DEPARTMENT OF COMMERCE

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OF THE
BUREAU OF STANDARDS

S. W. STRATTON, DIRECTOR

No. 52

REGULATION OF ELECTROTYPING SOLUTIONS

[2d Edition]
Issued June 28, 1916

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REGULATION OF ELECTROTYPING SOLUTIONS

CONTENTS

I. INTRODUCTION

As noted in the previous edition of this circular, the Bureau of Standards is engaged in a study of some of the problems of electrotyping, especially those relating to the operation and regulation of the solutions. This work is being conducted in cooperation with a committee of the International Association of Electrotypers.

II. OUTLINE OF THE ELECTROTYPING PROCESS

While this circular is primarily of interest to electrotypers, a brief outline of the process may be desirable for the benefit of those not familiar with this industry.

The first step in the production of an electrotype plate consists in the preparation of an impression in wax of the type form. The molding wax usually consists of ozokerite, to which various substances have been added to produce the desired consistency. The molten wax is poured upon one side of a metallic plate or "case," either lead or copper. After taking the impression of the type form at a slightly elevated temperature by the use of suitable pressure, usually by means of a hydraulic press, the resultant "form" is "trimmed" and "built up" to produce the desired degree of relief in the finished plate.

The form is then coated with graphite, applied either by a wet or a dry process. After washing out the excess of graphite, the form is either introduced directly into the depositing bath, or, in some cases, is given a preliminary treatment (so-called "oxidizing") with copper sulphate and iron filings, whereby a thin film of copper is deposited upon the graphite. The baths are usually contained in lead-lined wooden tanks, with copper crossbars, from which the anodes and cathodes are suspended. Electrical connection to the graphited cathode surface is made by means of the suspending hook by either of two methods. In the first, known as the "case connection," the hook is in direct contact with the metallic case, portions of the wax being removed in order to bring the case and the graphite surface in contact, while the back of the case is insulated with wax. In the second method, known as the "face connection," the hook is in contact with a small copper plate inserted in the wax near the top of the form and in contact with the graphite surface. In the latter method the metallic case itself is not in the circuit, and there is less tendency for copper to deposit upon any accidentally exposed portions of the case.

After the copper is deposited to the desired thickness (usually 0.15 to 0.25 mm, or 0.006 to 0.010 inch) the form is taken from the bath, and the copper "shell" is loosened by means of hot water. After trimming, the back of the shell is treated with soldering fluid (usually zinc chloride) and coated with tin foil, after which it is laid face downward upon a heated pan. After the tin foil is melted upon the back of the shells, molten electrotype metal (usually containing from 3 to 4 per cent each of tin and antimony and from 92 to 94 per cent lead) is poured over them. The electrotype plates thus produced are cleaned, cut, and trimmed to the desired size and thickness and "finished" to a plane surface, which may be subsequently curved if desired.

In many cases, for halftone or other work in low relief, molding in thin sheet lead at very high pressures is practiced. The lead mold thus produced is usually covered with a very thin film of a wax or oil, and then lightly dusted with graphite to prevent the deposited metal from adhering too tenaciously. The subsequent steps are similar to those involved when wax molds are used.

For fine work, especially color halftones, or for plates requiring very severe service, nickel electrotypes (commonly called "steel" or "nickel steel") are frequently employed. In their preparation, a thin layer of nickel (usually about 0.025 mm or 0.001 inch) is first deposited upon the wax or lead mold, copper being then deposited back of the nickel, and the resultant nickel-copper shell being treated as above. The true "nickel electrotype" thus made should not be confused with a nickel-plated electrotype in which nickel is deposited upon the surface of a finished copper electrotype, thereby losing some of the detail of the original.

III. PROBABLE SCOPE OF THIS INVESTIGATION

The work thus far accomplished by the Bureau in this field has consisted principally of a study of the conditions and needs of the electrotyping industry by means of observations in commercial plants and systematic analyses of the solutions from a few typical plants. While it is yet too early to outline an exhaustive program for this work, the following phases of the work deserve attention:

1. The compilation and publication in convenient form of existing facts and data which have a bearing on the industry.

2. The determination and publication in tabular form of the useful physical constants of the solutions, e. g., the density, conductivity, etc.

3. The selection or devising of methods, preferably such as may be applied by those engaged in the industry, for determining the composition of the solutions and the properties (hardness, tensile strength, etc.) of the metallic deposits.

4. The study of the factors affecting the composition and physical properties of the deposits, and the selection of the limiting conditions (composition of solutions, temperature, current density, etc.) under which deposits of the desired properties may be

obtained with the greatest economy of time, attention, and electrical energy.

5. The effect of the preliminary operations (e. g., the composition and use of the wax and graphite in the preparation of electrotype molds) upon the character of the deposits.

6. The study of the purity of the anodes, salts, etc., now used in commercial work, with a view to recommending reasonable specifications for such materials.

The completion of the above suggested program will require the active services of a number of men for a considerable period, and will involve chemical, electrical, mechanical, and metallurgical studies. With the present available force and facilities, progress in this field, especially at first, may be slow, but efforts will be made to advance the work as rapidly as possible. While at present the investigation is being conducted primarily from the standpoint of electrotyping, much of the information obtained will doubtless also be applicable to the related industry of electroplating. As opportunity offers and demand for such information increases, the specific problems of electroplating may be studied.

IV. SCOPE OF THE PRESENT CIRCULAR

In order to render the information obtained in this work available as soon as possible, this circular will be revised from time to time. The principal new information in this edition is that relating to the study of copper electrotyping baths, from which certain specific recommendations have been made. There are also included in this edition tables giving the density of acid copper sulphate solutions and tables showing the weight and thickness of the copper deposited from such solutions by definite current densities in specified periods of time.

V. METHODS OF EXPRESSING CONDITIONS

1. METHODS OF EXPRESSING TEMPERATURE

The thermometer usually employed in scientific work is the centigrade thermometer, on the scale of which the melting point of ice is 0° and the boiling point of water (at a pressure of one atmosphere) is 100°, while on the Fahrenheit scale the corresponding points are 32° and 212°, respectively. In general, preference will be given to the centigrade scale, but in view of the prevalent use of the Fahrenheit scale the values on the latter will also be included. The conversion of temperatures from one scale to the other can be readily accomplished by the use of simple formulæ.

(*a*) To convert centigrade to Fahrenheit temperatures, multiply the degrees centigrade by 9/5 and add 32°.

Example: To convert 20° C to Fahrenheit—

$$\frac{9}{5} \times 20 + 32 = 68^\circ \text{ F}$$

(*b*) To convert Fahrenheit to centigrade temperatures, subtract 32° from the degrees Fahrenheit and multiply the remainder by 5/9.

Example: To convert 77° F to centigrade—

$$(77^\circ - 32^\circ)\,\frac{5}{9} = 45^\circ \times \frac{5}{9} = 25^\circ \text{ C}$$

The relation between the two scales may be readily seen from Tables 1 and 2 in the Appendix.

2. METHODS OF EXPRESSING DENSITY

For testing the density of solutions either a Baumé (B) or specific gravity (sp. gr.) hydrometer may be used. By the specific gravity of a liquid is meant the ratio of the weight of a given volume of the liquid to the weight of an equal volume of pure water, both the solution and the water being at a definitely stated temperature. Most of the hydrometers used for commercial work in this country are graduated for a temperature of 15.6° C (60° F) for both the liquid and water. Such hydrometers are said to be graduated for $\frac{60^\circ}{60^\circ}$ F. If, then, a certain solution has a specific gravity of 1.15, any definite volume of that solution at the above temperature weighs 1.15 times as much as does the same volume of water at that temperature. The specific gravity hydrometer is almost exclusively used for scientific work and is the basis of the tables of composition given in the Appendix. For the benefit of persons still using Baumé hydrometers tables (3 and 4) for the conversion of degrees Baumé to specific gravity and vice versa will be found in the Appendix of this circular.

3. METHODS OF EXPRESSING COMPOSITION

In view of the almost universal use of the metric system in scientific work, preference will be given in this circular and in reports on this work to that system. Since, however, the customary United States units are generally employed in electrotyping and similar commercial work, the values will also be expressed in such units. A complete list of the definitions and accurate tables of equivalents of the United States and metric units of weight and measure will be found in the Bureau of Standards Circular No. 47. The relations between the values most commonly used in this work will be found in Table 5 in the Appendix.

In this connection emphasis should be laid upon the great practical advantages connected with the use of the metric system throughout in such work, rather than the conversion of the results obtained by titration of the solutions (e. g., in grams per liter) into customary units (e. g., ounces per gallon) before adjusting the composition.

Among the numerous advantages of the metric system may be mentioned the following: (1) The fact that the units are related in multiples of 10 greatly simplifies calculations; (2) the simple relation between the units of capacity and volume (1 liter is equal to 1 cubic decimeter or 1000 cubic centimeters) facilitates the calculation of the capacity of tanks or vats; (3) the simple relation between the units of weight, volume, and capacity (1 kilogram is equal to the weight of a cuble decimeter or 1 liter of water at its maximum density) renders very convenient the calculation of the weight of a given volume of water or of any liquid of known specific gravity. These advantages are illustrated especially on pages 10 and 25, in which the corresponding calculations are made in metric and in customary units.

In order to actually use the metric system in regulating the solutions, the electrotyper should secure a meter rule (or a 60 cm four-fold rule) and either a set of metric weights to use on an ordinary scale or else a platform scale with metric graduations. Such scales can be secured with beams graduated in both metric and customary units. A copper measure, holding, say, 10 liters, will also be found convenient in removing portions of the solutions. The names of firms handling suitable metric apparatus will be furnished by this Bureau upon application. Tables and charts illustrating the use of the metric system, as well as a paper metric scale (which may be used to measure the capacity of the tanks), are also furnished free by this Bureau upon request.

From the approximate values in Table 5 the following simple rules may be derived:

(*a*) To obtain the approximate capacity of a rectangular tank in liters, multiply together the three dimensions (expressed in centimeters) and divide the product by 1000. For all practical purposes, it will be sufficiently accurate to express the capacity to the nearest 10 liters. It will probably be found convenient after determining the capacity of each tank to paint it upon the tank, thus avoiding remeasurement.

Example: Suppose a tank is 150 cm long, 75 cm wide, and 60 cm deep (up to the height of the solution). Then the tank has a capacity of 150 by 75 by 60 = 675 000 cubic centimeters, or 675 liters; or for practical purposes 680 liters.

(*b*) To obtain the approximate capacity of a rectangular tank in gallons divide the cubical contents (expressed in cubic inches) by 231.

