

## ADDRESSES

### CHEMICAL PATENTS<sup>1</sup>—II

#### THE APPLICATION FOR AND PROSECUTION OF APPLICATIONS FOR UNITED STATES LETTERS PATENT

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As to the requisites for the application, specification and claims, Section 4888 of the Revised Statutes prescribes as follows:

"Before any inventor or discoverer shall receive a patent for his invention or discovery, he shall make application therefor, in writing, to the Commissioner of Patents, and shall file in the Patent Office a written description of the same, and of the manner and process of making, constructing, compounding, and using it, in such full, clear, concise and exact terms as to enable any person skilled in the art or science to which it appertains, or with which it is most nearly connected, to make, construct, compound and use the same; and in case of a machine, he shall explain the principle thereof, and the best mode in which he has contemplated applying that principle, so as to distinguish it from other inventions; and he shall particularly point out and distinctly claim the part, improvement, or combination which he claims as his invention or discovery."

The formal papers of an application comprise the petition, specification, oath, and, if the nature of the case admits it, drawings of the invention. The petition and oath are of stereotyped form and need not be considered further here.

The following order of arrangement should be observed in framing the specification:

- 1—Preamble stating the name and residence of the applicant and the title of the invention.
- 2—General statement of the object and nature of the invention.
- 3—Brief description of the several views of the drawings, if any.
- 4—Detailed description.
- 5—Claim or claims.
- 6—Signature of inventor, or if dead, of his executor or administrator.

The applicant must be the inventor personally or if insane, his guardian, or if dead, his executor or administrator. An application cannot be made by an assignee although the patent may subsequently be issued to an assignee.

A model or specimen of the thing invented will be required or admitted only as a part of the application when on examination of the case the Examiner of the Patent Office shall find it to be necessary or useful. As a rule models or specimens are neither asked for nor desired.

In discussing this general topic, we shall confine ourselves to applications for patents relating to chemistry—to compositions of matter and to chemical processes—taking up the general theory and requirements of applications for patents for compositions of matter and for chemical processes with illustrations from issued chemical patents, and using the history of the Charles M. Hall patent for the electrolytic production of aluminum as an illustration of the prosecution of an application through the Patent Office as well as an illustration of a form of patent for a chemical process.

In its essentials a specification of a composition case does not differ from that of any other class of invention. The specification must set forth the precise invention for which a patent is solicited and explain the principle thereof and the best mode in which applicant has contemplated applying that principle in such manner as to distinguish it from other inventions. A composition presents three essential subjects for description: the ingredients, the manner of combining them and the proper-

ties and nature of the resulting product. Each of the ingredients should be described in such manner that those skilled in the art may know exactly what is meant to be included. A great many applications are defective in this particular, owing to the use of indefinite and inaccurate terms in defining ingredients. This results in many cases from the loose use of chemical terms, such, for example, as using the word "soda" when referring to a salt or compound of sodium other than sodium carbonate, and it is difficult or impossible to tell from the context what particular compound is meant. When chemical substances are referred to, the safest rule is to designate them by their correct chemical names instead of their old or colloquial names. If thus defined or designated, or the formula stated, no question can thereafter arise as to what is meant. If a term has a definite accepted meaning in an art, though not strictly accurate, that is sufficient, since all that is required is that the substance be defined so that there is no question as to what is meant. Loose, indefinite terms are quite frequent in applications for paint compositions in such terms as "varnish," "drier," "japan," etc., and in alloy cases in such terms as "brass," "bronze," "white metal," to designate ingredients. When it is taken into consideration that there are many kinds of varnishes, japans, and driers and of brasses, bronzes and white metals, differing in composition and many of them in a great degree, it is seen that such terms do not meet the requirement of the rules as to definite disclosure. It would be possible to make up a composition having the ingredients named in such a description and in the same proportions, and yet the resulting composition would differ materially from that which the applicant had in mind, because his varnish, japan or drier, or his brass, bronze or white metal was different from the ones used by the other.

In specifying the ingredients none should be specified which is not essential to the composition. If any ingredient can be dispensed with it should be so stated in the description or no claim can properly be made for the composition with that ingredient omitted. Also the nature and number of the ingredients should be so stated that experiment is not necessary to determine what may be used.

In describing the manner of producing the composition, the proportions of the ingredients and the process of compounding them should be correctly and precisely stated so that one skilled in the art can produce it without the necessity of experiment.

It is not necessary that the theory of action of the ingredients be known or stated in the description, though if it is known it is preferable to state it.

The nature of the resulting composition should be defined by a statement of its peculiar physical or chemical characteristics if possible.

Usually compositions are devised for some particular use, such as a paint, fertilizer, explosive, etc., and the use or uses to which they are intended to be put should be set forth in the description.

In order to be patentable the composition must have utility. This may be assumed for some products such as new chemical compounds, but as to others the utility of the product should be specified.

The claims in composition cases often introduce questions which seldom or never arise in connection with the claims in machine and process cases, particularly the former. As a rule inventors devise or invent a specific composition to meet some definite want. The ingredients involved have to be mixed within certain limits or proportions or in a certain manner to get a composition which will have the desired properties. The description sets forth such a composition and too often says nothing

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about equivalents, whether or not proportions may be varied or whether or not some ingredient or ingredients may be omitted without any material change in the resulting product.

The person who has discovered that certain ingredients mixed in certain proportions will produce a certain composition of matter has not discovered every composition of matter which may be made from those ingredients nor is he entitled to the broadest possible claim for a composition containing those ingredients.

It is a good rule that in claims for compositions of matter in which proportions are essential the proportions be specified either directly or by reference to the specification, or the identifying and distinguishing characteristics of the composition should be stated in lieu of proportions. The latter is the better practice, though requiring more skill and care in drafting the claim, because it more fully protects the real invention.

Claims omitting ingredients which the specification does not state may be omitted are without basis in the case since they are not for the applicant's invention.

The omission of an essential ingredient makes a new composition. In a machine case whether an element is essential or not can be told by inspection. This, as a rule, is not so in composition cases, but only by experiment can the essentiality of ingredients be determined. Therefore, in the absence of a statement that it is not essential an element is considered essential unless it is obvious that it may be omitted, for example, one which is added for merely fanciful effect, such as a coloring or a flavoring ingredient. If an ingredient merely improves the composition and is not absolutely essential, claims may be drawn both including and omitting it. But even in such a case a proper basis for the omission of such an ingredient should be laid in the description.

If the specification lays a basis for the omission of certain ingredients the claims which do not include all the ingredients should be so worded as not to limit them to the ingredients enumerated therein.

