within a few hours after being placed in service the tungsten electrodes would become coated with a very thin film of siliceous scale, which entirely destroyed the sensitivity of the electrode. This circumstance practically eliminates the tungsten electrode or any similar type of electrode from further consideration for use in scale forming products.

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# Chemistry in Incandescent Lamp Manufacture[1]

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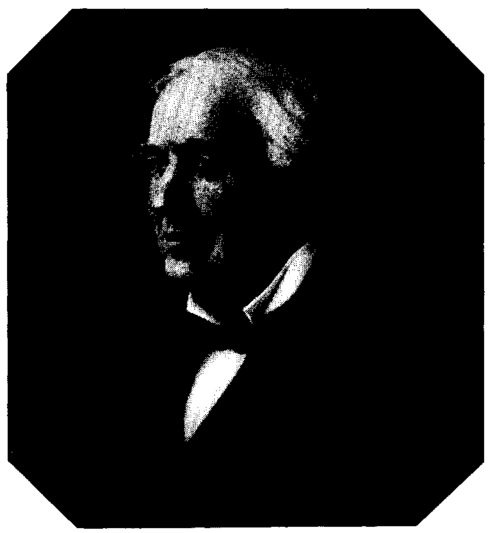
EDITOR'S NOTE—We are pleased to join, through the publication of this special article, in "Lights Golden Jubilee." The exact fiftieth anniversary of Thomas Alva Edison's successful experiment falls on October 21, 1929, and we take this occasion to unite in the world applause to our fellow member.

SINCE this year marks the fiftieth anniversary of the founding of the incandescent lamp industry, it may be interesting to review briefly some of the things which chemistry has done to help bring lamps to their present state of development. Other branches of science have played important parts, but this paper will describe those accomplishments on which chemistry has had a definite bearing.

The lamp manufacturer is faced with the usual problems in connection with the materials of which his product is made. He must deal with tungsten, molybdenum, nickel, copper, brass, and other metals. Glass for lamps is a problem in itself. In addition to the materials which actually appear in lamps, a wide variety of materials is used in processes incidental to lamp making. The manufacture of incandescent lamps is a highly developed art, most of the operation being done with automatic machinery. This necessitates close control of the uniformity of materials, much of which is of a chemical nature.



Thomas Alva Edison, Inventor of the First Practical Incandescent Lamp

### Tungsten

Tungsten (*5*, *6*, *7*) for incandescent lamp filaments is obtained from wolframite ore imported from China in the form of concentrates containing approximately 70 per cent tungstic oxide. A number of methods for extracting the tungstic acid from the ore are in use. One process involves extraction with hot caustic potash solution to form potassium tungstate, which is converted into tungstic acid by treatment with hydrochloric acid. In another method caustic soda is used as the extracting agent, the tungsten being converted successively into sodium tungstate, calcium tungstate, tungstic acid, ammonium paratungstate, and tungstic acid, the last step being accomplished by ignition. Tungstic acid is reduced to metal by heating in hydrogen.

### Gases

A mixture of argon (85 per cent) and nitrogen (15 per cent) is used in most gas-filled lamps. A few types, especially those designed for high-voltage service, are filled with nitrogen only. Argon is not used alone in lamps of general service voltages on account of arcing difficulties. Lamps containing argon may be operated at a higher efficiency than those containing nitrogen, so it is desirable to use the highest possible percentage of it.

Both argon and nitrogen are obtained from the air, the former by distillation of liquid air and the latter by removing oxygen from the air with hydrogen. This is done by the lamp manufacturers at a central point, from which both nitrogen and the argon-nitrogen mixture are shipped in steel cylinders to the lamp factories. Also, the gases are run through a purification process to remove impurities such as hydrogen, oxygen, and carbon dioxide.

Just before the gases are used for filling lamps in the factories, they are further purified. From the cylinders the pressure of the gas is reduced to 60 pounds per square inch and passed successively through a brass tube containing caustic soda or potash, a similar tube of phosphoric anhydride, a heated (550° C.) iron tube filled with copper, and a similar tube of copper oxide also heated to 550° C. The gases are cooled and the pressure reduced to 15 pounds per square inch and then passed through two glass tubes, the first containing caustic soda or potash and the second phosphoric anhydride. After this purification they are piped to the lamp-making machines, where they are still further dried by passing through several tubes of phosphoric anhydride. They are then ready to be used in lamps.