Example: The same tank as in (*a*) if measured in customary units, would be 59 inches long, 30 inches wide, and 24 inches deep. It therefore has a capacity of 59 by 30 by 24 = 42 480 cubic inches, which, divided by 231 = 184 gallons, or for practical purposes 180 gallons.

(*c*) In order to facilitate the removal of a definite percentage of the solution in a tank, a vertical scale in centimeters (or inches) may be scratched upon the lead lining of the tank, starting from the bottom. If, then, the normal height of the solution is 60 cm (24 inches), and it is desired to remove 10 per cent of the solution, all that is necessary is to siphon out the solution till the level is lowered 6 cm (2.4 inches).

(*d*) To convert grams per liter (g/l) to ounces per gallon (oz./gal.) multiply by 0.134. Thus, 50 g/l equals 50 × 0.134 = 6.7 oz./gal.

(*e*) To convert grams per liter (g/l) to pounds per gallon (lb./gal.) multiply by 0.0083; or to pounds per 100 gallons, multiply by 0.83. Thus, 50 g/l is equal to 0.415 lb./gal., or to 41.5 lb./100 gal.

(*f*) To convert grams per liter to percentage by weight divide the grams per liter by 10 times the specific gravity [1] of the solution. Thus, for a solution with a specific gravity of 1.15, 50 g/l is equal to

$$\frac{50}{10 \times 1.15} = 4.35 \text{ per cent.}$$

(*g*) To convert amperes per square decimeter (amp/dm^2) to amperes per square foot (amp/sq. ft.) multiply by 9.29. Thus, 5 amp/dm^2 is equal to 5 × 9.29 = 46.45 amp/sq. ft.

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[1] Or, more strictly speaking, the density of the solution, i. e., the weight of 1 cc.

(*h*) To convert kilograms per square centimeter (kg/cm²)—e. g., for tensile strength—to pounds per square inch (lb./sq. in.), multiply by 14.22. Thus, 2800 kg/cm² = 2800 × 14.22 = 39 800 lb./sq. in.

VI. GENERAL PRINCIPLES OF ELECTRODEPOSITION

1. DEFINITION OF ELECTRICAL TERMS

(1) The **current** (or current strength) is the rate at which the electricity passes through the circuit. It is expressed in **amperes.**

The **ampere** is defined as that current which, under specified conditions, deposits a definite weight (1.118 mg) of silver in one second. In practice the current is measured in terms of amperes by means of a suitable ammeter.

(2) The **resistance** of the circuit to the passage of an electrical current, is expressed in **ohms.**

The **ohm** is defined as equal to the resistance at 0° C of a uniform column of mercury having a mass of 14.4521 grams and a length of 106.3 cm.

(3) The **electromotive force** (or **voltage,** or **potential difference**) represents the electrical pressure used to force the electricity through the circuit. It is expressed in **volts.**

The **volt** is that electromotive force which will produce a current of one ampere in an electrical circuit of which the resistance is one ohm. The volt may also be expressed in terms of the Weston normal cell, the electromotive force of which at 20° C is 1.0183 V. The electromotive force is usually measured in terms of volts by means of a suitable voltmeter.

The voltage measured at the dynamo is the available voltage and is called the **applied electromotive force,** while the voltage measured between two parts of a circuit—e. g., between an anode and cathode—is called the **potential difference** between these two points.

(4) **Relation Between Fundamental Units.**—The values of these electrical units have been so chosen that a very simple relation exists between them. This relation (Ohm's law) may be expressed:

$$\text{Current} = \frac{\text{Electromotive force}}{\text{Resistance}} \quad \text{or} \quad C = \frac{E}{R}$$

or in terms of the above units

$$\text{Amperes} = \frac{\text{Volts}}{\text{Ohms}}$$

It is possible, therefore, to double the current either by doubling the voltage used or by reducing the resistance to one-half the former value.

Example: If in a given electrical circuit an electromotive force of 10 volts is applied, and the total resistance of the circuit is 2 ohms, the resultant current will be

$$\frac{10 \text{ volts}}{2 \text{ ohms}} = 5 \text{ amperes.}$$

In case the voltage is doubled we have

$$\frac{20 \text{ volts}}{2 \text{ ohms}} = 10 \text{ amperes.}$$

If, on the other hand, the resistance is decreased to one-half, we also obtain twice the original current, i. e.,

$$\frac{10 \text{ volts}}{1 \text{ ohm}} = 10 \text{ amperes.}$$

(5) The **current density** is the current per unit area, and is usually expressed either in amperes per square decimeter or amperes per square foot. In any given case the average current density at the anode or cathode can be readily calculated by dividing the total current by the exposed area of the anode or cathode. If two baths are in series the current density in each bath is obtained by dividing the total current by the anode or cathode area in that bath.

Example: Suppose that 500 amperes is passed through a bath in which the area of the cathodes is 93 square decimeters, or 10 square feet. The cathode current density is then

$$\frac{500}{93} = 5.4 \text{ amp/dm.}^2$$

or

$$\frac{500}{10} = 50 \text{ amp/sq. ft.}$$

(6) The **quantity of electricity** that passes is equal to the current multiplied by the time. The scientific unit is the **coulomb,** which is the quantity of electricity that passes when a current of one ampere flows for one second. The common commercial unit is the **ampere hour** (equal to 3600 coulombs), i. e., the quantity of electricity which passes when a current of one ampere flows for one hour.

(7) The **power** expended in the passage of an electrical current is equal to the product of the applied electromotive force and the current. When the electromotive force is expressed in volts and the current in amperes, the power is obtained in **watts.**

A **watt** is the power involved when a current of one ampere is passed through a circuit by the application of 1 volt. The usual commercial unit is the **kilowatt,** equal to 1000 watts, or to 1.34 horsepower.

(8) The **energy** consumed in such operations is equal to the applied power multiplied by the time. The units usually employed are the **watt hour** and the **kilowatt hour,** which are equal to the energy consumed when a power of 1 watt or 1 kilowatt, respectively, is applied for one hour. The kilowatt hour is the usual basis of **payment** for electrical energy.

2. APPLICATION TO ELECTRODEPOSITION

In general, in the **electrodeposition** of metals (as in electroplating and electrotyping) a current of electricity is caused to pass through a solution between two metallic surfaces, dissolving metal from the one and depositing it upon the other.

(1) The solution, or **electrolyte,** contains, in addition to water, a compound (usually a salt) of the metal to be deposited, together with, in most cases, one or more substances added to improve the conductivity of the solution or the character of the deposit. Thus, in the regular acid copper bath, the principal constituent is copper sulphate (a salt formed by the action of sulphuric acid upon copper), while sulphuric acid is added to increase the conductivity of the solution and to improve the character of the deposited metal.

(2) **Electrodes.**—The metallic conductors through which the current enters and leaves the solution are known as **electrodes.**

The electrodes from which the metal is dissolved are called **anodes.**

The electrodes upon which the metal is deposited are the **cathodes.**

(3) **Current Efficiency.**—If a definite current is passed through a suitable solution for a specified time, under the **ideal** conditions, a definite weight of the metal will be dissolved from the anode and the same weight of metal will be deposited upon the cathode.

Under any given conditions the amount of metal deposited is proportional to the quantity of electricity that passes. The weight of metal deposited by 1 ampere hour (3600 coulombs) differs with different metals. From the acid copper sulphate bath 1 ampere hour deposits theoretically 1.186 grams (0.0418 ounce) of copper (therefore 24 ampere hours, or 1 ampere day, deposits 28.46 grams, or almost exactly 1 ounce of copper).

The ratio of the weight of metal actually dissolved from the anode, or deposited upon the cathode, to the theoretical weight, represents the **current efficiency** at the anode or cathode, respectively.

Example: Twenty-four ampere hours (or 1 ampere day) should deposit 28.46 grams (or almost exactly 1 ounce) of copper. If, in any case, such a current actually deposits only 27.90 grams of copper, the current efficiency is $\frac{27.90}{28.46}$ or 98 per cent.

(4) **Resistance.**—As above noted (p. 11), the current produced by a given voltage, or the voltage required to produce a given current, is determined by the resistance of the circuit. The lower the resistance of the circuit the lower the voltage, and also the power, required to produce a given current and to deposit a given weight of metal. In any such circuit the resistance is made up of two parts, the **external** resistance (of the leads and connections) and the **internal** resistance (the resistance of the solution itself).

The **external resistance** should be reduced to the practical minimum by the use of sufficiently large copper bars and rods for conductors and by securing the best possible contacts (permanent wherever feasible). An abnormally high resistance at any point or in any connection is indicated by undue heating at or near that point. It is generally considered good practice in such work to pass not more and preferably less than 125 amperes per square centimeter, or 800 amperes per square inch cross section of the copper rods. (In case copper tubes are used, the actual cross section of copper should be considered.)

The **internal resistance** of the bath may be decreased, (*a*) by addition of appropriate well conducting materials to the solution, (*b*) by raising the temperature of the solutions, (*c*) by decreasing the distance between the anodes and the cathodes, or (*d*) by increasing the size of the electrodes. In general, in the last case, however, only the total current and not the current density is thereby increased.

(5) **Arrangement of Baths.**—When two or more baths are so arranged that the anodes in one bath are directly connected to the cathodes in the next, the baths are said to be in **series.** In such a case the internal resistance of the circuit is equal to the sum of the internal resistances of the separate baths, and the current that flows through the two baths is the same. If the resistance of the two baths is equal, the voltages between the anodes and cathodes in each bath will be equal to each other and approximately equal to one-half the voltage derived from the dynamo. If in any such case the voltage on the two baths is not approximately equal, it is

evidence that the composition of the solutions, or the size or distance of the electrodes in the two baths is not the same. *If the cathode areas in the two baths are appreciably different, the current densities, and therefore the character of the deposits, will vary.*

When the baths are arranged independently—i. e., the anodes and cathodes in each are directly connected to the dynamo—they are in **parallel.** In such a case the voltage on each bath is approximately that of the dynamo, and the current that passes through each circuit is inversely proportional to the resistance of each.

Example: Suppose that two baths are connected in parallel to a dynamo producing a current of 3 volts. If the total resistance of one bath is 0.01 ohm and of the second bath is three times as great—i. e., 0.03 ohm—the current flowing through the first bath will be three times as great as that through the second.

(1) $3/0.01 = 300$ amperes.
(2) $3/0.03 = 100$ amperes.