In selecting terms to define ingredients of composition generically there is danger of choosing terms which are too comprehensive. In *Bracewell vs. Passaic Print Works*, 107 F. R., 467, the use of zinc oxide, hydrate, or carbonate seemed to be imperative to the success of the process. The applicant, however, stated that any zinc compound may be employed with good results, which statement was incorrect since it was proved that a large number of them could not be thus used. Judge Cox said:

"It cannot be contended that the patentee knew that his statement that any zinc compound would operate successfully was false, but it is manifest that he did not know that it was true and he should have known it was true before he inserted it in his description and made his corresponding claim. He stretched his net to catch as infringers all users of zinc compounds and if he stretched it to the breaking point he has only himself to blame. The courts should be liberal in construing patents, but they cannot rewrite the description and claims, they cannot construct an entirely new patent even to save a meritorious invention. If the complainant's contention be correct a patentee can claim blindly an entire group of compounds, relying on the court, after subsequent investigation and experiment, to limit the claim to the one which gives the best results. This will not do."

In *Matheson vs. Campbell*, 78 F. R., 920, it was alleged by the patentees that any sulfo-acid treated by their process would give a "color producing black" and that, therefore, all sulfo-acids were equivalents. Judge Lacombe ruled that:

"The inventors did not make any such 'broad discovery.' They made the specific discovery that some disulfo-acids treated according to their process would produce their product. The broad discovery that all sulfo-acids may be thus transformed

they certainly did not discover . . . since most of them cannot be thus transformed by the process of the patent. . . . We are referred to no authority and know of no principle which will sustain applicant's contention that he can thus . . . speculate on the equivalents of his claimed invention, and thereby oblige the public to resort to experiments in order to determine the scope of the claims of the patent."

In defining ingredients generically, by statement of characteristics common to the several specific substances intended to be included, the characteristics or properties relied upon should be essential ones and not mere incidental ones which may be had in common by the substances but which are immaterial in so far as the particular composition is concerned. For example, in a non-corrodible alloy in which gold, platinum and iridium may be used interchangeably on account of their resistance to oxidation, and their specific gravity is unimportant, to define them in the claim as "heavy metals" is improper, since, while they *are* heavy metals, it is not this property which makes them equivalent in the alloy. Such a claim does not properly protect the invention since some heavy metals readily oxidize, and the claim would not cover alloys in which difficultly oxidizable metals not of high specific gravity are used. Essential, not non-essential, properties should be specified in identifying the ingredients.

The specification for a process patent also follows the usual rules as to making a full, clear and exact disclosure of the invention. As the practice of the process may be performed by various means, the claims are not to be restricted to the practice of the process in connection with the specific means disclosed. It is not necessary to disclose *every* mode or all the types of apparatus which may be employed but one particular mode or apparatus by which the process can be carried out should be set forth clearly and completely.

And this one means should be the best means known to the inventor at the time of making his application. If the specification sets forth a way of actually practicing the process and producing the result claimed, the theory of the inventor as to the mode of operation, if incorrect, may be disregarded, and will not be taken as limiting the invention. As such statement of mere theory is not necessary it had therefore better be entirely omitted, the specification setting forth the observed facts only.

It is better to always make a very full and detailed description of the way of practicing the process, even to the disclosure of apparently unimportant details. As a guide you might say that the disclosure should be just as detailed as a report of an important laboratory experiment. You may generalize, both in specification and claim from a detailed statement. The rules of the Patent Office, however, do not permit you to make more specific, mere general statements. In other words, if you have omitted an important detail, even if your whole process turns upon it, you may not afterwards add it to your specification. You might say that the specification may be subtracted from but may not be added to. These remarks apply as well to applications for all patents. The exclusion of an important originally omitted statement on the ground that it is "new matter" frequently wrecks the whole case.

Again, you may not always be able to tell at the beginning just what are the essentials of a process. You do not know what the prior art may show and on what you must rely in order to distinguish the invention in question from such prior art. Range of temperature, properties of substances employed, proportions of ingredients, pressures and all these and more may turn out to be of the utmost importance, yet if you have not made a definite statement in your specification you cannot claim it nor distinguish the invention from the prior art which may otherwise be a disclosure.

A process claim should not be for the *use* of a thing. Such claims are held to be invalid.

For example, in the famous ether case of many years ago wherein the use of ether as an anesthetic by inhalation of its vapors was claimed, the Court said:<sup>1</sup>

"It may not be amiss to speak of the character of the discovery upon which the patent is founded. Its value in securing insensibility during the surgical operation, and thus saving the patient from sharp anguish while it is proceeding, and mitigating the shock to his system, which would otherwise be much greater was proved on the trial by distinguished surgeons of the city of New York. They agreed in ranking it among the great discoveries of modern times; and one of them remarked that its value was too great to be estimated in dollars and cents. Its universal use, too, concurs to the same point. Its discoverer is entitled to be classed among the greatest benefactors of mankind. But the beneficent and imposing character of the discovery cannot change the legal principles upon which the law of patents is founded, nor abrogate the rules by which judicial construction must be governed. These principles and rules are fixed, and uninfluenced by shades and degrees of comparative merit. They secure to the inventor a monopoly in the manufacture, use, and sale of very humble contrivances of limited usefulness, the fruits of indifferent skill, and trifling ingenuity, as well as those grander products of his genius which confer renown on himself, and extensive and lasting benefits on society. But they are inadequate to the protection of every discovery, by securing its exclusive control to the explorer to whose eye it may first be disclosed. A discovery may be brilliant and useful, and not patentable. No matter through what long, solitary vigils, or by what importunate efforts, the secret may have been wrung from the bosom of Nature, or to what useful purpose it may be applied, something more is necessary. The new force or principle brought to light must be embodied and set to work, and can be patented only in connection or combination with the means by which, or the medium through which it operates. Neither the natural functions of an animal upon which or through which it may be designed to operate, nor any of the useful purposes to which it may be applied, can form any essential parts of the combination, however they may illustrate and establish its usefulness."

Obviously, the claim must be limited to some art involving a particular material or group of analogous materials. For example, the process of heating a *substance* to 1000° Centigrade to melt it, and then crystallizing it by cooling to 800° could be applied neither to water nor to platinum. The process is necessarily limited in scope to a few particular materials, and should not be claimed in any broader application.

Of course, one may claim his invention as broadly as the limits of the art allow, provided the claim is not broader than the original disclosure, which disclosure, in turn, must be sufficiently clear that one skilled in the art may be able to utilize the same. For example, a method involving coating canes with varnish and subjecting to a temperature of 240 to 300 degrees for not less than 3 hours was held void, because the limits of temperature stated were so wide apart that the process could not be carried out throughout the whole range claimed; hence, the claim was too broad in the sense of being inaccurate, vague and indefinite.

