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3,098,804

METAL TREATMENT

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This invention relates to the production of firmly adhering coatings on aluminum and aluminum alloys. More particularly, this invention relates to the production of firmly adhering galvanic coatings of metals, such as chromium and nickel, on aluminum and a variety of aluminum alloys. This application is a continuation-in-part of copending application Serial Number 828,451, filed July 21, 1959.

According to prior art practice, these metals have been plated on aluminum over a preliminary zinc coating produced by the well-known zincate immersion process. Usually brass or copper is plated over the thin zinc layer produced by this process, followed by plating with the nickel and/or chromium. Such process possesses certain inherent disadvantages. The zincate process and its attendant surface preparation is relatively complicated and not as easily handled as the preparation for plating of other common base metals such as steel or brass. Also, the copper, zinc and brass layers aggravate the corrosion of the plated aluminum product.

In order to overcome these disadvantages, it has been proposed to produce first an oxide coating by anodic treatment of the aluminum metal, followed by electroplating of the desired metal on the oxide coating. In the prior art, these oxide coatings were generally produced by anodizing in either an electrolyte of phosphoric acid alone or oxalic acid alone.

Anodizing in phosphoric acid or oxalic acid possesses certain inherent disadvantages. For example, oxalic acid anodizing requires the use of a post anodizing treatment to obtain adequate adhesion of the plated metal. Both phosphoric acid and oxalic acid anodizing are particularly sensitive to variations in aluminum alloy composition, and with many aluminum alloy products, it is difficult if not impossible to adherently plate metal particularly nickel and chromium, on an anodized surface produced in phosphoric acid alone or oxalic acid alone.

Additionally in the prior art it has not been possible to produce many plated articles from aluminum or aluminum alloys wherein the articles are characterized by multiple curved portions and accordingly require a desirable combination of formability coupled with good strength such as in automobile bumpers and wheel covers. Thus, while aluminum may have many advantages for such applications due to lightness in weight, corrosion resistance, etc., in the past, such articles have not been produced from aluminum. Accordingly, special alloys have been developed for such applications. However, satisfactorily adherent plates of nickel and chromium could not be produced on these alloys over anodized coatings produced in phosphoric acid alone or oxalic acid alone.

According to this invention, aluminum is anodized in an aqueous electrolyte consisting essentially of phosphoric

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acid and sulfuric acid, balance water, and thereafter a layer of metal such as nickel and/or chromium is electro-deposited on the anodized surface of said aluminum. By this method, firmly adherent metal layers can be provided on a wide variety of aluminum alloys.

In carrying out the method of this invention, the object to be treated may first be mechanically buffed and polished although this is not essential to the invention. The article is then cleaned in a suitable solution such as a mild or inhibited alkaline cleaner. The cleaned article may then be anodized. If a very smooth deposit is required on the finished article, the article to be plated may first be polished by conventional brightening treatments, e.g., chemical or electrochemical. On the other hand, where a matte or satin appearance is desired on the ultimate article, the aluminum metal may be subjected to a suitable etching treatment.

The clean aluminum is immersed as an anode in an aqueous electrolyte consisting essentially of from 5 to 45% by volume phosphoric acid, from 1 to 30% by volume sulfuric acid, balance water. An electric current is passed through the electrolyte for a time period ranging from 1 to 30 minutes, producing an oxide coating on the surface of the aluminum metal. A current density of from 12 to 120 a.s.f. is maintained, using a voltage of less than 40 volts. As used herein, the term "a.s.f." is an abbreviation of the expression "amperes per square foot." The electrolyte is maintained at a temperature ranging from 80 to 140° F. The oxide-coated metal is then plated with the desired metal such as chromium and nickel.

Where a chromium-plated aluminum metal article is to be subjected to severe corrosive environment or to severe bending stresses or impact, it is preferred first to electrodeposit a layer of nickel on the oxide-coated surface of the aluminum metal in a conventional nickel-plating bath. For some applications it has been found desirable to electrodeposit a plurality of layers of nickel on the oxide-coated surface of the aluminum metal, for example, two or three layers of nickel. Thereafter, a layer of chromium is electrodeposited on the nickel in a conventional chromium-plating bath. Where an article to be plated with chromium is not to be subjected to unusually severe corrosive environments or to unusually severe bending and impact stresses, the layer of chromium may be electrodeposited directly on the oxide-coated surface of the aluminum metal without electrodepositing an intermediate layer of nickel.