(6) **Insulation.**—In order to avoid irregularities in operation, and losses of electrical energy, precautions should be taken to provide suitable insulation between the anode and cathode bars, and between them and the lead linings or other metallic connections. The strips of wood usually used on the edge of the tanks to support the electrode rods readily become impregnated with the solutions and permit leakage of electricity from anodes to cathodes or to the linings. For such purposes strips of nonpermeable insulating material, such as albarene stone, hard rubber, glass, porcelain, wood impregnated with wax, etc., are preferable. By the use of tanks constructed of stoneware or similar material, the possibility of electrical leakage is greatly reduced. *No metallic connection, other than the electrical conductors should exist between tanks or to the earth.* To avoid this, all pipes leading into the tanks should have a suitable insulating joint, the simplest form of which is a few inches of rubber hose.

VII. THE DEPOSITION OF COPPER FROM ACID SULPHATE SOLUTIONS

1. GENERAL CONSIDERATIONS

The actual application of the principles of electrodeposition can probably best be illustrated by consideration of one of the simplest plating baths, viz, the acid-copper sulphate bath, such as is very generally used in electrotyping work. We will, therefore, discuss in some detail the factors influencing the operation of this bath as determined from a recent investigation made by this Bureau at the plant of the Royal Electrotype Co., of Philadelphia. Details of these experiments will be published in a Technologic Paper of the Bureau of Standards. Announcement will be made when this publication is available for distribution.

2. BIBLIOGRAPHY

The following are the more important publications consulted in the preparation of this section. More detailed references will be found in these publications.

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3. FACTORS AFFECTING DEPOSITION

The principal factors which may influence the deposition of copper in the acid sulphate bath are (1) Composition of the solution; (2) temperature of the solution; (3) composition and physical properties of the anodes; (4) character and condition of the cathode surfaces; and (5) current density and agitation employed.

The relation of these factors to the properties of the deposited copper will be briefly considered. In this connection it is important to note that the desirable properties of copper for a given purpose (e. g., electrotyping) have not thus far been clearly defined. The need for such definition is clearly shown by the fact that different specimens of copper deposited under actual electrotyping conditions have been found to vary in tensile strength from 1400 to 2800 kg/cm^2 (20 000 to 50 000 lb./sq. in.), and in elongation from 4 to 35 per cent.

4. METHODS OF TESTING THE DEPOSITS

In the studies made by the Bureau upon copper deposits the tensile strength and elongation of a large number of samples have been determined.

The **tensile strength** determinations were made in a Schopper testing machine by recording the load in kilograms required to break strips of the copper of uniform width (1.5 cm, or 0.6 inch) and of measured thickness (usually about 0.2 mm, or 0.008 inch). From the load in kilograms required to break the copper specimen with a measured cross section the load which would be required to break a copper specimen with a cross section of 1 cm^2 or 1 square inch, respectively, was calculated. The tensile strength in kilograms per square centimeter or pounds per square inch thus computed was taken as the measure of the ultimate strength, and indirectly may probably be considered an index of the hardness of the metal.

The **elongation** was determined by measuring the distance after fracture between two reference marks which had been placed upon the copper strip before placing in the testing machine. The distance between these two reference marks originally was 5 cm, or approximately 2 inches, and the change in length in that gauged length was computed in per cent. The elongation was taken as the measure of the ductility of the metal.

It is recognized that a number of factors may influence the wearing quality (on the press) of an electrotype plate, e. g. (1) the hardness (which, according to the method of measuring it, may represent resistance to penetration or to abrasion); (2) the ultimate strength of the copper sheet; (3) the elastic limit, which may determine the resistance of the metal to permanent deformation; (4) the ductility, which is the property by which the metal is enabled to change its form beyond the limit of elasticity before failure takes place (of special interest in curved plates); (5) the crystalline structure itself, especially in fine halftone work; and (6) the composition and application of the "backing-up" metal. Studies are now being conducted to determine which of these factors have the most marked effect upon the durability of the plates. If possible, such experiments will be correlated with service tests. While thus far no actual service tests have been conducted to determine the wearing qualities of electrotype plates produced from copper of known properties, after conference with practical electrotypers the following tentative specifications for such copper have been proposed by this Bureau, viz, a tensile strength of 2500 to 2800 kg/cm^2 (35 000 to 40 000 lb./sq. in.) and an elongation of 20 to 30 per cent. In the following discussion the production of such copper will be considered as the object of the work.

5. COMPOSITION OF THE SOLUTIONS

(1) **Relation to the Properties of the Copper.**—The usual acid copper sulphate baths contain from 15 to 25 per cent of copper sulphate (blue vitriol or blue stone) and from 1 to 10 per cent of

sulphuric acid. That such a wide range of composition is in use confirms the generally accepted conclusion that within wide limits the composition of the solution in itself has very little effect upon the character of the deposited copper. Since, however, in the usual method of operation (maintenance of a constant voltage) the composition and consequent conductivity of the solution determines the current density, it is highly important that for satisfactory operation the solution be kept nearly uniform in composition. Moreover, since the better the conductivity of the solution the lower the voltage (and power) required to obtain a given current density, it is obvious that the best conducting solution that will produce the desired results should be used. This is the more important since in poorly conducting solutions the additional electrical energy is not only wasted, but it also causes undue heating of the solution. (See p. 26.) It may be said in general that (1) the solution should contain a sufficient amount of copper sulphate to insure the constant presence of copper sulphate in the solution adjacent to the cathodes, but not enough to permit its crystallization at the anode; and (2) it should contain considerable sulphuric acid to increase the conductivity. The possible amounts of these two constituents are, however, limited by the fact that the presence of sulphuric acid reduces the solubility of copper sulphate, as may be seen from Table 7 (Appendix). From this table it is evident that in order to maintain a high concentration of copper sulphate (e. g., 200 g/l, or 27 oz./gal.) it is impracticable to have present over 100 g/l (or 13.4 oz./gal.) of sulphuric acid.

Results of the tests above referred to have led to the following conclusions respecting the effect of composition of the solution under otherwise uniform conditions:

1. Addition of copper sulphate causes a slight increase in the tensile strength of the deposited copper.

2. At low temperature (25° C or 77° F) an increase in the sulphuric acid content increases the tensile strength, and at high temperature (40° C or 104° F) decreases the tensile strength.

It is accordingly recommended that the solution, operated at not over 30° C (86° F), should contain from 50 to 80 g/l (7 to 11 oz./gal.) of sulphuric acid, and from 250 to 200 g/l (34 to 27 oz./gal.) of copper sulphate, and should have a specific gravity at 25° C (77° F) of 1.17 to 1.18 (21 to 22° B).

(2) **Use of Addition Agents.**—For the purpose of increasing the permissible rate of deposition of the copper and improving the

character of the deposit (see p. 30) various substances are frequently added to such solutions in addition to the copper sulphate and sulphuric acid. The various substances suggested for this purpose may be divided into three classes; (*a*) colloidal substances, e. g., gelatine, glue, albumen, and gum arabic; (*b*) inorganic compounds such as nitric acid, potassium nitrate, potassium chlorate, zinc sulphate, ferric sulphate, alum, and tin salts; and (*c*) organic substances such as alcohol, glycerol, cresol, sugar, molasses, benzoic acid, and tannic acid. There is no sharp distinction between these classes, since some of those in (*b*) or (*c*) may give rise to the formation of colloids, either directly by hydrolysis, by decomposition, or by reaction with the copper compounds. In general, it may be said that such substances usually reduce the size of the crystals in the deposits, making it possible to use higher current densities and still obtain coherent deposits. Such deposits are usually harder and more brittle, but of greater tensile strength than the normal deposits. Sieverts and Wippelman have shown that the deposits of copper from solutions containing organic colloids, may contain from 0.5 to 4 per cent of organic matter. Under such conditions the colloids must be frequently replenished.

While this Bureau is not prepared to state that any or all of the above substances, when used with care and adequate supervision, may not be beneficial, the presence of such materials in the solution may demand frequent (and perhaps complicated) chemical analyses, if the solution is to be maintained in a uniformly satisfactory condition. Until an opportunity is secured to study the behavior of such baths over a considerable period, it is impossible to make any definite recommendations concerning them, or to say how far the general objection above expressed applies to any particular case. *In general, it should be emphasized that such addition agents, even if satisfactory, can only affect the permissible rate of deposition and the character of the deposit; and at most, to only a slight extent influence the current or power required to deposit a given weight of copper.*

(3) **Changes Occurring in the Composition of the Solutions.**— Since the commercial copper sulphate and sulphuric acid, as well as the copper anodes, are usually fairly pure, and since, moreover, a small amount of the solution always adheres to the cathodes and is subsequently replaced, there is no marked tendency for impurities to accumulate in the solution. Under the ideal con-

ditions the same amount of copper should dissolve at the anodes as is deposited upon the cathodes, and the solution should remain practically constant in composition. In practice, however, it is usually found that the content of copper sulphate increases while that of free acid decreases; which change is indicated by an increase in the density of the solutions. This change is explained by the fact that in such cases more copper is dissolved from the anodes[2] than is deposited upon the cathodes. This effect is especially marked when low anode current densities are employed, and can therefore be remedied or at least reduced by the use of smaller anode surface. Some experiments in this connection in electrotyping baths have shown that by using narrow rectangular copper anodes properly spaced, instead of large anode plates, the consumption of acid may be reduced and incidentally there is less residual scrap copper. In general, for such copper baths, it is probably not necessary or desirable to have the anode surface greater than that of the cathodes.

Other factors may also cause changes in the composition of the solutions. Thus, the evaporation which always takes place will in itself cause an increase in both density and acidity if it is not corrected by the occasional addition of water to constant level. When the cases (cathodes) are removed from the baths a certain amount of the solution always adheres to them. The mechanical losses thus produced cause a decrease in both density and acidity if, as is customary, the bath is maintained at constant level by the occasional addition of water. Since in practice, however, the solutions usually increase in density and decrease in acidity, it is evident that the chemical changes discussed in the last paragraph more than balance the mechanical losses. In any event, it is desirable, at intervals, to determine and adjust the composition of the solution to that desired. Even though, as previously pointed out, the composition of the solution, under otherwise uniform conditions, does not in itself have a marked influence upon the character of the deposits, *it is absolutely essential that the solution be kept nearly uniform in composition,* since changes in it influence the current density produced with a given voltage. Adjustment of the solution usually consists in (*a*) the replacement of a portion of the solution with water to reduce the content of copper sulphate, and (*b*) the addition of the requisite

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[2] Owing to the formation of cuprous ions, as pointed out by Foerster, Zs. Elektrochem., 3, p. 480; 1897.

amount of sulphuric acid. Observations on electrotyping baths indicate that to maintain a constant composition of such solutions *it is seldom, if ever, necessary to add any copper sulphate.*

(4) **Method of Regulating the Composition.**[3]—(*a*) DETERMINATION OF THE DENSITY.—Before testing the solutions they should always be adjusted to the normal level of the vats by the addition of water if necessary, and thoroughly mixed. The hydrometer should be read always in the same way, preferably by floating it in a glass jar or cylinder containing the solution. By placing the eye slightly below the level of the solution, and then gradually raising the eye, the surface, first seen as an ellipse, becomes a straight line, the intersection of which with the hydrometer scale should be taken as the reading of the hydrometer. For practical purposes with colored solutions, and especially for comparison, the readings will usually be sufficiently accurate if made by observing the hydrometer scale above the surface of the liquid. The density should always be taken at approximately the same temperature, preferably that for which the hydrometer is graduated.