A patentable process claim must include all steps necessary to produce the stated or any useful result.

This seems to be clear, following the precedents of the decisions covering claims to structure. Each claim must define an operative process, however broadly stated. For example, in making a battery plate involving the four steps of pasting lead oxide on a grid, treating it with sulfuric acid, drying it and then electrolyzing as a cathode in a suitable bath to form a

spongy lead electrode—if these four steps are all essential steps in making the electrode, then the first two steps alone, for example, are not patentable, since a finished and useful product is not produced thereby.

Of course, one is entitled to subprocess claims where they can be properly drawn.

An applicant may properly in one case have claims covering the principal or essential steps of his process, and other claims including these steps together with other specific steps which are not absolutely necessary to the performance of the process, but which add to its efficiency or make its operation more perfect.

For example, a claim omitted the intermediate step, in a process of making sugar, of "agitating and reducing the temperature." It was contended and held that the omitted step was not essential to the performance of the process, although it was an improvement and rendered the process more practical.

Similarly, in the example covering the making of a battery plate referred to, if the step of drying the plate is not essential, but merely works toward greater efficiency and a better product it may be left out of a broad claim. The claim without this additional step covers a complete, useful and operative invention by itself, and therefore is a true subprocess claim under the specific process. As a rule a process claim should be expressed independently of the specific structure of the article treated and independently of the structure of the apparatus in which the process is carried out. So far as the steps of the process are concerned, it is clear that it is immaterial whether a battery grid, or plate, for example, is made of woven lead wire gauze or of a reticulated lead plate made of V-shaped bars cast together, since the steps of pasting, treating with acid, drying and electrolyzing can be carried out with the one electrode structure as well as with the other. Similarly, if the claim specified "applying the lead oxide paste with a *trowel* and treating the electrode with acid in a *rubber-lined wooden tank*," it would be objectionable. These structures have no patentable relation to the process steps.

Again, it was held that "it is better form in a method claim to state that a mixture is heated at a moderate temperature than to state that the same is heated over a water bath."

This rule cannot always be applied, however, in individual cases, especially in mechanical processes. Here the best we can say is that whatever structure is essential should be set forth to the minimum extent and only in the broadest possible terms, since no patentability is involved over the broad proposition in specifying particular structure.

Coming now to certain patents which we shall consider as illustrating the application of the principles heretofore set forth, showing different forms of statement in the specifications and different modes of claiming both compositions of matter and processes, together with other points which will be referred to in connection with the particular illustration, let us examine

(a) The "Calcium Carbide" Patent number 541,138, issued to Thomas L. Willson on June 18, 1895.

(b) The "Aspirin" Patent number 644,077, issued to Felix Hoffman, assignor to the Farbenfabriken of Elberfeld Company, on February 27, 1900.

(c) One of the "Adrenalin" Patents, number 753,177, issued to Jokichi Takamine, on February 23, 1904.

(d) Patent number 977,053, issued to Ludwig Taub, assignor to the Farbenfabriken on Nov. 29, 1910, for an amid of beta-beta-diethyl-propionic acid.

(e) Patent number 1,053,300, issued to Georg Korndörfer and Baptist Reuter, assignors to Farbwerke vormals Meister, Lucius & Brüning on Feb. 18, 1913, for derivatives of diamino-dioxyarsenobenzene and

(f) The Reissue of the latter patent.

(g) Hamilton Young Castner's Patent for the process of

<sup>1</sup> Morton vs. N. Y. Eye Infirmary, F. C. No. 9865.

manufacture of sodium and potassium, No. 452,030, issued May 12, 1891, and

(h) Charles M. Hall's Patent for the process of reducing aluminum by electrolysis, No. 400,766, issued April 2, 1889.

(a) THE "CALCIUM CARBIDE" PATENT

This patent describes the manufacture of commercial calcium carbide in which finely divided coke and pulverulent lime, mingled together, are subjected to the action of an alternating current in an electric furnace. A drawing illustrates the furnace diagrammatically in vertical section.

The patent reads as follows:

"This invention relates to the production of a new form of crystalline calcium carbide.

"Before my invention calcium carbide has existed in an amorphous condition, due either to the method of its preparation, or to the impurities contained in it.

"By my invention herein described, calcium carbide is produced in a new form, namely, in crystalline condition, having a bluish or purplish iridescence. The carbide so existing is in a condition particularly applicable, on account of its purity, for conversion into other compounds.

"In order to produce this product, I proceed as follows: I take very finely divided coke and lime, mechanically reduced to a pulverulent condition, and I mingle them thoroughly mechanically in the proportion of thirty-five per cent of coke and sixty-five per cent of lime. I then subject them to the action of the electric current in a furnace, such as that shown in the accompanying drawing, which is a vertical section, and in which

"A represents an inclosing brick work; B, an internal lining, preferably of carbon, which, though preferable, is not in all cases necessary; C, one of the conducting poles, by preference of broken carbon; D, a removable pole of compacted carbon; E, a tap hole for removing the melted product, if desired; F, adjusting mechanism for raising the carbon pole D; and G an alternating current dynamo, which may, therefore, be made commutatorless.

"In starting this furnace, the carbon pole D and the conducting bodies C are connected to the poles of an alternating current dynamo having a mean potential, say, of fifty-five volts, and sufficient amperage to produce the amount of material required, having regard to the size of the furnace. Using, for instance, a pole having eight inches on the side, an amperage of about fifteen hundred is desirable. Under these conditions, by reason of the alternation of the current, a feeding in of the mingled lime and carbon between the poles is effected to an extent which does not occur when a direct current is used. Furthermore, it has been generally understood that the production of calcium carbide was due to an electrolytic action and not due to a smelting action. I have demonstrated that the action is purely a smelting action, under the conditions of the process here described. Furthermore, that the block of molten calcium carbide which forms beneath the elevating pole is itself a good conductor of electricity and can be built up from the bottom to any desired height without preventing the operation of the process. A height, for instance, of two or more feet is entirely practicable in the apparatus above described. If preferred, of course, the fused calcium carbide may be tapped out as the same is formed, in the ordinary way, as applied to iron and steel. The liquid calcium carbide thus produced, when allowed to cool, crystallizes into the form above described, and when broken exhibits the iridescent surfaces above named. By this process, likewise, the yield of calcium carbide per electric horse power is almost doubled over a process of using a direct current, which is, evidently, a matter of the utmost importance, while, at the same time, the process proceeds uniformly and without break, by reason of the substantial uniform feeding in of the material.