A series of aluminum alloys has been developed which exhibit the combination of good formability and strength necessary for applications such as automobile bumpers and wheel covers. These alloys are designated by the letters A thru E in Table I below wherein the percentages by weight of elements other than aluminum contained therein are given. In addition, a small but effective amount of titanium is added for grain refinement.

Such alloy compositions generally fall within the range of not more than 0.25% silicon, not more than 0.25% iron, 1.30 to 3.70% copper, not more than 0.25% manganese, 0.20 to 1.20% magnesium, not more than 0.30% zinc, a small but effective amount of titanium sufficient for

grain refinement, and not more than 0.05% each, 0.15% total of other elements, balance aluminum.

area of 6 sq. inches per sample were fabricated from a sheet of each of the commercial aluminum alloys design-

Table I

		Silicon	Iron	Copper	Man-ganese	Mag-nesium	Zinc	Others	
								Each	Total
A	Min			3.35		0.85			
	Max	0.25	0.25	3.65	0.10	1.15	0.10	0.05	0.15
B	Min			2.85		0.85			
	Max	0.25	0.25	3.15	0.10	1.15	0.10	0.05	0.15
C	Min			2.50		0.40			
	Max	0.25	0.25	3.00	0.10	0.60	0.10	0.05	0.15
D	Min			1.95		0.25			
	Max	0.25	0.25	2.25	0.10	0.45	0.10	0.05	0.15
E	Min			1.35		0.25			
	Max	0.25	0.25	1.65	0.10	0.45	0.10	0.05	0.15

Table II below gives typical results of strength and elongation tests performed on these alloys after treatment according to the conventional temper designations indicated in Table II below. The samples on which these tests were performed were 0.090 inch thick standard

noted by the aluminum association as 1100, 2024 and 6061. The percentages of elements other than aluminum in these alloys are indicated in Table III below. The figures in Table III indicate maximums unless a range is shown.

Table III

	Silicon	Iron	Copper	Manganese	Magne-sium	Chromium	Zinc	Tita-nium	Others	
									Each	Total
1100	1.0 Si-Fe		0.20	0.05			0.10		0.05	0.15
2024	0.50	0.50	3.8-4.9	0.3-0.9	1.2-1.8	0.10	0.25		0.05	0.15
6061	0.40-0.8	0.7	0.15-0.40	0.15	0.8-1.2	0.15-0.35	0.25	0.15	0.05	0.15

A.S.T.M. sheet samples for alloys A, B, and C and 0.032 inch thick standard A.S.T.M. sheet samples for alloys D and E. Variations in alloy composition within the above limits may produce variations from the typical properties indicated in Table II below.

Table II

		Tensile strength, p.s.i.	Yield strength, p.s.i.	Elong., percent in 2"
A	T4	55,000-58,000	36,000-40,000	20-24
	T4	49,500-52,200	28,100-31,900	24-29
B	T3	50,500-52,000	32,000-33,600	23-37
	T4	43,500-47,200	24,000-27,400	22-26
C	T3	44,400-45,500	27,700-28,500	22-25
	T4	35,800-37,000	16,900-19,000	17-24
D	T3	34,800-39,700	23,000-23,600	23-36
	T4	30,600-33,700	13,400-16,800	19-24
E	T3	30,900-33,900	13,600-16,500	19-25

The temper designations of Table II are the standard designations recommended by the aluminum association. The heat treatment employed on the above alloys may comprise heating the metal to a temperature of at least about 850° F. and maintaining the metal at that temperature for a period of time which may be as short as about 10 minutes. The metal is then quenched in a suitable means such as water. Following quenching, the metal with the temper designation of T3 is cold worked. The above alloys may be formed into products such as automobile bumpers or wheel covers. However, such articles cannot be adherently plated with metals such as nickel and/or chromium when anodized in conventional anodizing baths. When anodized in a phosphoric acid-sulfuric acid electrolyte they may be plated with nickel and/or chromium whereby an attractive plate characterized by excellent adherence may be produced. In plating articles such as automobile bumpers or wheel covers of the above alloys, best results have been achieved by anodizing in a sulfuric acid-phosphoric acid anodizing bath, plating three layers of nickel on the anodized metal followed by plating with chromium.