(*b*) DETERMINATION OF ACIDITY.—*Principle.*—The amount of free sulphuric acid in the electrolyte is determined by measuring the volume of an alkali solution of known strength which is required to neutralize the acid present in a measured volume of the copper sulphate solution. An indicator (methyl orange) is added to the solution in order to show, by its change in color, when all the acid is neutralized by the alkali. The method as applied to such solutions is not original, having been published by Wogrinz [4] in 1913. Even before that time it was used in a few electrotyping establishments in this country.[5]

Apparatus.—The only essential pieces of apparatus for making this test are a pipette to measure the copper sulphate solution, a burette to measure the standard alkali, and a few bottles. Fig. 1 illustrates a form of apparatus that has been found convenient in this work. Many other forms of reservoir burettes will be found equally suitable. This apparatus and also the necessary solutions can doubtless be secured for a moderate charge from any local chemist or chemical supply house.

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[3] The method of adjustment to be described, based upon some tables recently prepared for this work, is different from that given in the first edition of Circular 52, being more accurate and more convenient.

[4] Chem. Ztg., **37**, p. 869; 1913.

[5] Private communication from Wilson Yerger.

Solutions (*Alkali*).—A sodium hydroxide (caustic soda) solution of any **known** and convenient strength may be employed. It is, however, probably most convenient to obtain a **normal** solution, 1 cc of which will neutralize 0.049 g of sulphuric acid. If, therefore, a sample of 10 cc. (or 1/100 of a liter) of the copper solution be titrated, each cc of the sodium hydroxide required is equivalent to 4.9 g per liter (or for most practical purposes 5 g/l or 0.67 oz./gal.) of sulphuric acid. If solutions of other strength are employed, the calculations should be correspondingly changed. Since the sodium hydroxide may change in strength on standing (owing to its attacking the glass of the container) it should either be renewed or restandardized by a chemist, at intervals of about six months.

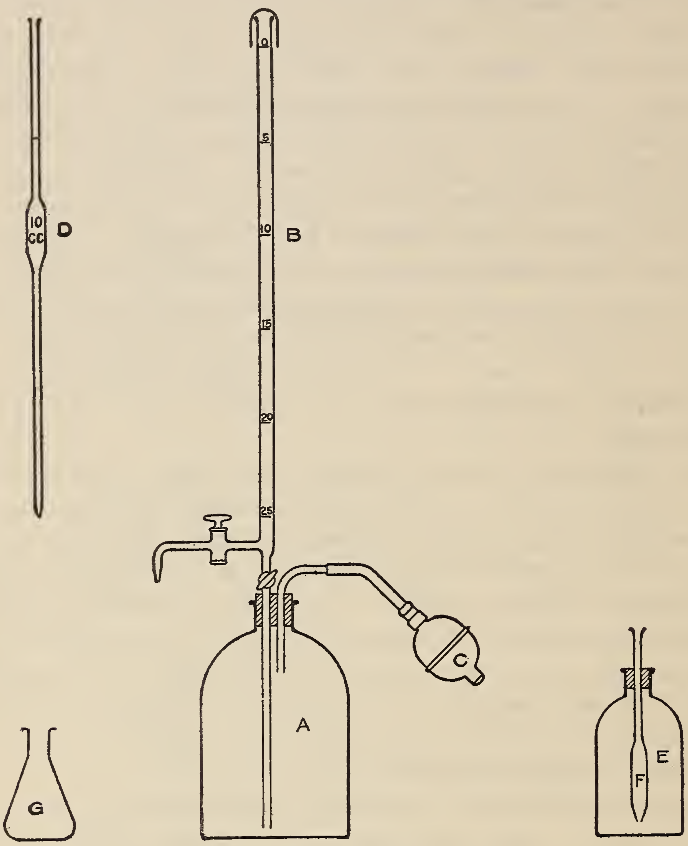

Fig. 1.—*Apparatus for testing acidity*

Explanation of symbols: A, 2-liter bottle; B, 25-cc burette with side stopcock; C, atomizer bulb; D 10-cc pipette; E, 500-cc bottle; F, pipette (about 2 cc); G, 100-cc flask.

Methyl orange solution, containing 1 part of methyl orange in 5000 parts of water, is used as an indicator, i. e., it changes color when all the sulphuric acid is neutralized.

Titration.—To carry out the titration, measure with a pipette 10 cc of the copper sulphate solution to be tested (after the bath has been thoroughly mixed) and run it into a small flask. Add to it about 2 cc of the methyl orange solution. To adjust the sodium hydroxide solution to the zero mark in the burette, turn the center stopcock so that the burette is connected with the stock bottle, and with the bulb pump the solution until it is above the zero mark, and then shut off this stopcock. Next turn the side stopcock and allow the solution to run into any convenient vessel (for waste) until all air is displaced from the side tube and the lower edge of the curved surface of the liquid is just at the zero mark. Now run the sodium hydroxide solution into the copper sulphate solution slowly, with constant shaking, until the violet color of the solution just disappears. If a decided green color or appreciable precipitate appears, too much alkali has been added and a new portion should be titrated. Note the position of the lower edge of the curve at the end of the titration. After completing the titrations, allow the burette to empty and put a little vaseline on the stopcocks.

Calculation.—To find the number of grams per liter of sulphuric acid in the copper solution, multiply by five the number of cubic centimeters of alkali used in the above titration.

(*c*) ADJUSTMENT OF COMPOSITION.—Having determined the specific gravity and the acidity of any such solution, it is a simple matter to adjust the composition to that desired, provided the specific gravities of solutions of known content of copper sulphate and sulphuric acid are known. Such a table, recently prepared [6] by this Bureau, is given as Table 8 in the Appendix. It was found that the density of these solutions at any temperature depends principally upon the total content of copper sulphate plus sulphuric acid. In Table 8 (Appendix) are shown the amounts of copper sulphate plus sulphuric acid (in grams per liter and ounces per gallon) in solutions of given specific gravity [7] at 25° C (77° F).

In order to determine the amount of copper sulphate (crystals) present in any solution of known specific gravity and acidity, all that is necessary is to deduct the determined acid content from

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[6] For details, see Bureau of Standards Scientific Paper No. 275.

[7] The specific gravities given in the table are for 25°/4° C. For all practical purposes, however, the values obtained at room temperatures with hydrometers calibrated, e. g., at 60°/60° F, may be used. If a Baumé hydrometer is used the corresponding specific gravity should be determined from Table 3.

the total content corresponding to the observed specific gravity as given in Table 8. Thus, if the solution has a specific gravity of 1.19, it contains 321 g/l of copper sulphate plus sulphuric acid. If it contains 50 g/l of sulphuric acid the copper sulphate content is 321 − 50 = 271 g/l.

The usual process of adjusting such a solution may be carried out as follows:

1. Determine from Table 8 the amount of copper sulphate (in g/l) in the solution.
2. Divide the difference between the desired copper sulphate content and the amount present, by the amount present, to obtain the percentage of the solution to be replaced with water.
3. Subtract this percentage of the sulphuric acid present from the amount originally present, and finally deduct the remainder from the amount of acid desired in order to obtain the amount (g/l) of acid to be added.
4. Multiply the number of grams per liter required by the number of liters in the tank and divide by 1000 to determine the number of kilograms of acid to be added.
5. To determine the pounds of acid to be added for each 100 gallons of solution in the bath multiply the number of grams per liter required by 0.83.

Example: Suppose a bath containing 680 liters (180 gallons) was found to have a specific gravity of 1.19 and to contain 50 g/l of sulphuric acid, and it is desired to adjust it to a specific gravity of 1.18 and an acid content of 60 g/l.

From Table 8 it may be noted that a solution with a specific gravity of 1.19 contains 321 g/l of copper sulphate plus sulphuric acid; therefore the present solution must contain 321 − 50 = 271 g/l of copper sulphate.

Similarly, a solution with a specific gravity of 1.18 contains a total of 303 g/l; therefore the desired solution contains 303 − 60 = 243 g/l of copper sulphate.

$$\begin{array}{lll} & 271 \text{ g/l} & \text{(copper sulphate present)} \\ \text{subtract} & \underline{243} \text{ g/l} & \text{(copper sulphate desired)} \\ \text{difference} & 28 \text{ g/l} & \end{array}$$

$$\frac{28 \quad \text{(difference)}}{271 \quad \text{(amount present)}} = 10.3 \text{ per cent,}$$

or for practical purposes 10 per cent.

Therefore, remove 10 per cent of 680 liters (68 liters) or of 180 gallons (18 gallons) of the solution from the tank. Since 10 per cent of the sulphuric acid present is thereby removed, there is left only 90 per cent of the original acid, or the equivalent of only $50 - 5 = 45$ g/l. To obtain 60 g/l it is therefore necessary to add $60 - 45 = 15$ g/l of sulphuric acid.

A bath containing 680 liters will therefore require $680 \times 15 =$ 10 200 grams, or 10.2 kilograms of sulphuric acid. One containing 100 gallons will require $15 \times 0.83 = 12.5$ pounds of acid or one with 180 gallons 1.8×12.5 or 22.5 pounds of acid; after which the bath should be filled with water to the desired level.

(*d*) NOTES.—1. In the above operations and calculations extreme accuracy is not required, since all that can be accomplished is to keep the composition of the solutions approximately constant. The chief value of such tests will be to enable the operator to avoid obtaining defective work, or in cases where the work may prove defective to determine immediately whether the composition of the solution or some other condition is at fault.

2. A permanent record of all titrations and of all changes in or additions to the solutions should be kept. Such records will ultimately furnish valuable information regarding the operation of the bath.