"It is essential, in order to produce the new material here described, to have the lime and coke very finely divided. This is done by a powdering machine, and then the powdered lime

and coke are thoroughly mingled by a grinder or mixer so as to bring these bodies into most intimate contact. The action of the alternating current is likewise essentially different from that of a direct current, producing a series of explosions, the effect of which seems to be to feed the pulverized material into the arc. This is very important in accomplishing a rapid and uniform conversion.

"What I claim as my invention, and desire to secure by Letters Patent, is

"As a new product, crystalline calcium carbide existing as masses of aggregated crystals, substantially as described."

It will be noted that the patentee sets forth in the order given: (1) the object of his invention; (2) the prior art; (3) the properties of his new product; (4) a general outline of his manner of producing his product; (5) the essentials of his process; and (6) his claim.

The use of the words "preferable," "necessary" and "for instance" should be noted as implying what is and what is not essential and what is purely illustrative.

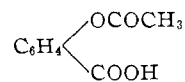
The distinctive property of the product over the art, viz., "crystalline" as contrasted with "amorphous," is the basis of the claim. The property of "having a bluish or purplish iridescence" is incidental rather than essential and is therefore omitted in the claim.

It will be noted that the word "crystalline" in the first paragraph had better been omitted—the invention related to a new form of calcium carbide, not of crystalline calcium carbide. "Crystalline" calcium carbide was what was new, and there was no previous form of "crystalline" calcium carbide.

(b) THE "ASPIRIN" PATENT<sup>1</sup>

This patent describes the making of a compound having certain properties which distinguish it from the prior art.

"In the *Annalen der Chemie und Pharmacie*, Vol. 150, pages 11 and 12, Kraut has described that he obtained by the action of acetyl chlorid on salicylic acid a body which he thought to be acetyl salicylic acid. I have now found that on heating salicylic acid with acetic anhydride a body is obtained, the properties of which are perfectly different from those of the body described by Kraut. According to my researches the body obtained by means of my new process is undoubtedly the real acetyl salicylic acid,



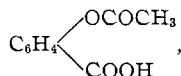
Therefore the compound described by Kraut cannot be the real acetyl salicylic acid, but is another compound. In the following I point out specifically the principal differences between my new compound and the body described by Kraut.

"If the Kraut product is boiled even for a long while with water (according to Kraut's statement), acetic acid is not produced, while my new body when boiled with water is readily split up, acetic and salicylic acid being produced. The watery solution of the Kraut body shows the same behavior on the addition of a small quantity of ferric chlorid as a watery solution of salicylic acid when mixed with a small quantity of ferric chlorid—that is to say, it assumes a violet color. On the contrary, a watery solution of my new body when mixed with ferric chlorid does not assume a violet color. If a melted test portion of the Kraut body is allowed to cool, it begins to solidify (according to Kraut's statement) at from 118° to 118.5° Centigrade, while a melted test portion of my product solidifies at about 70° Centigrade. The melting points of the two compounds cannot be compared, because Kraut does not give the melting point of his compound. It follows from these details that the two compounds are absolutely different.

"In producing my new compound I can proceed as follows:

<sup>1</sup> Kuehmsted vs. Farbenfabriken, 179 F. R., 701.

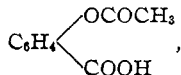
(without limiting myself to the particulars given): A mixture prepared from fifty parts of salicylic acid and seventy-five parts of acetic anhydride is heated for about two hours at about 150° Centigrade in a vessel provided with a reflux condenser. Thus a clear liquid is obtained, from which on cooling a crystalline mass is separated, which is the acetyl salicylic acid. It is freed from the acetic anhydride by pressing and then recrystallized from dry chloroform. The acid is thus obtained in the shape of glittering white needles melting at about 135° Centigrade, which are easily soluble in benzene, alcohol, glacial acetic acid, and chloroform, but difficultly soluble in cold water. It has the formula



and exhibits therapeutical properties.

"Having now described my invention and in what manner the same is to be performed, what I claim as new, and desire to secure by Letters Patent, is

"As a new article of manufacture the acetyl salicylic acid having the formula



being when crystallized from dry chloroform in the shape of white glittering needles, easily soluble in benzene, alcohol and glacial acetic acid, difficultly soluble in cold water, being split by hot water into acetic acid and salicylic acid, melting at about 135° Centigrade, substantially as hereinbefore described."

The patentee sets forth the nearest prior art product and compares its properties with the properties of his new product, showing that the two compounds are different. He then describes the process by which he prepares his new product and sets forth the characteristic properties of his new product including the structural formula.

The claim describes the product by the use of its structural formula and by a statement of characteristic properties. The enumeration of characteristic properties in the claim should be especially noted as a good form for such a claim.

#### (c) THE "ADRENALIN" PATENT

This patent relates to a substance consisting of the blood-pressure-raising principle isolated from the suprarenal glands chemically combined with a nonsuprarenal substance whereby the stability of a water solution of said principle is maintained.

It is cited because it contains a *Disclaimer* "to so much of claims 3 and 4 as cover the benzoyleated compounds."

Claims 3 and 4 read as follows:

"3—As a new article of manufacture, a substance having the properties of the hemostatic, astringent and blood-pressure-raising principle of the suprarenal glands substantially free from non-blood-pressure-raising constituents thereof, which is soluble in water and the water solution of which is practically inert to the oxygen of the air and gives a green coloration with ferric chlorid and a red coloration with iodine.

"4—As a new article of manufacture a compound of the crystallizable blood-pressure-raising constituent of the suprarenal glands substantially free from the non-crystallizable constituents thereof which is soluble in water and which when in water solution is practically inert to the oxygen of the air and gives a green coloration with ferric chlorid and a red coloration with iodine."

The law provides that whenever, through inadvertence, accident or mistake, and without any fraudulent or deceptive intention, a patentee has claimed as his invention or discovery more than he had a right to claim as new "he may make disclaimer of such part of the thing patented and thereby render valid the part which is truly and justly his own."

In a case concerning this adrenalin patent<sup>1</sup> one of the refer-

<sup>1</sup> Parke-Davis & Co. vs. Mulford, 189 F. R., 95; 196 F. R., 496.

ences related to a certain monobenzoyleated salt physiologically active comparably with adrenalin. This was the salt of a base constituted in part by the atoms which together make up the active principle of adrenalin and for the remainder by a benzoyl radical. The Court found that Takamine was therefore not the first to broadly discover a stable and pure salt having the physiological activity of the suprarenal gland and held claims 3 and 4 to be anticipated on the broad construction. Hence Takamine disclaimed the broad construction as we have seen thus in so far validating the claims held to be invalid.

