acid, sulfuric acid, phosphoric acid and nitric acid, or salts thereof, are preferred. Of the above acidic materials, however, sulfuric acid is the most preferred.

The surface treated web 1a is then applied to the surface 7 of thermal drying means 8, preferably a thermal drying cylinder such as a Yankee drying cylinder, employing preferably an adhesive to supplement the adhesion process. Examples of typical adhesive compounds which may be used include carboxymethyl cellulose, polyvinyl alcohol, anionic starch, various

- 8 -

soluble natural polymers such as gums and the like, and synthetic resins such as polyamide resins, and the like. Adhesion of the treated web 1a to the cylinder surface 7 is preferably facilitated by the mechanical compressive action exerted thereon, generally using one or more press rolls 9, which form a nip in combination with thermal drying means 8 and which brings the web into more uniform contact with the thermal drying surface 7.

10 The web is then dried on the thermal drying surface preferably to a consistency of at least about 92%, and more preferably to a consistency of at least about 97%.

15 The creping means 11 then removes the dried, creped sheet 20 from the thermal drying surface, the creping action disrupting bonds between respective fibers and causing a softening effect to be imparted to the sheet. In general, the creping means is a doctor blade which crepes and removes the sheet from the thermal drying surface.

20 Sheet 20 has a high degree of bulk softness. "Bulk softness" is measured by conducting a Handle-O-Meter test (HOM) according to TAPPI T-498. The bulk softness (reciprocal of stiffness) of a given sheet is then calculated by dividing the HOM value by the square of the caliper of a given single sheet being tested, the quotient thereof being multiplied by  $10^5$ . For example, in single-ply tissue and towel applications, depending on the type of furnish employed, bulk softness, expressed as  $\text{HOM}/(\text{Caliper})^2 \times 10^5$  is preferably not more than about 1.0, and more preferably not more than about 0.8, and most preferably not more than about 0.6.

35 An important aspect of this invention is the ability of the subject method to produce sheets in an



- 9 -

extremely broad basis weight range. The basis weight of a given sheet is determined according to TAPPI test number T-410. The basis weight of the sheet produced by the subject method can, in general, range from about 5 pounds per 3,000 square feet (118.8 kg/3000<sup>2</sup>), and preferably from about 8 pounds per 3,000 square feet (190.1 kg per 3000<sup>2</sup>), up to about 50 pounds per 3,000 square feet (1180 kg/3000<sup>2</sup>), and preferably up to about 40 pounds per 3,000 square feet (950.4 kg per 3000<sup>2</sup>).

Each sheet must have enough structural integrity so that it will be capable of being manufactured without being damaged. A measure of the structural integrity of a sheet is breaking length. This procedure is run according to TAPPI T-222, except that instead of a 15-millimeter-wide sample strip, a one-inch (25.4 mm) wide strip is used. Average breaking length (BL<sub>avg</sub>) of a sheet is then calculated after the tensile strength of the sample in the machine direction (MD) and cross-machine direction (CMD), respectively, is determined, using the following equation:

$$BL_{avg} = \frac{685 (\text{tensile MD} + \text{tensile CMD})}{(2) \quad (\text{Basis Weight})}$$

In order to insure runnability of a web on papermaking equipment, the BL<sub>avg</sub> of preferably at least about 150 meters, and more preferably at least about 250 meters is provided. Furthermore, if the creped sheet has too high a BL<sub>avg</sub> value, it will be too harsh to the feel and, therefore, unacceptable to the consumer. Thus, it is preferred that the BL<sub>avg</sub> be not more than about 450 meters, and more preferably not greater than about 400 meters.

Another important sheet property is its ability to absorb water. The water absorbency parameter is expressed as the number of seconds it takes for a single sheet (4.5 inches by 4.5 inches) (11.43 cm by

- 10 -

11.43 cm) to absorb 0.1 cc of water, the test being described in TAPPI T-432.

If the method of the present invention is employed in a wet treatment step, as opposed to the prior art methods in which chemical bonding inhibitor per se is employed, an unexpected increase in the absorbency will result. Preferably, an increase in water absorbency of at least about 50%, and more preferably at least 100%, and most preferably at least 150% can be provided.

#### EXAMPLE 1

A series of three experiments (A-C) was conducted, employing a paper machine having a configuration set out in FIGURE 1. In the first experiment, 0.25% of a chemical bonding inhibitor material, namely, Quaker 2001, a cationic quaternary ammonium compound produced by Quaker Chemical Company, at a pH of 5.48 was sprayed on the formed web at a consistency of about 10%. In Experiments B and C, 25 and 50 ml of a 10% solution of sulfuric acid was added to 4 gallons (15.2 liters) of bonding inhibitor solution prior to spraying same on the wet web. This produced a pH of 2.78 and 2.48, respectively, immediately after treatment of the wet web in Experiments B and C. Sheets produced in Experiments A-C were tested, and the results re water absorbency and bulk softness are summarized in Table I.