6. TEMPERATURE OF THE SOLUTIONS

(1) **Effect of Temperature of Deposition.**—A rise in temperature of the solution may affect the character of the deposit in two ways: (1) By increasing the conductivity of the solution and therefore the current density produced by a given voltage, and (2) by causing a change in the physical properties of the deposited copper even with the same current density. The first effect is particularly significant with respect to seasonal variations, since with lower temperatures of the baths (as after a cold night) the baths will not deposit as rapidly as at normal temperatures. From the standpoint of economy, the use of relatively high temperatures in the baths is desirable, because of the increased conductivity and consequent saving of power.

In the regular electrotyping operations where copper is deposited upon graphited wax molds, the permissible maximum temperature is limited by the softening point of the wax. Owing to the great variations in the composition of molding wax, it is impossible to state any definite limits, but it appears probable that for such work the highest safe temperature will not exceed 40° C (104° F).

In the recent studies made by this Bureau, the temperature was therefore varied from 25° to 40° C (77° to 104° F). Within this range it was found that the temperature *has a more marked* *influence upon the character of the deposited metal than any other single factor.* In all cases, under otherwise similar conditions, a rise in temperature from 25° to 40° C caused a decided decrease in the tensile strength and therefore probably in the hardness of the copper, accompanied by a much coarser crystalline structure. This effect of temperature was most marked with medium current density (4 to 6 amp./dm² or 35 to 55 amp./sq. ft.), and with high acid content. In some cases this change in temperature reduced the tensile strength from 2800 to 1400 kg/cm² (40 000 to 20 000 lbs./sq. in.). In order to obtain satisfactory deposits at medium current density the solution should therefore be maintained between 25° and 30° C (77° and 86° F), and even with high current densities the temperature should not exceed 35° C (95° F).

(2) **Regulation of the Temperature.**—(*a*) RELATION BETWEEN THE RESISTANCE OF THE BATH AND THE AMOUNT OF HEAT PRODUCED.—Whenever an electrical current is passed through any solution, the **amount of heat** produced in a given time is directly proportional to the resistance of the solution and to the square of the current; in other words, H (heat produced) $= C^2R$. Obviously, the lower the resistance of the solution the less heat will be produced in it when a given current is passed. If, however, the resistance of the solution is lowered (e. g., by the addition of sulphuric acid) and the voltage is maintained constant, the current produced will be greater, and since the heat evolved is proportional to the **square** of the current the increased production of heat due to the increased current will be greater than the decrease due to reduced resistance. This may be illustrated by a simple example.

Example:[8] (*a*) Suppose the resistance is 0.02 ohm, and a voltage of 4 volts is employed, then the current passing will be $\frac{4}{0.02} = 200$ amperes. In this case the heat produced in one second may be considered as equal to C^2R or $(200)^2 \times 0.02 = 800$ joules (heat units).[9]

(*b*) Suppose now that the resistance is reduced to one-half (i. e., to 0.01 ohm) and the voltage is retained at 4 volts. Then the current produced will be $\frac{4}{0.01}$ or 400 amperes, and the heat produced will be equal to $(400)^2 \times 0.01 = 1600$ joules.

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[8] In these calculations used solely for illustration, the external resistance has been neglected.

[9] A *joule* is a heat unit equal to 0.2387 calories. A *calorie* is the amount of heat required to raise the temperature of 1 gram of water 1° C.

(*c*) Suppose that after reducing the resistance to 0.01 ohm, as in (*b*), the voltage is reduced to 2 v, thus producing $\frac{2}{0.01} = 200$ amperes, i. e., the same current (and rate of deposition) as in (*a*). In this case the heat produced is equal to $(200)^2 \times 0.01 = 400$ joules. In other words, by reducing the resistance to one-half, without changing the voltage, the heat produced is twice as great, while a reduction of the resistance to one-half without changing the current causes a production of only one-half the amount of heat. An appreciation of the above relation should remove the prevalent misunderstanding as to whether reducing the resistance of a solution increases or decreases the heating effect.

(*b*) THE TEMPERATURE PRODUCED IN THE BATH.—The above considerations refer only to the total heat evolved, and not to the **temperature** of the bath, which is the factor to be controlled. The actual temperature of the bath is influenced by (*a*) the amount of heat liberated in the bath, (*b*) the volume of the bath (the larger the bath the less is the rise in temperature produced by a given amount of heat), (*c*) the heat lost by radiation and conduction (which depends largely on the temperature of the surroundings), and (*d*) the heat used in evaporating part of the solution (which depends principally on the temperature, volume, and distribution of the air used for agitation). Without a knowledge of all these factors, it would be impossible to calculate the resultant temperature of the bath in any particular case. As is well known, however, the tendency under usual conditions is to raise the temperature above that desired.

(*c*) CONTROL OF TEMPERATURE.—In order to maintain an approximately uniform temperature, all such baths should be provided with suitable means for heating or cooling the solutions, such as a lead coil (electrically insulated), through which either hot water (or steam) or cold water can be circulated. The use of live steam to heat the solutions or of ice to cool them, as sometimes practiced, is at best a temporary expedient and is almost certain to cause irregularities in the composition and operation of the baths. The most difficult problem is that of maintaining, in warm weather, the temperature recommended (i. e., not above 30° C (86° F), when a considerable amount of work is carried on in the bath. It is recognized that this can be accomplished only by careful attention, and the use either of considerable amounts of air and of cooling water or of a number of large tanks which are agitated even during the periods between their actual use.

7. COMPOSITION AND PHYSICAL PROPERTIES OF THE ANODES

The exact **composition** of the copper anodes used in such work is of little practical importance, since at the present time the amounts of impurities likely to be present in commercial copper are so small as to have little or no effect upon the operation of the baths. Moreover, as previously noted, the accumulation of impurities in the baths (even when used for years) is practically prevented by what amounts to a constant renewal of solutions in the process of removing the portion of the solution adhering to the cathodes and the portions required to adjust the density at intervals. The actual **source**[10] of the copper (e. g., whether "electrolytic" or "lake" copper) is of little or no significance; indeed, the "electrolytic" copper is usually at least as pure as the "lake" copper. The **state** (or physical condition) of the copper may, however, play an important part. Of the three available classes of copper anodes, viz, cast, electrolytically deposited, and rolled, the latter are at present used almost exclusively in electrotyping work. This preference seems to be well founded, since it has been shown by Pfanhauser and others that the use of electrolytically deposited, and especially cast, copper anodes gives rise to the formation of fine particles, consisting principally of metallic copper. This behavior may be attributed to the fact that the crystalline structure of the electrolytically deposited or cast copper is usually coarser than that of rolled copper. The production of the fine particles is objectionable on account of the loss of copper, and the "fouling" of the solution, with danger of such particles lodging upon a cathode and forming nuclei for irregular growths. It is probable that the presence of this fine copper slime also increases the consumption of sulphuric acid, owing to the purely chemical solubility of such fine copper in copper sulphate—sulphuric acid solutions agitated with air.[11]

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[10] In this connection the distinction should be emphasized between, on the one hand, the source and method of refining the copper; and, on the other hand, the method of preparing the copper for use as anodes.

In general copper occurs in nature either in the free state as metallic copper; or in compounds, such as sulphides or carbonates. In the first form, which is found principally in the Lake Superior region, the copper requires only a mechanical treatment and melting before casting into bars of nearly pure copper, the so-called "lake copper." The ores containing compounds of copper (such as occur in the Western States) contain numerous impurities, including gold and silver, and require in general a much more complicated treatment, involving as a rule mechanical separation, smelting, and subsequent refining by electrolysis. The product, usually known as "electrolytic copper," is usually at least as pure as the "lake" copper, and is therefore equally satisfactory wherever pure copper is desired.

In present practice, as stated above, *rolled* copper plates are generally used as anodes. In the preparation of such plates the copper must first be melted and cast, in which operation any differences in structure due to the original method of refining entirely disappear. In the above paragraph objection is made not to the use for anodes of "electrolytic" copper, but of "electrolytically deposited" copper, which has not been subsequently cast and rolled into suitable plates.

[11] In this connection it may be mentioned that if at any time it is necessary to filter these solutions, the use of woolen cloth is to be recommended, since it is much less attacked than is cotton.

8. CHARACTER AND CONDITION OF THE CATHODE SURFACE

In general, in this country at least, commercial electrotypes are deposited either upon lead or upon graphited wax molds. The former require no special consideration, since the lead is itself a conductor, and requires the addition of no conducting surface. In the use of wax molds, however, there is good reason to believe that many of the practical difficulties encountered in the production of electrotypes are due to defects or irregularities in the composition of the molding wax or of the graphite, or in the method of taking the impression or coating it with graphite. It is impossible for this Bureau to furnish any accurate information upon this subject until an opportunity is secured to study or devise adequate methods for testing the properties of molding wax and graphite, and of formulating specifications by means of which uniformly satisfactory products can be secured. It may be safely stated, however, that under proper conditions it is possible to so coat the molds with graphite that no preliminary. treatment with iron filings and copper sulphate is required. It is, therefore, doubtful whether this common process of metallizing (erroneously called "oxidizing") of the forms is really an advantage. The time thereby saved in the bath is probably more than balanced by the time and cost of the operation, and by the danger of scratching the forms, especially in halftone work. Moreover, it is reasonably certain that the thin film of copper thus deposited chemically is less hard and coherent than the normal electrolytic deposit. It is probable, however, that the preliminary coating with copper not only saves some time, but also produces a shell of more uniform thickness, owing to the subsequent simultaneous electrodeposition of copper at all parts of the plate.

In the experiments conducted by this Bureau, the wax molds were coated in a wet graphiting machine, and were placed in the bath with no further treatment. It is probable, however, that in all essential respects the conclusions reached from the study of the shells produced under such conditions will apply to those made under other conditions, such as with dry graphiting or even upon lead or other metallic forms.