#### (d) TAUB PATENT NO. 977,053

This describes the making of and claims by its characteristic properties the amid of beta-beta-diethylpropionic acid. The specification sets forth succinctly the process and the properties of the resulting product, giving the equations according to which the reaction proceeds. It is a good example of a well-drawn specification.

The salient features of the specification read as follows:

"The new product is a crystalline compound soluble in hot alcohol and hot benzene. When reacted upon by hot alkalies it is split up into the beta-beta-diethylpropionic acid and ammonia.

"In carrying out my process practically I can proceed as follows (the parts being by weight): 390 parts of beta-beta-diethylpropionic acid are heated to 100° C. with 140 parts of PCl<sub>3</sub>, the product of the reaction is separated from the phosphoric acid by decanting of the formed acid chlorid and the chlorid is distilled *in vacuo*. The chlorid of beta-beta-diethylpropionic acid is then treated with an excess of ammonia, the amid is filtered off and crystallized from benzene."

Another reason for referring to this patent is that it contains the statement:

"The present application is a division of my application serial No. 523,805, filed October 21, 1909," *i. e.*, an earlier application.

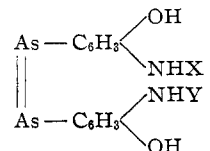
If several inventions claimed in a single application are of such nature that a single patent may not be issued to cover them, two or more separate inventions not being claimable in one application, the inventor is required to limit himself to whichever invention he elects, the other inventions being made the subject of *divisional* applications. He must file a new separate application for each division but the filing date of the original or parent application is taken as the filing date of the division.

#### (e), (f) KORNDÖRFER PATENT NO. 1,053,300

This patent relates to improvements in derivatives of diaminodioxarsenobenzene and is referred to, first, because of the form of the claims, and second, because it has been reissued.

When the original patent is inoperative or invalid by reason of a defective or insufficient specification, or by reason of the patentee claiming as his invention or discovery more than he had a right to claim as new, provided the error arose through inadvertence, accident, or mistake, and without any fraudulent or deceptive intention, the patent may be surrendered and a "reissue" granted in its place to run for the remainder of the life of the original patent.

In this case the original patent stated that the general formula of the compound was



wherein "X" and "Y" stand for "the residue known as methylene-sulfonic acid (—CH<sub>2</sub>SO<sub>2</sub>H) and "X" may be replaced by hydrogen, whereas the Reissue states that either "X" or "Y" may be replaced by hydrogen.

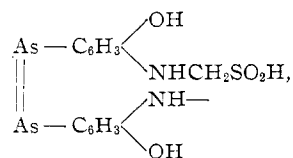
In accordance with the original statement the claims of the

original patent covered the acid *per se* and the acid radical in but one form while in the Reissue (No. 13,848) the claims cover the acid radical in two forms and the sodium salt specifically.

A comparison of claim 2 of the original with claim 2 of the Reissue will indicate the line of difference, a different form of the radical being claimed in each case.

Claim 2 of the original reads:

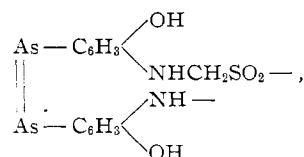
"2—As new products, the derivatives of diaminodioxarsenobenzene, the radicals of which have the general formula



said derivatives being yellowish powders, difficultly soluble in water, insoluble in alcohol, ether and acetone, dissolving in alkalis with formation of salts, these alkali-salts being yellowish powders readily soluble in water."

Claim 2 of the Reissue reads:

"2—As new products, the derivatives of diaminodioxarsenobenzene, the radicals of which have the general formula



the acid derivatives being yellowish powders, difficultly soluble in water, insoluble in alcohol, ether and acetone, dissolving in alkalis with formation of salts, these alkali salts being yellowish powders readily soluble in water."

It will be noted that, besides the change in the radical, the word "acid" is inserted before "derivatives" in the Reissue claim. The error of statement in the original was thus corrected and the damage repaired.

The rules for drawing up a specification and claims for a process patent do not differ materially from those heretofore given when considering product patents. The apparatus used is nearly always capable of illustration by drawings, either diagrammatic or of the actual apparatus.

I shall call your attention to but two process patents.

#### (g) THE CASTNER PATENT

This patent (No. 452,030 of May 12, 1891) is for a process of manufacturing sodium and potassium from caustic soda or potassa without distillation by treating the caustic alkali, while constantly maintained at a temperature of not more than 20° C. above its melting point, to the action of the electric current.

The specification is illustrated with a drawing showing in section an electric furnace or cell suitable for the process. The specification first sets forth the prior art, the reasons for the commercial failure of the processes of the prior art and outlines the process of the patent and the reasons for its commercial success. The particular feature accentuated is that the processes of the prior art were not and had never been proved to be commercially practicable while the process of the patent possessed this characteristic. The subsequent commercial success of the Castner process proved that these assertions were well founded. The law recognizes a patentable difference between a laboratory experiment not capable of commercial utilization or a bare statement of a scientific fact and a commercially successful process. It is clear that the latter would require more than the average expected skill of one skilled in the art in order to bring it into being, even starting from the previous disclosures.

Having set forth the foregoing statements the specification

then describes the process, with reference to the drawing, setting forth a practical means of practicing the process, even giving specific directions as to size and as to the amount of current required in a typical apparatus.

It is unusual in a patent drawing to have it drawn to scale but when it is done and the process described as having been actually carried out, the argument of inoperativeness cannot be made against it. This was particularly valuable in a case like the present where the argument as to patentability rested on the commercial inoperativeness of the prior art.

The frequent use, throughout the specification, of such words as "preferably," "suitable," "as described," and "conveniently" indicates that the matter set forth is subject to variation and is not essential to the invention in the form described. These words are in sharp contrast to the feature which is said to be "necessary," *viz.*, the limit of temperature which should not be exceeded.

The specification concludes with the statement that "I do not of course confine myself to the particular form of apparatus shown, but simply describe it as one of the forms which may be employed," thus bringing out the fact that his descriptive statement is illustrative only and not limiting.

The claims are but two in number, illustrating that a great number of claims is not always necessary to adequately protect an invention. The claims read as follows:

"1—The hereinbefore-described process of manufacturing the alkaline metals, which consists in treating the caustic alkali while constantly maintained at a temperature of not more than 20° Centigrade above its melting point to the action of the electric current, substantially as described.

"2—In an apparatus for the manufacture of alkaline metals, the combination, with positive and negative electrodes, of the gauze or screen interposed between said electrodes and a superposed vessel or dome for collecting the separated metal, substantially as set forth."