In an illustrative example of the process of this invention, 1" by 3" rectangular samples having a total surface

In these tests, all the samples were given the following treatment, it being understood that the conventional cold water rinsing operations after the various steps are not recited:

(1) Cleaned for 5 minutes in a mild inhibited alkaline cleaner of the carbonate-phosphate-silicate type contained in a 30-gallon rectangular stainless steel tank at a temperature of 160° F. to a water break-free surface.

(2) Etched by immersion for 5 minutes in a mild aqueous etching solution containing 25% by weight sulfuric acid, balance water contained in a 4-liter Pyrex beaker, at a temperature of 180° F.

(3) Anodized in a 20-liter Pyrex rectangular tank equipped with stirring devices and lead cathodes for 2 minutes with direct current in an aqueous phosphoric acid-sulfuric acid electrolyte, containing 300 mls./liter of aqueous 85% by weight H₃PO₄ solution and 30 mls./liter of aqueous 96% by weight H₂SO₄ solution, the balance of the electrolyte being distilled water. The temperature was maintained at 100° F. with a thermostated electric immersion heater. The current densities were maintained at 36 a.s.f. In general, the voltages necessary to maintain a given current density vary with the alloy, the cell geometry and the anodizing conditions and the required voltage varies as the anodizing progresses. In anodizing the samples employed in these tests the voltages required to maintain a constant current density of 36 a.s.f. ranged from about 2 to 30 volts.

(4) Electroplated as a cathode with chromium in a 20-liter Pyrex rectangular tank equipped with stirring devices and lead anodes for 30 minutes with a direct current in an aqueous electrolyte containing 250 grams per liter of CrO₃, 2.5 grams per liter of H₂SO₄, balance distilled water. The temperature was maintained at 60° F. by use of a cold water jacket surrounding the 20-liter Pyrex tank. The current densities were maintained at 200 a.s.f. The voltages required to maintain a constant current density of 200 a.s.f. ranged from 6 to 8 volts.

(5) The samples were rinsed briefly in distilled water, dried and buffed to a decorative brightness.

All samples had a lustrous, attractive chromium plate characterized by excellent adherence as shown by absence

of flaking off of the chromium when the samples were bent over a 3/4" diameter mandrel.

In another example of the practice of this invention, 1" by 3" rectangular samples having a total surface area of 6 square inches per sample were fabricated from sheets of the commercial aluminum alloys designated by the Aluminum Association as 1100, 2011, 2024, 3003, 6061, 6063, 7073, and the alloy designated in Table I above by the letter "C." The percentages of elements other than aluminum in the above commercial alloys are indicated in Table IV below. The figures in Table IV indicate maximums unless a range is shown.

Table IV

Alloy	Silicon	Iron	Copper	Manganese	Magnesium	Chromium	Zinc	Titanium	Others	
									Each	Total
1100--	1.0 Si+Fe		0.20	0.05			0.10		0.05	0.15
2011--	0.4	0.7	5.0-6.0				0.30		0.05	0.15
2024--	0.5	0.5	3.8-4.9	0.30-0.9	1.2-1.8	0.10	0.25		0.05	0.15
3003--	0.6	0.7	0.20	1.0-1.5			0.10		0.05	0.15
6061--	0.40-0.8	0.7	0.15-0.4	0.15	0.8-1.2	0.15-0.35	0.25		0.05	0.15
6063--	0.20-0.6	0.35	0.10	0.10	0.45-0.9	0.10	0.10	0.10	0.05	0.15
7075--	0.5-0.7	0.7	1.2-2.0	0.30	2.1-2.9	0.18-0.40	5.1-6.1	0.20	0.05	0.15

In these tests the samples were given the following treatment, it being understood that the conventional cold water rinsing operations after the various steps are not recited:

(1) Cleaned for 5 minutes in a mild inhibited alkaline cleaner of the carbonate-phosphate-silicate type contained in a 30-gallon rectangular stainless steel tank at a temperature of 160° F. to a water break-free surface.