9. CURRENT DENSITY AND AGITATION EMPLOYED

Next to the temperature the current density is the most important of the factors determining the properties of the deposited copper. This is readily understood since, as previously stated, the current density determines the rate of deposition of the copper, and therefore, under otherwise uniform conditions, the size of the individual crystals. The latter in turn determines largely the hardness, tensile strength, and ductility of the deposited copper. In general it may be stated that the finer the grains or crystals the greater the tensile strength and therefore probably the hardness of the metal. The ductility of such copper was found to increase with increased tensile strength up to a tensile strength of about 2800 kg/cm ^{2} (40 000 lbs./sq. in.) and then to decrease, i. e., copper with a higher tensile strength was relatively brittle. It is generally recognized that an increase in the current density causes a decrease in the size of the crystals up to a certain point, while with current densities beyond that point the deposit becomes coarse and irregular with the characteristic "burnt" appearance. The generally accepted explanation of the latter effect is that with too high current densities the solution adjacent to the cathode becomes impoverished in copper, and in consequence an appreciable part of the current is consumed in decomposing sulphuric acid, with the evolution of hydrogen, which causes the deposit to be rough or spongy. In accordance with this view the obvious method of avoiding the difficulty, and of obtaining coherent deposits with high current densities, is to insure a fresh supply of copper sulphate in the solution near the cathode. This can be accomplished (*a*) by having the solution rich in copper sulphate, and (*b*) by constantly bringing fresh portions of the solution to the surface of the cathode. As is well known, the latter operation is commonly accomplished by suitable agitation of the solution, usually performed in electrotyping baths by means of a current of compressed air. That this method is efficient in permitting more rapid deposition of the copper is shown by the fact that in practice, in "still" baths, the current density that can be employed is seldom over 3 amperes per square decimeter (28 amperes per square foot), and usually much less; while in baths agitated with air, current densities of 6 to 8 amp./dm ^{2} (56 to 74 amp./sq. ft.), or even higher, have been used successfully in commercial work. The extreme possibilities of the effect of agitating the solutions are shown in results obtained by Bennett[12] who, by very rapidly rotating a small cathode, was able, with current densities up to 430 amp./dm ^{2} (4000 amp./sq. ft.), to obtain coherent deposits of copper having unusually high tensile strength. While any such degree of agitation is manifestly impracticable in electrotyping baths, the results are of interest as indicating the desirability of having the highest

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[12] Trans. Amer. Electrochem. Soc., 21, p. 253; 1912.

convenient degree of agitation, especially between the anode and cathode surfaces.

In the experiments conducted by the Bureau a very thorough agitation was used and current densities from 2.5 to 10.5 amp./dm^2 (23 to 98 amp./sq. ft./) were employed. It was found that at low temperature (25° C or 77° F) the tensile strength increases with increased current density. At high temperature (40° C or 104° F) an increase in current density from 2.5 amperes to 4.5 amp./dm^2 (about 25 to 40 amp./sq. ft.) causes a decrease in tensile strength; and an increase in current density from 4.5 to 10.5 amp./dm^2 (42 to 98 amp./sq. ft.) produces an increased tensile strength. It is therefore recommended that, at low temperature, current densities from 4.5 to 9.5 amp./dm^2 (about 40 to 90 amp./sq. ft.) be employed, and, at high temperature, from 8 to 10 amp./dm^2 (about 75 to 95 amp./sq. ft.). *For uniform results it is essential that the current density be measured by means of an ammeter.* While the voltmeter is a useful and important adjunct to such baths, it does not serve to maintain uniform current conditions, unless all other conditions are absolutely uniform. By the use of an ammeter it is possible not only to directly measure the current, but also to ascertain whether other conditions are uniform, since in a solution of definite composition and temperature and with electrodes of a given size and distance apart a definite voltage will always produce a given current density.

As previously noted (p. 18) it is possible by the use of various addition agents to increase the permissible current density and thereby the rate of deposition. To deposit copper at the rate claimed by inventors of some of the baths of this type it would require about 22 amp./dm^2 (200 amp./sq. ft.), i. e., over twice the maximum which is used in the regular baths. So far as known, however, no such current densities are in actual use even in baths containing addition agents. With such high current densities it is obvious that special care is required to avoid excessive heating of the solutions, or other abnormal behavior.

10. DISTANCE BETWEEN ELECTRODES

One of the important factors in determining the current density is the distance between the anodes and cathodes. From the standpoint of economy it is obvious that reducing this distance decreases the resistance of the bath and therefore decreases the voltage (and power) required to produce a given current density.

Practically, however, the minimum distance is determined by the convenience of handling the cases without mechanical injury and the possibility of producing a uniform and satisfactory agitation between electrodes very close together. In general, it may be stated that the better the agitation the closer may the electrodes be placed. It was found impossible to detect any marked effect of the distance itself (from 6 to 15 cm or 2.5 to 6 inches) upon the quality of the copper. It is probable, however, that in special cases, especially for forms with deep recesses, the greater distance is an advantage. For most purposes a distance of 7.5 cm (3 inches) will probably be satisfactory.

11. DISTRIBUTION OF THE COPPER—THICKNESS OF SHELLS

As previously stated the rate of deposition of the copper and therefore the average thickness of the deposits produced in a given time depends primarily upon the current density employed. From a knowledge of the weight of copper deposited from such solutions by one ampere hour (1.1186 g) and the specific gravity of copper (8.9), it is a simple matter to calculate the theoretical weight and thickness of copper deposited by a given current in a specified time. Tables 9, 10, and 11 give such data (in metric and customary units) calculated upon the assumption that the current efficiency is 100 per cent and that the copper is uniformly distributed over a plane surface. In other words, the tables give, respectively, the maximum weight and average thickness of copper produced by a given current in a given time, and conversely the minimum time required to produce a deposit of specified thickness with a given current density.

Since the current efficiency in acid sulphate baths under normal conditions is seldom less than 95 per cent, and is frequently 98 or 99 per cent, no correction need be made for this factor in practical work.

On the other hand, it should be emphasized that the assumption that the copper will be uniformly distributed, even over a plane surface, is never realized in practice, at least when deposited directly upon graphited wax molds. Owing to the appreciable time required for "covering" (usually 15 to 20 minutes) it would be expected that those portions first covered should have the thickest deposits. While this factor no doubt has some influence, it is almost invariably found in practice that the copper on such shells is much thicker on the bottom and two vertical edges than in the center, or near the top edge. Even when considerable care was used, it was found that differences in thickness of from 20 to 30 per cent were found in different parts of the plane portion of the form. This behavior, which may be explained by the greater current density near the edges, is most marked when the anodes are appreciably larger, and especially deeper, than are the corresponding cathode surfaces. For this, as well as other reasons, it is therefore not desirable to use anodes larger than the cathodes.

Even more marked than the above differences are those observed in different parts of the type surface, as determined from a study of a considerable number of finished plates from different plants. Probably all electrotypers have realized that upon a type surface the copper is not so thick as upon the plane surface near the edges; but most will be surprised to learn that in shells having a thickness on the plane surface of 0.15 to 0.2 mm (0.006 to 0.008 inch) the copper on the printing surfaces is usually only 0.05 to 0.10 mm (0.002 to 0.004 inch) in thickness. It is obvious, therefore, that any specification by which an electrotyper agrees to furnish a shell "from 0.011 to 0.014 inch in thickness" is misleading and practically impossible of fulfillment.

The exact factors influencing the distribution and uniformity of the copper on such surfaces have not yet been thoroughly studied. In general, it may be stated that the greater the distance between the anodes and cathodes, the more uniform the deposit. It was found that there is no appreciable difference in the current density (and therefore weight of copper deposited) upon a given area, whether plane, halftone, or type form. In using Table 9 the approximate weight of copper deposited upon a given area of such a surface in a specified time (excluding the period required for covering) will be obtained. (This will include the weight of the copper on the rough edges.) In using Tables 10 and 11, however, it is necessary to make some allowance for the variation in thickness. In practice it has been found that the thickness on a smooth or halftone surface will be from 80 to 90 per cent of that derived from these tables, while upon a type form the thickness on the printing surface will be only 30 to 50 per cent of that calculated. The values in the tables are therefore useful chiefly in computing the relative thickness of shells produced under different conditions.

In general, it is believed that the quality (hardness and ductility) of the copper has a greater influence upon the wearing qualities than the exact thickness, provided the latter be such as to insure a coherent shell, free from holes or other imperfections.

12. SUMMARY AND RECOMMENDATIONS ON COPPER ELECTROTYPING

(1) **Properties of Deposited Copper.**—(*a*) In general, the finer the crystals, the higher is the tensile strength and therefore probably the hardness of the copper.

(*b*) The ductility (as measured by the permanent elongation after fracture) increases with increased tensile strength up to a tensile strength of about 2800 kg/cm^2 (40 000 lbs./sq. in.) and then decreases, i. e., the copper becomes relatively brittle.

(*c*) It is believed that copper with a tensile strength of 2500 to 2800 kg/cm^2 (35 000 to 40 000 lbs./sq. in.) and an elongation of 20 to 30 per cent will be found satisfactory for electrotype plates.

(2) **Agitation.**—The best possible agitation should be employed, especially between the anodes and cathodes.

(3) **Composition of Solutions.**—(*a*) Under otherwise uniform conditions an increase in the amount of copper sulphate causes a slight increase in tensile strength.

(*b*) Under otherwise uniform conditions, an addition of sulphuric acid at low temperature (25° C or 77° F) causes an increase in tensile strength; and at high temperature (40° C or 104° F) causes a decrease in tensile strength.

(*c*) The solution should contain from 50 to 80 g/l (7 to 11 oz./gal.) of sulphuric acid, and from 250 to 200 g/l (34 to 27 oz./gal.) of copper sulphate. The specific gravity of the solution should be from 1.17 to 1.18 (21° to 22° B.).

(*d*) Whatever the composition employed, it should be kept as nearly constant as possible, to insure uniform working conditions.

(4) **Temperature.**—(*a*) A rise in temperature under otherwise uniform conditions always decreases the tensile strength. This effect is most marked with high acid content and with medium current density.

(*b*) The solution should be maintained between 25° and 30° C (77° to 86° F), unless high current densities are used (over 8 amp./dm^2 or 75 amp./sq. ft.), in which case the temperature may be allowed to rise to 35° C (95° F).

(5) **Current Density.**—(*a*) At low temperature, the higher the current density (up to 9.5 amp./dm^2 or 90 amp./sq. ft.) the higher the tensile strength of the copper. Good results can be obtained from 4.5 to 9.5 amp./dm^2 (40 to 90 amp./sq. ft.).

(*b*) At high temperature an increase in current density from 2.5 to 4.5 amp./dm^2 (about 25 to 40 amp./sq. ft.) causes a decrease in tensile strength. With current densities from 4.5 to 10.5 amp./dm^2 (40 to 100 amp./sq. ft.) the tensile strength increases.

At 35° C (95° F) current densities of 8 to 10 amp./dm^2 (about 75 to 95 amp./sq. ft.) will yield the most satisfactory deposits.

(6) **Thickness of Shells.**—On smooth or halftone surfaces, the thickness of copper will usually be from 80 to 90 per cent of that calculated for the current and time used. On type surfaces the copper will be only 30 to 50 per cent as thick as calculated.