TABLE I

	<u>A</u>	<u>B</u>	<u>C</u>
Water Absorbency (Sec.)	37.64	11.60	14.10
HOM/(Caliper) <sup>2</sup> x 10 <sup>5</sup>	1.02	0.55	0.53

The effect of employing an acidic bonding inhibitor treatment solution is clearly demonstrated by the above experimental comparison. Specifically, the



- 11 -

stiffness of the subject sheets (B and C) have been substantially reduced (softness increased) by employing the method of the subject invention to about one-half that of the sheet (A) employing a chemical bonding inhibitor per se. At the same time, the water absorbency of sheets B and C is dramatically increased, as compared to its higher pH counterpart, so that the sheets produced in Experiments B and C are 324% and 260%, respectively, more absorbent than the sheet produced in Experiment A.

The over-all quality and surface-perceived softness, respectively, of a cellulose sheet are subjectively determined to a great extent by the hand-feel as discerned by the ultimate consumer. Objective testing of the sheets in question are a measure of these properties and cannot totally act as a substitute for such a subjective determination. Therefore, the over-all quality and surface-perceived softness of sheets such as those produced by the method of this invention can best be subjectively determined by polling randomly selected respondents who have compared cellulosic sheets prepared by various methods, including the subject method. More specifically, four sheets prepared in substantially the same manner, except for pH of the chemical bonding inhibitor employed, were compared. Each of the four sheets was then rated by a group of ten respondents with respect to over-all quality and surface-perceived softness. An average over-all quality and surface-perceived softness for each of the sheets tested was then determined by adding the total points received for each of the sheets tested from a given respondent and dividing that total by ten, i.e., the total number of respondents. An over-all quality factor and surface-perceived softness factor for sheets produced by the subject invention were then

determined by dividing the average over-all quality, or the average surface-perceived softness, for the subject sheet by the comparable average value of the sheet, using a chemical bonding inhibitor per se. For example, a series of four experimental sheets, including the three cellulosic sheet products described in Example 1 (Nos. A-C), were tested, and an average over-all quality factor and an average surface-perceived softness factor for each were determined.

10

TABLE II

	<u>A</u>	<u>B</u>	<u>C</u>
Average Over-all Quality Factor	1.0	3.6	3.4
Average Surface-perceived Softness Factor	1.0	3.4	3.5

15

Accordingly, the sheets produced by the method of this invention have an over-all quality factor and a surface-perceived softness factor of preferably at least about 1.5, and more preferably at least about 2.0, and most preferably at least about 2.5.

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- 13 -

CLAIMS

1.

5 A method for producing a highly absorbent, cellulosic sheet having excellent quality and a high level of surface-perceived softness, respectively, which comprises

- a) forming a web of cellulosic fibers;
- b) treating the web with an acidic chemical material for inhibiting the formation of papermaking bonds between said cellulose fibers and said web, the consistency of the treated web immediately after said treatment step being from about 5% by weight, up to about 50% by weight, and the pH of the treated web immediately after the treatment step being not more than about 5.0;
- c) adhering the treated web to a thermal drying means, and drying said treated web thereon; and
- d) creping the dried web, the dried web having an over-all quality factor and a surface-perceived softness factor, respectively, of at least about 1.5.

2.

25 The method of claim 1, wherein at least one surface of the web is treated with the acidic chemical bonding inhibitor.

3.

30 The method of claim 1, wherein the initial pH of the web prior to treatment is not less than about 6.0.

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- 14 -

4.

The method of claim 3, wherein the initial pH is not more than about 6.5.

5.

5 The method of claim 4, wherein the initial pH is not less than about 7.0.

6.

10 The method of claim 1, wherein the pH immediately after the treatment step is not more than about 4.0.

7.

The method of claim 6, wherein the pH is not more than about 3.0.

8.

15 The method of claim 1, wherein the acidity level is adjusted in the treatment step by employing an inorganic acid.

9.

20 The method of claim 1, wherein the pH of the wet web is first adjusted to the prescribed level and, thereafter, the sheet is treated with a chemical bonding inhibitor material.

10.

25 The method of claim 1, wherein the pH of the chemical bonding inhibitor is adjusted to a level which will facilitate the requisite minimum pH level of the treated web at a pH of not greater than 5.0.

11.

30 The method of claim 1, wherein the acidity level is adjusted with an organic acid.

12.

35 The method of claim 1, wherein the respective over-all quality and surface-perceived softness factors of the sheet are at least about 2.0.





- 15 -

13.