(2) Anodized in a 20-liter Pyrex rectangular tank equipped with an air agitation device and lead cathodes with direct current in an aqueous electrolyte containing phosphoric acid and sulfuric acid or phosphoric acid alone or sulfuric acid alone in the amounts indicated in Table V below. These amounts are in terms of the percentages of aqueous 85% by weight H₃PO₄ solution and aqueous 96% by weight H₂SO₄ solution in the electrolyte, the balance of the electrolyte being water. The anodizing time, electrolyte temperature, current densities and the voltages required to maintain such current densities are given in Table V below:

Table V

Sample No.	Alloy	Anodizing conditions			Temp., ° F.	Current density, a.s.f.	Volt.
		Anodizing time, min.	Percent H ₃ PO ₄	Percent H ₂ SO ₄			
1-----	1100-----	5	15	3	130	48	15
2-----	2011-T4-----	10	10	8.3	130	36	15
3-----	2011-T4-----	2	10	8	130	54	10
4-----	2011-T4-----	5	15	15	90	48	15
5-----	2011-T4-----	5	15	25	110	96	10
6-----	2011-T4-----	4	40	15	90	72	15
7-----	2024-T3-----	10	10	8.3	130	36	15
8-----	2024-T3-----	10	10	8.3	130	36	15
9-----	3003-H14-----	10	15	3	90	12	25
10-----	3003-H14-----	5	15	3	130	48	15
11-----	6061-T6-----	5	15	3	130	48	15
12-----	6063-T5-----	5	15	3	130	48	15
13-----	7075-T6-----	10	10	8.3	130	36	15
14-----	C-T4-----	10	5	10	100	40	18
15-----	C-T4-----	2	5	10	130	72	16
16-----	C-T4-----	2	10	8	130	40	10
17-----	C-T4-----	5	10	10	90	48	21
18-----	C-T4-----	5	10	10	130	48	13
19-----	C-T4-----	5	10	15	130	84	10
20-----	C-T4-----	5	15	15	90	48	15
21-----	C-T4-----	2	15	15	130	96	8
22-----	C-T4-----	5	15	25	110	96	10
23-----	C-T4-----	4	40	15	90	60	15
24-----	C-T4-----	4	40	15	110	72	10
25-----	C-T4-----	5	0	15	90	60	15
26-----	C-T4-----	5	0	15	130	48	7
27-----	C-T4-----	5	0	25	90	96	13
28-----	C-T4-----	5	0	25	130	48	1
29-----	C-T4-----	5	15	0	90	21	30
30-----	C-T4-----	5	15	0	110	72	10
31-----	C-T4-----	5	15	0	130	72	10
32-----	C-T4-----	5	25	0	90	84	15
33-----	C-T4-----	5	25	0	110	84	15
34-----	C-T4-----	5	40	0	90	24	25
35-----	C-T4-----	5	40	0	110	84	18
36-----	C-T4-----	5	40	0	110	84	18

(3) All samples were electroplated as a cathode with nickel in a 20-liter Pyrex rectangular tank equipped with a cathode rod agitating device and nickel anodes for 60 minutes with a direct current in an aqueous electrolyte having a pH of between 3 and 4, containing 12 ounces per gallon of NiSO₄·7H₂O, 27 ounces per gallon, NiCl₂·6H₂O, 5 ounces per gallon of H₃BO₃, and 1/2% by volume of a wetting agent, balance water. The temperature of the electrolyte was maintained at 135° F. by means of an electric immersion heater. The current densities were maintained at 40 a.s.f. and the voltages required to main-

tain a constant current density of 40 a.s.f. ranged from 2 to 4 volts.

(4) Samples 2, 7, 8, 13 and 14 were additionally electroplated as a cathode with chromium in a 20-liter Pyrex rectangular tank equipped with mechanical stirring and lead anodes for 5 minutes with a direct current in an aqueous electrolyte containing 33 ounces per gallon of CrO₃, 0.33 ounce per gallon of H₂SO₄, balance water. The temperature of the electrolyte was maintained at 100° F. by means of an electric immersion heater. The current densities were maintained at 200 a.s.f. and the voltages required to maintain such current densities ranged from 5 to 8 volts. The remaining samples were not plated with chromium.