VIII. NICKEL ELECTROTYPING SOLUTIONS

Although this Bureau has made numerous observations upon the composition and operation of commercial nickel electrotyping solutions, it is not yet prepared to make specific recommendations concerning them. To do so will require an extensive study of the numerous problems of both nickel typing and nickel plating. In fact, the production of nickel electrotypes, especially upon wax molds, is in some respects a more difficult operation than nickel plating, since cracking and curling are much more likely to occur in the former case. The whole subject of the best composition of nickel solutions and anodes and the operation of the baths is greatly in need of study, which will be conducted as rapidly as facilities permit. In connection with such a study, it is hoped to make some tests upon **cobalt** plating,[13] and especially **cobalt** electrotyping, in which very promising results have been obtained. In the meantime the Bureau will be pleased to answer so far as possible any inquiries in this connection.

IX. NOTICE OF NEW EDITIONS

Any person may secure one copy of this circular upon request addressed to the Bureau of Standards, Washington, D. C.

Notices of new editions of this circular will be published in the various technical journals.

X. APPENDIX

TEMPERATURE CONVERSION TABLES

TABLE 1

Conversion of Centigrade (C) to Fahrenheit (F) Temperatures

°C	°F	°C	°F	°C	°F	°C	°F
0	= 32	30	= 86	55	= 131	80	= 176
5	= 41	35	= 95	60	= 140	85	= 185
10	= 50	40	= 104	65	= 149	90	= 194
15	= 59	45	= 113	70	= 158	95	= 203
20	= 68	50	= 122	75	= 167	100	= 212
25	= 77						

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[13] Kalmus, Harper, and Savell, Jour. Ind. Eng. Chem., 7, p. 379; 1915.

TABLE 2

Conversion of Fahrenheit (F) to Centigrade (C) Temperatures

°F	°C	°F	°C	°F	°C	°F	°C
0	−17.8	55	12.8	110	43.3	165	73.9
5	−15.0	60	15.6	115	46.1	170	76.7
10	−12.2	65	18.3	120	48.9	175	79.4
15	− 9.4	70	21.1	125	51.7	180	82.2
20	− 6.7	75	23.9	130	54.4	185	85.0
25	− 3.9	80	26.7	135	57.2	190	87.8
30	− 1.1	85	29.4	140	60.0	195	90.6
32	0.0	90	32.2	145	62.8	200	93.3
35	1.7	95	35.0	150	65.6	205	96.1
40	4.4	100	37.8	155	68.3	210	98.9
45	7.2	105	40.6	160	71.1	212	100.0
50	10.0						

TABLES FOR CONVERTING DEGREES BAUMÉ [a] TO SPECIFIC GRAVITY AND VICE VERSA [b]

TABLE 3

Conversion of Degrees Baumé (B) to Specific Gravity (Sp. gr.)

°B	Sp. gr.	°B	Sp. gr.	°B	Sp. gr.	°B	Sp. gr.
0	1.000	8	1.058	16	1.124	24	1.198
1	1.007	9	1.066	17	1.133	25	1.208
2	1.014	10	1.074	18	1.142	26	1.218
3	1.021	11	1.082	19	1.151	27	1.229
4	1.028	12	1.090	20	1.160	28	1.239
5	1.036	13	1.098	21	1.169	29	1.250
6	1.043	14	1.107	22	1.179		
7	1.051	15	1.115	23	1.189		

TABLE 4

Conversion of Specific Gravity (Sp. gr.) to Degrees Baumé (B)

Sp. gr.	°B	Sp. gr.	°B	Sp. gr.	°B	Sp. gr.	°B
1.00	0.00	1.07	9.49	1.14	17.81	1.21	25.17
1.01	1.44	1.08	10.74	1.15	18.91	1.22	26.15
1.02	2.84	1.09	11.97	1.16	20.00	1.23	27.11
1.03	4.22	1.10	13.18	1.17	21.07	1.24	28.06
1.04	5.58	1.11	14.37	1.18	22.12	1.25	29.00
1.05	6.90	1.12	15.54	1.19	23.15		
1.06	8.21	1.13	16.68	1.20	24.17		

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[a] This scale for liquids heavier than water is defined by the formula: $\text{Baumé} = 145 - \dfrac{145}{\text{Sp. gr.}\ \frac{60^\circ}{60^\circ}\ \text{F}}$, where sp. gr. $\frac{60^\circ}{60^\circ}$ F represents the specific gravity of the solution at 60° F, referred to water at 60° F.

[b] These tables are given in more complete and accurate form in Circular No. 19 of the Bureau of Standards.

MISCELLANEOUS TABLES

TABLE 5

Approximate Equivalents of Metric and Customary United States Units

Length:

1 meter (m) = 39.37 inches
1 decimeter (dm) = 3.937 inches
1 centimeter (cm) = 0.3937 inch
1 inch = 2.54 centimeters
1 foot = 30.48 centimeters

Area:

1 square meter (m^2) = 1550 square inches
1 square decimeter (dm^2) = 15.5 square inches
1 square centimeter (cm^2) = 0.155 square inch
1 square inch = 6.45 square centimeters
1 square foot = 9.29 square decimeters

Volume:

1 cubic meter (m^3) = 35.31 cubic feet
1 cubic decimeter (dm^3) = 61.02 cubic inches
1 cubic centimeter (cm^3) = 0.061 cubic inch
1 cubic inch = 16.39 cubic centimeters
1 cubic foot = 28.32 cubic decimeters

Capacity (liquid measure):

1 liter (l) = 1000 milliliters (or about 1000 cubic centimeters)
1 liter = 1.057 quarts
1 milliliter (ml) or cc[a] = 0.034 fluid ounce
1 gallon = 231 cubic inches
1 gallon = 3.785 liters
1 quart = 0.946 liter

Mass (weight):

1 kilogram (kg) = 1000 grams
1 kilogram = 2.205 avoirdupois pounds
1 gram (g) = 0.0353 avoirdupois ounce
1 avoirdupois ounce = 28.35 grams
1 avoirdupois pound = 453.6 grams
1 liter of water weighs 1 kilogram

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[a] The milliliter, the true unit of capacity, is at present commonly designated (e. g., on volumetric apparatus) as a cubic centimeter (usually abbreviated cc), from which, however, it has a slightly different value. For all practical purposes, however, the term cubic centimeter and the abbreviation cc may be employed.

TABLE 6

Approximate Conversion Factors for Metric and Customary United States Units

1. Grams per liter (g/l) multiplied by 0.134 = ounces per gallon (oz./gal.)
2. Grams per liter multiplied by 0.0083 = pounds per gallon (lb./gal.)
3. $\frac{\text{Grams per liter}}{\text{10 multiplied by specific gravity}}$ = percentage by weight
4. Amperes per square decimeter (amp./dm^2) multiplied by 9.29 = amperes per square foot (amp./sq. ft.)
5. Kilograms per square centimeter (kg/cm^2) multiplied by 14.22 = pounds per square inch (lb. /sq. in.)

TABLE 7

Solubility of Copper Sulphate at 15° C (59° F) in the Presence of Sulphuric Acid [a]

Sulphuric acid present in grams per liter [b]	Solubility of copper sulphate (crystallized salt) in grams per liter	Sulphuric acid present in grams per liter [b]	Solubility of copper sulphate (crystallized salt) in grams per liter
0	395	60	245
10	348	80	231
20	308	100	215
30	280	120	197
40	260	140	180
50	253		

[a] Derived from Pfanhauser: Herstellung von Metallgegenstände, p. 11.

[b] From the context in the original it appears that by "1 per cent sulphuric acid" is meant 10 grams per liter, etc., on which basis the values given have been calculated.

TABLE 8

Total Concentration of Copper Sulphate Plus Sulphuric Acid in Solutions of Given Specific Gravity

Specific gravity at $\frac{25^\circ}{4^\circ}$ C [a]	Copper sulphate plus sulphuric acid		Specific gravity at $\frac{25^\circ}{4^\circ}$ C [a]	Copper sulphate plus sulphuric acid	
	Grams/liter	Ounces/gallon		Grams/liter	Ounces/gallon
1.01	20	2.7	1.13	217	29.1
1.02	36	4.8	1.14	234	31.3
1.03	52	7.0	1.15	251	33.6
1.04	68	9.1	1.16	268	35.9
1.05	84	11.3	1.17	286	38.3
1.06	100	13.4	1.18	303	40.6
1.07	117	15.7	1.19	321	43.0
1.08	133	17.8	1.20	339	45.4
1.09	150	20.0	1.21	357	47.8
1.10	166	22.3	1.22	375	50.2
1.11	183	24.5	1.23	393	52.6
1.12	200	26.8			

[a] The specific gravity determined at 25° C (77° F) based upon the density of water at 4° C (39.2° F)

TABLE 9

Weight of Copper Deposited from Acid Sulphate Solution [a]

(*a*) IN METRIC UNITS

Current density used (amp./dm²)	Weight of 1 dm² of deposit (grams)					
	1 hour	2 hours	3 hours	4 hours	5 hours	10 hours
0.5	0.59	1.19	1.78	2.37	2.96	5.93
1.0	1.19	2.37	3.56	4.74	5.93	11.86
1.5	1.78	3.56	5.34	7.12	8.90	17.79
2.0	2.37	4.74	7.12	9.49	11.86	23.72
2.5	2.96	5.93	8.90	11.86	14.82	29.65
3.0	3.56	7.12	10.67	14.23	17.79	35.58
3.5	4.15	8.30	12.45	16.60	20.76	41.51
4.0	4.74	9.49	14.23	18.98	23.72	47.44
4.5	5.34	10.67	16.01	21.35	26.68	53.37
5.0	5.93	11.86	17.79	23.72	29.65	59.30
5.5	6.52	13.05	19.57	26.09	32.62	65.23
6.0	7.12	14.23	21.35	28.46	35.58	71.16
6.5	7.71	15.42	23.13	30.84	38.54	77.09
7.0	8.30	16.60	24.91	33.21	41.51	83.02
7.5	8.90	17.79	26.68	35.58	44.48	88.95
8.0	9.49	18.98	28.46	37.95	47.44	94.88
8.5	10.08	20.16	30.24	40.32	50.40	100.81
9.0	10.67	21.35	32.02	42.70	53.37	106.74
9.5	11.27	22.53	33.80	45.07	56.34	112.67
10.0	11.86	23.72	35.58	47.44	59.30	118.60

[a] One ampere hour deposits 1.186 g copper

(*b*) IN CUSTOMARY UNITS [a]