The method of claim 1, wherein the treatment step comprises spraying said chemical bonding inhibitor onto the wet web.

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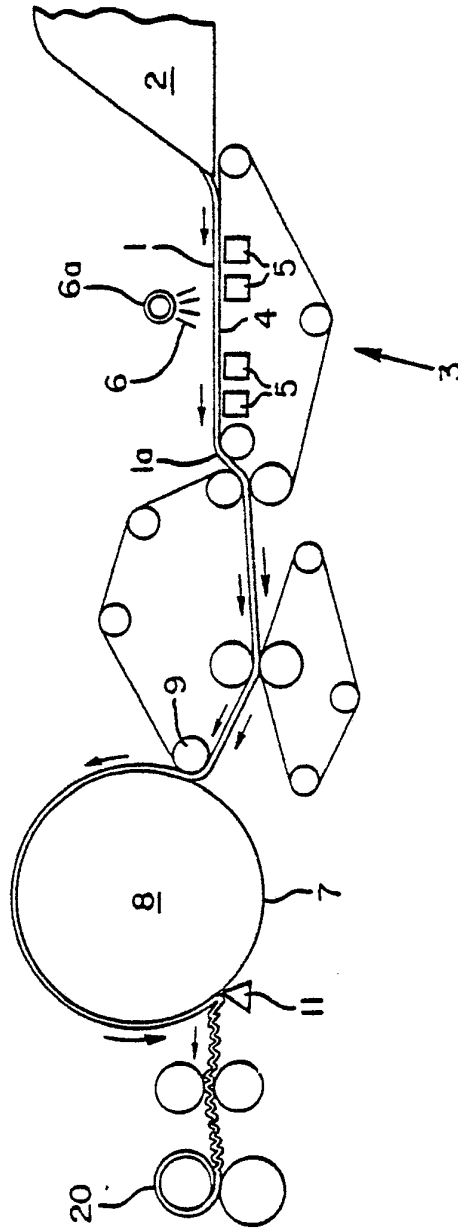
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30

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FIG. 1



# INTERNATIONAL SEARCH REPORT

International Application No **PCT/US81/01046**

<b>I. CLASSIFICATION OF SUBJECT MATTER</b> (If several classification symbols apply, indicate all) <sup>3</sup>		
According to International Patent Classification (IPC) or to both National Classification and IPC		
INT. CL. <b>D21H 5/24</b>		
U.S. CL. <b>162/111</b>		
<b>II. FIELDS SEARCHED</b>		
Minimum Documentation Searched <sup>4</sup>		
Classification System	Classification Symbols	
U.S.	162/111, 112, 158, 184, 185, 186	
Documentation Searched other than Minimum Documentation to the Extent that such Documents are included in the Fields Searched <sup>5</sup>		
<b>III. DOCUMENTS CONSIDERED TO BE RELEVANT</b> <sup>14</sup>		
Category <sup>6</sup>	Citation of Document, <sup>16</sup> with indication, where appropriate, of the relevant passages <sup>17</sup>	Relevant to Claim No. <sup>14</sup>
X	US,A, 2,940,890, PUBLISHED 14 JUNE 1960, BRAUN.	1-13
A	US,A, 2,683,088, PUBLISHED 06 JULY 1954, REYNOLDS.	1-13
X	US,A, 2,032,645, PUBLISHED 03 MARCH 1936, YOUTZ.	1-13
A	US,A, 1,986,291, PUBLISHED 01 JANUARY 1935, SCHUR.	1-13
X	DE,A, 2,314,060, PUBLISHED 10 OCTOBER 1974, HONSHU PAPER COMPANY LIMITED.	1-13
A	GB,A, 852,678, PUBLISHED 26 OCTOBER 1960, CANADIAN ANILINE AND EXTRACT COMPANY, LIMITED.	1-13
X	AU,A, 160293, PUBLISHED 25 JUNE 1953, PERSONAL PRODUCTS CORPORATION.	1-13
<p><sup>8</sup> Special categories of cited documents: <sup>15</sup></p> <p>"A" document defining the general state of the art</p> <p>"E" earlier document but published on or after the international filing date</p> <p>"L" document cited for special reason other than those referred to in the other categories</p> <p>"O" document referring to an oral disclosure, use, exhibition or other means</p> <p>"P" document published prior to the international filing date but on or after the priority date claimed</p> <p>"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</p> <p>"X" document of particular relevance</p>		
<b>IV. CERTIFICATION</b>		
Date of the Actual Completion of the International Search <sup>1</sup>	Date of Mailing of this International Search Report <sup>2</sup>	
OCT 22 1981	27 OCT 1981	
International Searching Authority <sup>1</sup>	Signature of Authorized Officer <sup>18</sup>	
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