(5) The samples were rinsed briefly in distilled water, dried and buffed to a decorative brightness. Chromium plating of exceptional adherence was produced on samples 2, 7, 8, 13 and 14. A nickel plating of exceptional adherence was produced on samples 1, 3-6, 9-12 and 15 to 25. No peeling whatsoever was observed on any of the samples 1 through 25 even after severe grinding or bending. All of the samples on which these adherent platings were produced, i.e., samples 1-25, were anodized in a phosphoric acid-sulfuric acid electrolyte embodying the principles of this invention as can be seen from Table V. Adherent platings could not be produced on the remaining samples which were anodized either in phosphoric acid alone or sulfuric acid alone. Samples 26 and 28 could not be plated at all while with the remainder of samples 26-36 the platings exhibited very poor adhesion.

In another example of the practice of this invention, a substantial number of automobile bumpers characterized by multiple transverse and longitudinal curved portions were fabricated from solution heat treated aluminum alloys in the T4 temper of the type designated by the letters B, C and D in Table I above. The percentages of elements other than aluminum are indicated in Table VI below.

Table VI

Alloy	Si	Fe	Cu	Mn	Mg	Zn	Ti
B-----	.05	.13	2.91		.96		.01
C-----	.13	.15	2.69	.01	.48	.02	.01
D-----	.11	.18	2.14	.05	.37	.03	.02

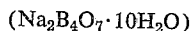
These bumpers were fabricated from sheets of the above aluminum alloys having thicknesses ranging from about 0.090 to 0.125 inch. These sheets were fabricated into bumpers in the conventional manner generally employed

in the fabrication of steel bumpers, i.e. the sheets were pickled in a conventional pickling bath, dried, blanked (i.e., cut to the desired shape for forming), polished with abrasive belts, subjected to a conventional treatment to make them more suitable for retaining a lubricant, rinsed, dried, the lubricant applied and the bumper then formed by stamping in a conventional press. After stamping, the bumper was subjected to one or more trimming operations to remove excess metal.

These bumpers were subjected to the following treatment, it being understood that the conventional water rinsing operations after the various steps are not recited:

(1) Buffed to a decorative brightness.

(2) Cleaned for 5 minutes by soaking in a solution containing 60 g./l. of sodium tetraborate



plus a small quantity of wetting agent, balance water contained in a 2500 gallon rectangular steel tank at a temperature of 170° F.

(3) Anodized in a 2500 gallon vinyl plastic-lined steel rectangular tank equipped with air agitation and lead cathodes for 5 minutes with direct current in an aqueous phosphoric acid-sulfuric acid electrolyte containing 15% by volume of aqueous 85% by weight H_3PO_4 solution and 15% by volume of aqueous 96% by weight H_2SO_4 solution, the balance of the electrolyte being water. The temperature was maintained at 100° F. with lead steam coils. The current density was maintained at 40 a.s.f. The voltages required to maintain a constant current density of 40 a.s.f. ranged from about 2 to 13 volts.

(4) Electroplated as a cathode with nickel in a 2500 gallon vinyl plastic-lined steel rectangular tank equipped with continuous filtration and external heat exchange and nickel anodes for 5 minutes with a direct current in an aqueous electrolyte having a pH of 3.7, containing 300 g./l. of $\text{NiSO}_4 \cdot 6\text{H}_2\text{O}$, 45 g./l. of $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$, 40 g./l. of H_3BO_3 , .5 g./l. of a wetting agent, balance water. The temperature of the electrolyte was maintained at 130° F. by means of an external heat exchanger. The current density was maintained at 40 a.s.f. and the voltages required to maintain a constant current density of 40 a.s.f. ranged from 4 to 7 volts.

(5) Transferred to a second nickel plating bath and electroplated as a cathode with nickel in a 10,000 gallon vinyl plastic-lined steel rectangular tank equipped with air agitation and nickel anodes for 16 minutes with a direct current in an aqueous electrolyte having a pH of 3.7 containing 300 g./l. of $\text{NiSO}_4 \cdot 6\text{H}_2\text{O}$, 45 g./l. of $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$, 40 g./l. of H_3BO_3 , .5 g./l. of wetting agent, .25 g./l. of coumarin (1,2-benzopyrone), .25 g./l. of formaldehyde (HCHO), balance water. The temperature of the electrolyte was maintained at 130° F. by means of a heat exchanger. The current density was maintained at 60 a.s.f. and the voltage required to maintain a constant current density of 60 a.s.f. ranged from 10 to 15 volts.