#### (h) THE HALL PATENT

This patent (No. 400,766, April 2, 1889) is the basic patent which led to the present electrolytic production of aluminum: to that wonderful commercial success which reduced the price of aluminum from \$12 a pound to 19 cents and which was instrumental in building up a business from practically nothing to a production of 160,000,000 pounds a year, giving employment to 45,000 men in its various branches and involving an invested capital of \$175,000,000. Of course this was not all due to this particular patent but this patent was the root of the industry.

I have chosen this patent to illustrate Patent Office procedure and the history of a Patent through the Courts.

Hall made his first button of aluminum according to this process on February 23, 1886, less than a year after he was graduated from Oberlin College and when he was but two months and a half past 22 years of age. He filed his patent application July 9, 1886.

When an application is filed in the Patent Office it is given a Serial No. and a date of filing. Hall's application received the application serial No. 207,601. The Patent Office, for the examination of applications, is divided into 43 divisions. Those relating primarily to applications for chemical products and processes are Division 2, Medicines; Division 3, Electrochemistry-Metallurgy; Division 6, Bleaching and Dyeing; Chemicals; Explosives; Fertilizers; Liquid Coating Compositions; Plastic Compositions; Preserving; Sugar and Salt; Substance Preparation; and Division 31, Alcohol; Ammonia, Water and Wood Distillation; Mineral Oils; Oils, Fats and Glue, etc. The Hall application would fall into and be assigned for examination to Division 3, Electrochemistry-Metallurgy, where it would be filed to await its turn for examination, the applications coming up *seriatim* in order of date of filing.

The prior art U. S. patents are all classified into classes and sub-classes, the patents of each sub-class being kept in separate compartments so that they may be readily inspected in the course of a search. As the issued U. S. patents now number upwards of 1,125,000 the search is a formidable affair in any conspicuous art. In addition to the U. S. patents, the Examiners of each Division have a classified, and more or less complete, set of certain foreign patents, generally only the English, French and German as well as a rather incomplete reference list to literature other than patent publications. With the present equipment of the Patent Office it is practically impossible, both from the standpoint of the references at hand, and from the standpoint of time and men to make the searches, to make a complete and final search in any art. The wonder is that the hard-working and hard-worked officials of the Patent Office do as well as they do.

When the application comes up for examination, if correct as to form, a search is made and the applicant (or his attorney) is informed by mail of the result. This result is almost invariably a rejection of some or all of the claims on the references named and it is then the duty of the applicant to amend his application and to distinguish, if he can, his invention from what is shown in the references cited. This procedure goes back and forth until either the application based on the amended claims is allowed or finally rejected as being unpatentable. If finally rejected by the Primary Examiner an appeal may be taken to the Board of Examiners-in-Chief; from them to the Commissioner of Patents and from him to the Court of Appeals of the District of Columbia. After this a still further appeal lies to the U. S. District Court with an appeal from it to the U. S. Circuit Court of Appeals.

Hall, in his original application, set forth his invention, generically as follows:

"My invention consists in a process and apparatus for the reduction of aluminum by electrolysis from alumina dissolved in melted anhydrous salts or compounds; and the means employed are:

"First, suitable solvents for alumina in the form of chemical compounds and combinations which fuse at a moderate heat and, when melted, dissolve alumina so as to bring it into a liquid or dissolved condition which will admit of electrolysis.

"Second, suitable crucibles and receptacles for containing the solvents and dissolved alumina at a melting temperature; and

"Third, suitable electrodes or poles for the purpose of transmitting electricity through the fused and liquid mass in order to reduce from alumina the metal aluminum."

He then describes his solvent, stating the proportions of fluorides of aluminum actually used by him and that cryolite possesses the qualities desired, describing how he proceeds to melt the solvent, dissolve the alumina therein and reduce the aluminum therefrom.

The manner in which the invention may be carried out is described by reference to a drawing, particular attention being paid to the construction of the crucibles and of the electrodes.

As to the prior art he says:

"I am aware that aluminum has been produced by the electrolysis of fused masses containing the chloride of aluminum or other haloid compounds of that metal; and I am aware that in these operations it is alleged that rods or plates made of alumina and carbon have been introduced into the crucibles so as to be acted on by the evolved chlorine or other gas and thereby regenerate the chloride or other corresponding compound so as to produce aluminum from alumina indirectly.

"But my device differs from the above mentioned in that melted solvents are employed to dissolve alumina, and that alumina is added in a condition to be readily dissolved by and in the melted mass, and being dissolved is acted upon and decomposed in solution by electricity; and thus aluminum is re-

duced or liberated from alumina dissolved in melted anhydrous compounds or salts. . . . I believe that I am the first to produce aluminum from solutions of alumina dissolved in melted anhydrous compounds or salts."

The original application contained 16 claims of which 1-10 inclusive were directed to the process and 11-16 to the apparatus. The issued patent contains but three claims, all relating to the process, the first claiming a bath of "fluorides of aluminum and a metal more electro-positive than aluminum" without reference to the anode; the second being restricted to the fluoride of sodium as the metal more electro-positive than aluminum and to a carbonaceous anode, and the third like the second excepting that the fluoride of lithium is added to that of aluminum and sodium.

Let us put ourselves in the mental attitude of Hall at the time of filing his application. He had dreamed about and worked upon the problem of how to produce aluminum cheaply even when he was a boy in the high school and all through his college course he had kept this goal in sight. Now after all these years and all his work, in the enthusiasm of youth, he had reached his goal. He believed himself to be the first to reach the goal, the first, as he says in his application "to produce aluminum from solutions of alumina dissolved in melted anhydrous compounds or salts" so that the alumina may be decomposed electrolytically.

What a shock he must have had when he received the first action from the Patent Office on October 28, 1886, after nearly four months of impatient waiting, stating that all his process claims seemed to be anticipated by a French patent—that to Héroult, for electrolyzing a solution of alumina in molten cryolite, the very thing which he had done specifically, and that all his claims for apparatus were likewise anticipated upon various cited U. S., English and French patents.

It was furthermore stated that the apparatus claims should be divided out and submitted in a new application and that the process claims 2-9 set forth such modifications of the generic process that they were inadmissible in the case, which should comprise but the broad generic process and one specific modification.

But it was not as bad as first sight would make it seem. In the first place the Héroult French patent was dated April 23, 1886, and we have seen that the disclosure in a foreign patent is not to be considered unless the subject matter is patented prior to the making of the invention in question or more than two years prior to the filing of the U. S. application for patent. Now the Héroult patent was dated less than three months prior to the filing of Hall's application and we have seen that Hall made his first button of aluminum according to his invention on February 23, 1886, just two months before the issue of the French patent.

Accordingly Hall took advantage of this rule of law and, as prescribed by the Rules of Practice of the Patent Office, filed an affidavit setting forth, among other formal requirements, that he had completed his invention in the United States before the date of the French patent. This disposed of Héroult for the time being.