Current density used (amp./sq. ft.)	Weight of 1 square foot of deposit (ounces)					
	1 hour	2 hours	3 hours	4 hours	5 hours	10 hours
1	0.04	0.08	0.13	0.17	0.21	0.42
5	.21	.42	.63	.84	1.05	2.09
10	.42	.84	1.26	1.67	2.09	4.18
15	.63	1.26	1.88	2.51	3.14	6.28
20	.84	1.67	2.51	3.35	4.18	8.37
25	1.05	2.09	3.14	4.18	5.23	10.46
30	1.26	2.51	3.77	5.02	6.28	12.55
35	1.46	2.93	4.39	5.86	7.32	14.64
40	1.67	3.35	5.02	6.69	8.37	16.73
45	1.88	3.77	5.65	7.53	9.41	18.83
50	2.09	4.18	6.28	8.37	10.46	20.92
55	2.30	4.60	6.90	9.20	11.50	23.01
60	2.51	5.02	7.53	10.04	12.55	25.10
65	2.72	5.44	8.16	10.88	13.60	27.19
70	2.93	5.86	8.79	11.71	14.64	29.28
75	3.14	6.28	9.41	12.55	15.69	31.38
80	3.35	6.69	10.04	13.39	16.73	33.47
85	3.56	7.11	10.67	14.22	17.78	35.56
90	3.77	7.53	11.30	15.06	18.83	37.65
95	3.97	7.95	11.92	15.90	19.87	39.74
100	4.18	8.37	12.55	16.73	20.92	41.84

[a] One ampere hour deposits 0.04184 ounce of copper.

TABLE 10

Thickness of Copper Deposited from Acid Sulphate Solution [a]

(*a*) IN METRIC UNITS

Current density used (amp./dm²)	Thickness of deposited copper (millimeters)					
	1 hour	2 hours	3 hours	4 hours	5 hours	10 hours
0.5	0.007	0.013	0.020	0.027	0.033	0.067
1.0	.013	.027	.040	.053	.067	.133
1.5	.020	.040	.060	.080	.100	.200
2.0	.027	.053	.080	.107	.133	.267
2.5	.033	.067	.100	.133	.167	.333
3.0	.040	.080	.120	.160	.200	.400
3.5	.047	.093	.140	.187	.233	.466
4.0	.053	.107	.160	.213	.267	.533
4.5	.060	.120	.180	.240	.300	.600
5.0	.067	.133	.200	.267	.333	.666
5.5	.073	.147	.220	.293	.367	.733
6.0	.080	.160	.240	.320	.400	.800
6.5	.087	.173	.260	.347	.433	.866
7.0	.093	.187	.280	.373	.466	.933
7.5	.100	.200	.300	.400	.500	.999
8.0	.107	.213	.320	.426	.533	1.066
8.5	.113	.227	.340	.453	.566	1.133
9.0	.120	.240	.360	.480	.600	1.199
9.5	.127	.253	.380	.506	.633	1.266
10.0	.133	.267	.400	.533	.666	1.333

[a] Based upon the following data: One ampere hour deposits 1.186 g copper. Specific gravity of copper =8.9. One ampere per square decimeter therefore deposits 0.01333 mm copper in one hour.

(*b*) IN CUSTOMARY UNITS [a]

Current density used (amp./sq. ft.)	Thickness of deposited copper (inches)					
	1 hour	2 hours	3 hours	4 hours	5 hours	10 hours
1	0.00006	0.00011	0.00017	0.00023	0.00028	0.00057
5	.0003	.0006	.0008	.0011	.0014	.0028
10	.0006	.0011	.0017	.0023	.0028	.0056
15	.0008	.0017	.0025	.0034	.0042	.0085
20	.0011	.0023	.0034	.0045	.0056	.0113
25	.0014	.0028	.0042	.0056	.0071	.0141
30	.0017	.0034	.0051	.0068	.0085	.0169
35	.0020	.0040	.0059	.0079	.0099	.0198
40	.0023	.0045	.0068	.0090	.0113	.0226
45	.0025	.0051	.0076	.0102	.0127	.0254
50	.0028	.0056	.0085	.0113	.0141	.0282
55	.0031	.0062	.0093	.0124	.0155	.0311
60	.0034	.0068	.0102	.0136	.0169	.0339
65	.0037	.0073	.0110	.0147	.0184	.0367
70	.0040	.0079	.0119	.0158	.0198	.0395
75	.0042	.0085	.0127	.0169	.0212	.0424
80	.0045	.0090	.0136	.0181	.0226	.0452
85	.0048	.0096	.0144	.0192	.0240	.0480
90	.0051	.0102	.0152	.0203	.0254	.0508
95	.0054	.0107	.0161	.0215	.0268	.0536
100	.0056	.0113	.0169	.0226	.0282	.0565

[a] One ampere per square foot deposits 0.0000565 inch of copper in one hour.

TABLE 11

Time Required for Copper Deposition from Acid-Sulphate Solutions [a]

(*a*) IN METRIC UNITS

Current density used (amp./ sq. ft.)	Time required to produce deposit of specified thickness (millimeters)																			
	0.1		0.2		0.3		0.4		0.5		0.6		0.7		0.8		0.9		1.0	
	Hrs.	Mins.	Hrs.	Mins.	Hrs.	Mins.	Hrs.	Mins.	Hrs.	Mins.	Hrs.	Mins.	Hrs.	Mins.	Hrs.	Mins.	Hrs.	Mins.	Hrs.	Mins.
0.5	15	....	30	1	45	1	60	2	75	2	90	3	105	4	120	4	135	4	150	5
1.0	7	30	15	....	22	31	30	1	37	31	45	1	52	32	60	2	67	32	75	2
1.5	5	....	10	....	15	....	20	....	25	1	30	1	35	1	40	1	45	1	50	1
2.0	3	45	7	30	11	16	15	....	18	46	22	31	26	15	30	1	33	46	37	31
2.5	3	....	6	....	9	....	12	....	15	....	18	1	21	1	24	1	27	1	30	1
3.0	2	30	5	....	7	30	10	....	12	30	15	....	17	31	20	1	22	31	25	1
3.5	2	9	4	18	6	26	8	35	10	43	12	52	15	....	17	9	19	18	21	26
4.0	1	53	3	45	5	38	7	30	9	23	11	16	13	8	15	.....	16	53	18	46
4.5	1	40	3	20	5	....	6	40	8	20	10	....	11	41	13	20	15	.....	16	41
5.0	1	30	3	....	4	30	6	....	7	30	9	....	10	30	12	.....	13	30	15	....
5.5	1	22	2	44	4	5	5	28	6	49	8	11	9	33	10	55	12	17	13	38
6.0	1	15	2	30	3	45	5	....	6	15	7	30	8	45	10	.....	11	15	12	30
6.5	1	9	2	18	3	28	4	37	5	46	6	55	8	5	9	14	10	23	11	32
7.0	1	4	2	8	3	13	4	17	5	22	6	26	7	30	8	35	9	39	10	43
7.5	1	....	2	....	3	....	4	....	5	....	6	....	7	....	8	.....	9	.....	10	....
8.0	....	56	1	53	2	49	3	45	4	41	5	38	6	34	7	30	8	26	9	23
8.5	....	53	1	46	2	39	3	32	4	25	5	18	6	11	7	4	7	57	8	50
9.0	....	50	1	40	2	30	3	20	4	10	5	....	5	50	6	40	7	30	8	20
9.5	....	47	1	35	2	22	3	10	3	57	4	45	5	32	6	19	7	7	7	54
10.0	....	45	1	30	2	15	3	....	3	45	4	30	5	15	6	.....	6	45	7	30

[a] One ampere per square decimeter requires 7.504 hours to deposit 0.1 mm of copper.

TABLE 11—Continued

Time Required for Copper Deposition from Acid-Sulphate Solutions—Continued

(*b*) IN CUSTOMARY UNITS

Current density used (amp./sq. ft.)	Time required to produce deposit of specified thickness (inches)																			
	0.001		0.002		0.003		0.004		0.005		0.006		0.007		0.008		0.009		0.010	
	Hrs.	Mins.	Hrs.	Mins.	Hrs.	Mins.	Hrs.	Mins.	Hrs.	Mins.	Hrs.	Mins.	Hrs.	Mins.	Hrs.	Mins.	Hrs.	Mins.	Hrs.	Mins.
1	17	43	35	25	53	8	70	50	88	33	106	16	123	58	141	41	159	23	177	6
5	3	32	7	5	10	38	14	10	17	43	21	15	24	48	28	20	31	53	35	25
10	1	46	3	32	5	19	7	5	8	51	10	38	12	24	14	10	15	56	17	43
15	1	11	2	22	3	32	4	43	5	54	7	5	8	16	9	26	10	37	11	48
20	....	53	1	46	2	39	3	32	4	26	5	19	6	12	7	5	7	58	8	52
25	....	42	1	25	2	7	2	50	3	32	4	15	4	57	5	40	6	22	7	5
30	....	35	1	11	1	46	2	22	2	57	3	32	3	8	4	43	4	19	5	54
35	....	30	1	1	1	31	2	1	2	32	3	2	3	32	4	3	4	33	5	4
40	....	27	....	53	1	20	1	46	2	14	2	39	3	6	3	32	3	59	4	26
45	....	24	....	47	1	11	1	35	1	58	2	22	2	46	3	9	3	32	3	56
50	....	21	....	42	1	4	1	25	1	46	2	7	2	29	2	50	3	11	3	32
55	....	19	....	39	....	58	1	17	1	37	1	56	2	15	2	35	2	54	3	13
60	....	18	....	35	....	53	1	11	1	29	1	46	2	4	2	22	2	39	2	57
65	....	16	....	33	....	49	1	5	1	22	1	38	1	54	2	11	2	27	2	43
70	....	15	....	30	....	46	1	1	1	16	1	31	1	46	2	1	2	17	2	32
75	....	14	....	28	....	42	....	57	1	11	1	25	1	39	1	53	2	8	2	22
80	....	13	....	27	....	40	....	53	1	6	1	20	1	33	1	46	2	0	2	13
85	....	12	....	25	....	38	....	50	1	2	1	15	1	27	1	40	1	52	2	5
90	....	12	....	24	....	36	....	47	....	59	1	11	1	23	1	34	1	46	1	58
95	....	11	....	22	....	34	....	45	....	56	1	7	1	18	1	29	1	41	1	52
100	....	11	....	21	....	32	....	42	....	53	1	4	1	14	1	25	1	36	1	46

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