Owing to the extreme importance of having pure gases with which to fill lamps, most of the lamp manufacturers have developed purification systems of their own. One uses silver oxide instead of copper oxide in a system similar to the one described above, another treats the gas with metallic sodium, and still another absorbs oxygen with a solution of hydroquinone. The object in each case, however, is to remove as completely as possible all impurities, but especially hydrogen, oxygen, and water.

### Getters

The word "getter" (*1*, *2*, *3*, *4*, *8*, *9*) has become a rather general term to designate any material which, when placed inside of a lamp, assists in the clean-up of residual gases or reduces the blackening of the bulb. Getter in one form or another is used in practically all incandescent lamps. The

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high temperatures of lamp filament operation and small amounts of materials which affect lamp performance have made getter a fascinating problem for chemists and physicists.

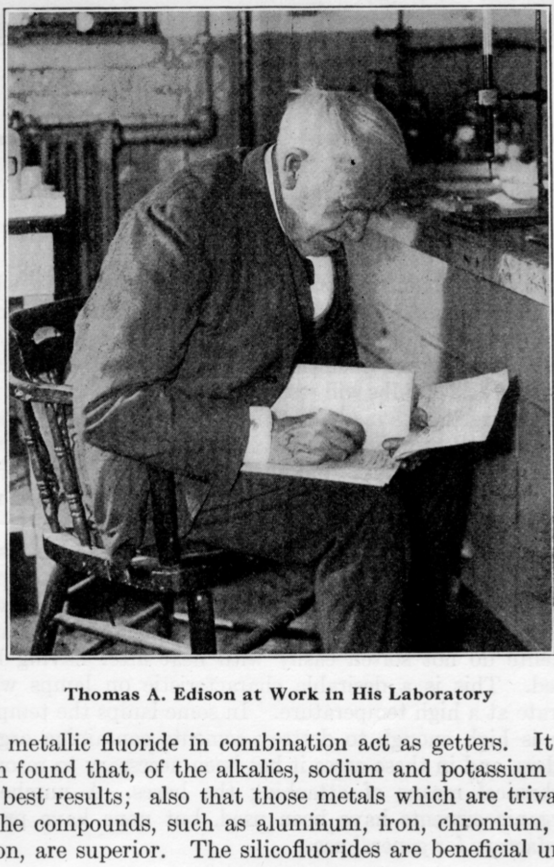
In gas-filled lamps the inert gas used in filling is considered a getter, and no other getters are strictly essential to the manufacture of good-quality lamps. However, it is general practice to use a small amount of red phosphorus in the smaller sizes of large lamps. Some manufacturers have found it desirable to add traces of organic material to the getter gas.

Edison's Original Menlo Park Chemical Laboratory as Restored at Dearborn, Mich., by Henry Ford

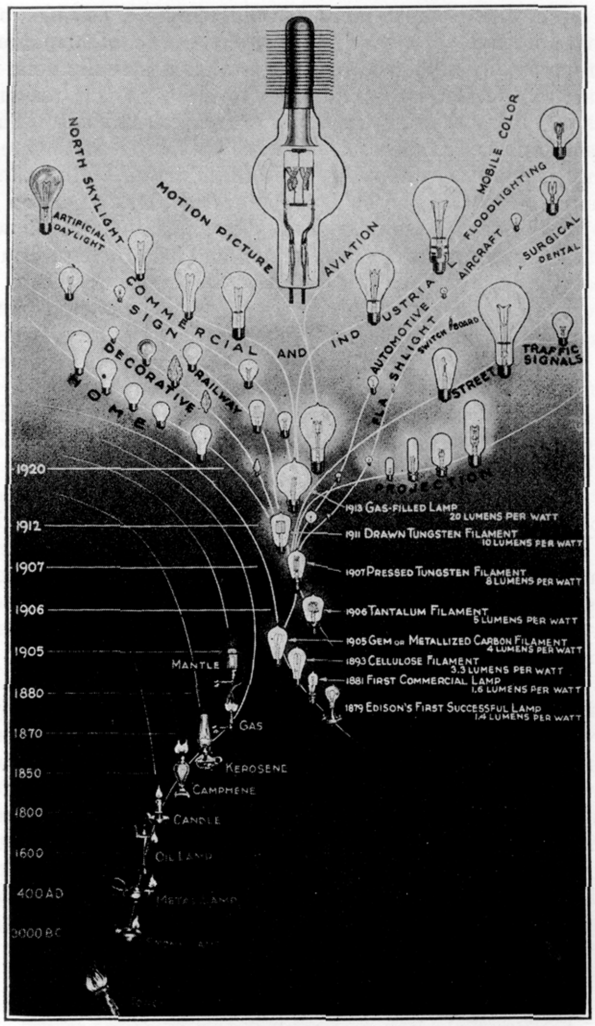
Water vapor in a lamp seriously impairs its quality and every effort is made to keep it out, driers being used at the exhaust machines and in the getter gas lines. Water vapor greatly accelerates the rate at which tungsten from the filament is deposited on the bulb. The action appears to be of a cyclic nature—the water reacting with the hot filament to form a compound, which volatilizes and is deposited on the bulb where the action is reversed, again forming water which returns to attack the filament leaving the metallic tungsten on the bulb. Apparently carbon breaks up this cycle without impairing the lamp if present in the proper amount. Phosphorus also assists in the removal of water vapor.