He then amended his application by striking out all of his original claims and substituting 9 new claims in place of the original 16, 4 of the new claims relating to the process and 5 to the apparatus, the requirement for division as to the apparatus claims having in the meantime been taken up to the Commissioner of Patents on appeal and the Examiner's holding on the point reversed.

To this amendment the Examiner replied:

"The claims presented in this application are objectionable, being so broad as to include subject matter substantially different from that described in the specification. Rule 37 requires



the claims to set forth specifically the actual improvement described.

"Applicant's invention consists in fusing a compound composed of the fluorides of aluminum and sodium, with or without the fluoride of lithium, adding alumina to the same, and passing a current of electricity through the fused mass, and the claims should be restricted accordingly. With reference to the apparatus claims, it is denied that there is any combination between the vessel and its contents. These claims are held to be mere aggregations of well-known apparatus. Claim 7 is objected to, as alternative in form, and, as presented, covers merely an oxidized copper plate. If applicant desires to cover the use of copper anodes in the process described, he should present a claim giving the steps of the process above quoted, specifying the use of copper anodes therein."

The new apparatus claims were further objected to as being informal. For example, new claim 5 reads as follows:

"In apparatus for the reduction of aluminum by electrolysis, the combination of a metal receptacle lined with carbon, a solution of alumina dissolved in a melted anhydrous salt or compound contained in said receptacle, and means for passing a current of electricity through the said solution, substantially as and for the purpose described."

Concerning these claims the Examiner said "The clauses in the various apparatus claims describing the process are thought to be unnecessary and should be cancelled. The various parts of the apparatus should not be described by their functions in the process, but by their construction and relation to the other parts of the apparatus."

New claim 7 objected to as being alternative in form and, as presented, covering merely an oxidized plate, which was not patentable read as follows:

"In apparatus for the reduction of metals by electrolysis, an anode of copper or its equivalent coated with oxide or its equivalent which at a red heat protects the underlying metal and transmits an electric current, substantially as described."

To this latter Hall made reply:

"The Examiner says that my 7th claim as amended February 2, 1887, covers only an oxidized copper plate. I would like to know what he means and what he wishes. An *anode* composed of an oxidized copper plate is new. An oxidized copper plate is old. It cannot be used in aqueous solution as the copper oxide is an insulator at ordinary temperature. Such an anode is an anode only when used as such and at a red heat, or the temperature of the melted compound."

And to this the Examiner rejoined:

"The Examiner means just what was said, that the 7th claim is alternative in form, and, as presented, covers merely an oxidized plate. Applicant admits that an oxidized copper plate is not, *per se*, new, and accordingly not patentable. The mere statement that it is used in an apparatus for electrolyzing aluminum does not add any new feature which invests it with patentable novelty. As stated, it must be claimed as one element of a combination, which as a combination, is new and patentable. The claim as drawn appears to be an attempt to cover any metal plate which becomes covered with oxide, and can be used as an electrode at the temperature employed, which is more than the applicant is fairly entitled to."

Hall responded to this by again cancelling his claims, submitting 14 claims in place of the 9 cancelled, of which 14 claims 1, 2 and 3 were broadly for the process; claims 4 to 7 were an attempt to protect his oxidized copper anode; claim 8 an attempt to protect a carbon-lined metal receptacle as a cathode and claims 9-14 for the apparatus.

In the 18th communication between the applicant and the Patent Office, dated April 9, 1887, the Examiner replies to this last amendment of Hall's as follows:

"Applicant's apparatus consists of an anode of oxidized copper,

and a carbon-lined iron crucible used as a cathode, with means for heating the same. The applicant admits that the oxidized copper plate is not *per se* new, the iron carbon lined vessel is old, the carbon cathode is old and the apparatus is held to be a mere aggregation anticipated by the references of record. . . . There seems to be an unnecessary multiplicity of claims. The apparatus claims are informal. The words 'with or without lithium' make the claims in which they appear alternative. Claims 4, 5, 6, 7 and 8 cover the 'use' of various parts of the apparatus, which use is not patentable. . . . The claims describing an 'anode of oxidizable metal coated with a protecting compound, which at red heat transmits an electric current' are functional. . . . Claims 11, 12 and 13 which introduce the solution as one element of the combination are improper. There seems to be no combination between the vessel and its contents."

To this Hall replied again, for a third time cancelling all of his claims, this time submitting eight claims instead of the fourteen cancelled, of which eight claims 1-3 related to the process and claims 4-8 to the apparatus.

He does not seem to have changed his process claims much during his various amendments and substitutions but to have directed his efforts mostly towards the apparatus. One cannot but wonder whether, at this stage of the proceedings, he did not regard the apparatus claims as the more important. We have seen that the various parts of his apparatus were all old and that the essence of his invention consisted in fusing a compound composed of the fluorides of aluminum and of a metal more electropositive than aluminum, dissolving alumina therein and passing an electric current through the fused mass. It may be that at this point of time Hall himself did not appreciate that the process, regardless of the form of apparatus used, was the broad and valuable invention.

However that may be, the Patent Office again rejected all the apparatus claims and the patent finally issued without any such. One of the peculiar things about the case is that although Hall made such a fight over his oxidized copper anodes, when his claims were finally formulated the only ones referring to anodes were for carbonaceous anodes, no written explanation appearing in the file for the sudden change. This point was afterwards brought up in the litigation over this patent.

The claims as finally issued read as follows:

"1—As an improvement in the art of manufacturing aluminum, the herein-described process, which consists in dissolving alumina in a fused bath composed of the fluorides of aluminum and a metal more electro-positive than aluminum, and then passing an electric current through the fused mass, substantially as set forth.

"2—As an improvement in the art of manufacturing aluminum, the herein-described process, which consists in dissolving alumina in a fused bath composed of the fluorides of aluminum and sodium, and then passing an electric current, by means of a carbonaceous anode, through the fused mass, substantially as set forth.

"3—As an improvement in the art of manufacturing aluminum, the herein-described process, which consists in dissolving alumina in a fused bath composed of the fluorides of aluminum, sodium, and lithium, and then passing an electric current, by means of a carbonaceous anode, through the fused mass, substantially as set forth."

About the time of the last above-referred-to action by the Patent Office, an interference was declared between the application of Hall and one which had been filed by Héroult, the patentee of the French patent which we have seen Hall had overcome by affidavit in the first instance.