(6) Transferred directly without rinsing to a third nickel plating bath and electroplated as a cathode with nickel in a 10,000 gallon vinyl plastic-lined steel rectangular tank equipped with air agitation and nickel anodes for 8 minutes with a direct current in an aqueous electrolyte having a pH of 3.7 containing 300 g./l. of $\text{NiSO}_4 \cdot 6\text{H}_2\text{O}$, 45 g./l. of $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$, 45 g./l. of H_3BO_3 , .5 g./l. of wetting agent, .1 g./l. of coumarin (1,2-benzopyrone), 1 g./l. of saccharin (O-sulfobenzoyl imide) and .005 g./l. of tris (aminophenyl) methanol, balance water. The temperature of the electrolyte was maintained at 130° F. by means of a heat exchanger. The current density was maintained at 80 a.s.f. and the voltages re-

quired to maintain a constant current density of 80 a.s.f. ranged from 10-15 volts.

(7) Electroplated as a cathode with chromium in a 2500 gallon lead lined steel rectangular tank equipped with air agitation and lead anodes for 4 minutes with direct current in an aqueous electrolyte containing 250 g./l. of CrO_3 , 2.5 g./l. of H_2SO_4 , balance water. The temperature of the electrolyte was maintained at 105° F. by means of lead steam coils. The current density was maintained at 125 a.s.f. and the voltages required to maintain a constant current density of 125 a.s.f. ranged from 5-8 volts.

(8) Rinsed in cold water followed by a hot water rinse, and dried in air.

A lustrous chromium plating of exceptional adherence was produced. No peeling whatsoever was observed even after severe grinding or bending.

As used herein, the term "aluminum" is meant to cover high purity aluminum, commercial purity aluminum and aluminum alloys.

It will be understood that various changes, omissions and additions may be made to the invention without departing from the spirit and scope thereof as set forth in the appended claims.

What is claimed is:

1. A method of producing a firmly adherent coating of a metal selected from the group consisting of nickel and chromium

on an article of an aluminum alloy containing by weight not more than 0.25% silicon, not more than 0.25% iron, 1.30 to 3.70% copper, not more than 0.25% manganese, 0.20 to 1.20% magnesium, not more than 0.30% zinc, a small but effective amount of titanium sufficient for grain refinement, impurities in normal amounts, balance substantially aluminum,

said method comprising the steps of

immersing said aluminum alloy as an anode in an aqueous electrolyte consisting essentially of phosphoric acid and sulfuric acid, balance water, passing an electric current through said electrolyte for a time period of from 1 to 30 minutes, at current density of from 12 to 120 amperes per square foot and a voltage of less than 40 volts, thereby producing an oxide coating on the surface of said article

thereafter electrodepositing a layer of metal selected from the group consisting of nickel and chromium directly on said oxide-coated surface.

2. The method of claim 1 wherein said electrolyte contains from 5 to 45% phosphoric acid and from 1 to 30% sulfuric acid, balance water and wherein said electrolyte is maintained at a temperature ranging from 80 to 140° F.

3. The method of claim 1 wherein at least one layer of nickel is electrodeposited on said oxide-coated surface and thereafter a layer of chromium is electrodeposited on said nickel.

4. An adherently plated aluminum automobile bumper characterized by multiple curved portions, a tensile strength of from about 30,000 to 58,000 p.s.i., a yield strength of from about 13,000 to 40,000 p.s.i. and a percentage elongation in two inches of from about 17 to 29 produced by the method of claim 3.

5. The composite article produced by the method of claim 1.

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UNITED STATES PATENT OFFICE
CERTIFICATE OF CORRECTION

Patent No. 3,098,804

July 23, 1963

Henry J. Wittrock

It is hereby certified that error appears in the above numbered patent requiring correction and that the said Letters Patent should read as corrected below.

Column 3, Table II, in the column entitled "Elong., percent in 2" third entry thereof, for "23-37" read -- 23-27 --; same table, same column, seventh entry thereof, for "23-36" read -- 23-26 --.

Signed and sealed this 9th day of June 1964.

(SEAL)

Attest:

ERNEST W. SWIDER
Attesting Officer

EDWARD J. BRENNER
Commissioner of Patents