An interference is a proceeding instituted by the Patent Office for the purpose of determining the question of priority of invention between two or more parties claiming substantially the same patentable invention. The Patent Office first formulates



a "declaration of interference" setting forth and defining the issue in interference; the parties are then each required to file an affidavit or "preliminary statement" setting forth the date of conception, disclosure and reduction to practice, if any, of the invention. If the invention has not been actually reduced to practice, the date of filing the application is taken as the date of constructive reduction to practice. The parties then take their testimony to support their respective cases, the limits of time within which the testimony of each is to be taken, being fixed by the Patent Office, but the exact time within such limits and the place of taking the testimony being fixed by the party himself upon due notice to his adversary who is entitled to attend and cross-examine the witnesses produced. The testimony is generally taken by question and answer upon a typewriting machine or stenographically, before a Notary Public, who swears the witnesses.

The testimony of each is then printed and the case argued orally upon printed briefs before the Examiner of Interferences, who sits as a Judge in such cases. From his decision there is an appeal to the Board of Examiners-in-Chief, a tribunal of three members; from them an appeal lies to the Commissioner of Patents; from him to the Court of Appeals of the District of Columbia. If the litigant is still unsatisfied he can bring the case up afresh in the District Court of the United States whence an appeal lies to the United States Circuit Court of Appeals.

But Hall did not have to go through with all this because his interference was with one who had made his invention abroad and in such case, in the absence of a printed publication issued prior to the date of the U. S. invention or more than two years prior to the filing of the application therefore, the only thing that counts is, when was the foreign invention introduced into this country. And as the date of Héroult's application was later than February 23, 1886, the date of Hall's actual reduction to practice, the interference was decided in Hall's favor.

After this Hall once again cancelled his specification and claims and submitted new ones, the patent finally issuing with the claims as above quoted.

Altogether there were over 30 communications between the inventor and the Patent Office. Neither the final specification nor the claims bear any resemblance to the original specification and claims. Yet the germ of a great invention was present at the beginning and the final form, as we shall see later, gave adequate protection when the patent came before the Courts.

2 RECTOR STREET, NEW YORK

## PROXIMATE ANALYSIS OF NITROCELLULOSE SOLUTIONS AND SOLVENTS<sup>1</sup>

By A. D. CONLEY

Received April 9, 1915

The purpose of this article is to give a few rapid approximate methods which the writer has found useful in the analysis of commercial nitrocellulose solutions. They are intended for use in laboratories where apparatus is limited. No claims for originality are made.

### CONSTITUENTS OF NITROCELLULOSE SOLUTIONS

Although many nitrocellulose solvents are known, comparatively few of them find commercial use. These are mostly the ketones and acetates of the methyl, ethyl, propyl, butyl, and amyl groups. The commercial solvents are mixtures of two or more of the above compounds and include: amyl acetate, ethyl acetate, acetone, wood alcohol, ethyl methyl ketone, and light acetone oil. Amyl acetate, the best all-round solvent, is high in price, and for this and other reasons, non-solvents are nearly always used in connection with the solvents. The most important of these are fusel oil, denatured alcohol, benzol and toluol, and petroleum benzine. Some of these materials will

<sup>1</sup> Abstract of thesis presented at University of Maine in part fulfillment of requirements for degree of Chemical Engineer, June, 1914.

be found in practically every nitrocellulose solution, and in individual cases there may be found some of the amyl acetate substitutes, or some material intended to mask the odor, or perhaps some material designed to raise the flash point. In the pure state all the material may be readily identified by the ordinary methods of organic analysis, but mixtures of impure commercial materials with nitrocellulose offer considerable difficulty if a quantitative analysis is necessary.

### USES OF NITROCELLULOSE SOLUTIONS

**LEATHER FINISHING**—C. A. Higgins<sup>1</sup> and Thomas Callan<sup>2</sup> have discussed the use of nitrocellulose solutions in leather finishing.

**METAL LACQUERS**—For objects where there is little need of handling, or where only a temporary protective coating is desired, nitrocellulose solutions make a very satisfactory lacquer. They are easily and rapidly applied, they may be air-dried, or if speed is desirable, oven-dried at almost any temperature below 100° C. This does away the baking necessary with japans or with some of the synthetic resin lacquers, such as Bakelite. Ordinarily a nitrocellulose film will not stand washing with water to any extent and this renders it unsuitable for many purposes.

Lacquers may be classified: by the surface for which they are designed, as brass or silver lacquers; by the method by which they are applied, as brush, dip, or spray lacquers; or by the surface which they produce, as matt, satin, or glossy lacquers. Brush lacquers are usually the highest in total solids and in resin content. The same lacquer will often serve both as a dip or a spray lacquer. In the latter case the lacquer is blown on the work in the form of a fine spray by the use of a jet of compressed air in a suitable apparatus.

Resin lacquers, sometimes called gum lacquers, are thus defined by Worden.<sup>3</sup> "Resin lacquers are solutions of gum resins or resins in fusel oil and pyroxylin solvents without linseed or other drying oil and hence are not varnishes." Many of the same solvents are found here as in nitrocellulose solutions.

### DETERMINATION OF NITROCELLULOSE

Worden<sup>4</sup> suggests precipitating the nitrocellulose with fusel oil but this method was not found satisfactory as it does not give a complete precipitation.

Benzine, carbon tetrachloride, chloroform, and water all precipitate nitrocellulose. Water and benzine are less satisfactory because they precipitate the resins as well. Chloroform is convenient because it does not precipitate many of the resins and is also easily removed from the precipitated nitrocellulose by drying at moderate heats.

A simple way is to take a 200 cc. stoppered graduated cylinder and place in the neck a small funnel, capable of holding the desired amount. The funnel can be quickly removed when the solution has reached the mark without danger of spilling any of the solution on the sides of the cylinder. The chloroform should be added in small amounts with vigorous shaking. It will be necessary to thin very heavy solutions and to use the diluted solution for this determination. Since the amount of nitrocellulose is usually stated in terms of ounces per gallon, it is convenient to take 10 cc. when

$$\text{ounces per gallon} = \frac{\text{weight precipitate in grams} \times 378.5}{28.35}$$

This amount usually gives a precipitate convenient to handle. When nitrocellulose is dissolved there is an increase in volume, so that the result obtained is the amount actually present in a gallon and not the amount that must be added to a gallon of

<sup>1</sup> "The Uses of Collodion in Leather Finishing," C. A. Higgins, *Leather Trades Year Book*, 1914, pages 148-151; *J. Soc. Chem. Ind.*, **33**, 704.

<sup>2</sup> "The Constitution of Collodion Enamels for Leather," Thomas Callan, *Leather World*, **4**, 523.

<sup>3</sup> "Nitrocellulose Industry," Vol. I, p. 305, D. Van Nostrand Co., 1911.

<sup>4</sup> *Ibid.*, Vol. I, p. 358.