In vacuum-type lamps the getter problem is somewhat more complex owing to the high degree of vacuum required for successful operation. Getters for these lamps consist of two essential parts, red phosphorus and a second substance which is usually a fluoride. These materials are finely ground by ball-milling in a solution of nitrocellulose in amyl acetate. The nitrocellulose acts as a binder to hold the getters on the filaments during the manufacturing process. Getters are applied by dipping or spraying the filaments in amounts varying with the type of lamp. An ordinary 25-watt lamp contains approximately 0.5 mg. of getter, of which 15 per cent is phosphorus. In a good lamp of the vacuum type the pressure of the residual gases in the finished product is somewhat less than one micron. To obtain this degree of vacuum with exhaust pumps would require a considerable length of time. However, pressure of the order of 20 microns can be readily obtained, and this is what is actually done in practice. Getter is depended on to clean up this residual gas and bring the pressure down to the desired point. This is accomplished in a few seconds when the lamp is first lighted. The clean-up of residual gases is the function of the phosphorus. Most of the evidence indicates that the action is physical rather than chemical. Experiments have shown that the gases are driven to the bulb and held there so firmly that they are not released at the operating temperature of the lamp. There probably is some reaction between phosphorus and oxygen to form phosphoric anhydride, which in turn reacts with water, but this action of phosphorus appears to be of minor importance. Although phosphorus is universally used, other materials which sublime readily, such as arsenic, sulfur, and iodine, have a getter action similar to that of phosphorus.

It has been found that, in order to make lamps that do not blacken excessively during life, another getter material is necessary. This material is vaporized from the filament and settles on the bulb, where its action apparently takes place, materially reducing the blacking caused by deposited tungsten. A number of theories have been advanced, some physical and some chemical, but the exact mechanism of the action has not been definitely established. A wide variety of materials which will retard bulb blackening have been found. In practically all vacuum lamps, however, fluorine compounds are used. In Europe calcium fluoride is used extensively, while in America double fluorides such as cryolite have been found more desirable. A compound analogous to cryolite with iron substituted for aluminum finds extensive use in the larger sizes of vacuum lamps. The field of double fluorides has been quite thoroughly investigated. Most of these compounds having an alkali and metallic fluoride in combination act as getters. It has been found that, of the alkalies, sodium and potassium give the best results; also that those metals which are trivalent in the compounds, such as aluminum, iron, chromium, and boron, are superior. The silicofluorides are beneficial under



Thomas A. Edison at Work in His Laboratory




**A Pictorial History of Artificial Light**

some conditions if present in small quantities, probably owing to the affinity of silicon tetrafluoride, which is one of the decomposition products, for water.

## Basing Cements

In basing cement it is necessary to have a material which will not deteriorate readily under the influence of atmospheric conditions or heat. It must not be too rigid or have a materially different coefficient of expansion than that of glass or cracked bulbs will result.

Most large lamps made in America are based with cements consisting of combinations of Bakelite, shellac, and rosin cut in alcohol as binders and marble flour as the filler. Present manufacturing practice requires that bases be filled with cement somewhat in advance of their application to the lamps. Bakelite is a particularly useful constituent, since cements containing it do not harden readily at room temperatures, with the result that filled bases may be held several days without becoming unfit for use. Cements containing Bakelite do not soften easily with heat after having been baked. This is a desirable characteristic on lamps which operate at a high temperature. In some lamps the temperature is high enough to destroy cements containing organic binders, and in these cases it has been necessary to resort to mechanical means of attaching the bases. A number of inorganic cements have been used, but none have proved satisfactory for general use.

In basing cements containing Bakelite, malachite green is used extensively as a rough temperature indicator. Ammonia volatilized from the Bakelite and the heat of basing combine to destroy the green color at approximately the minimum time and temperature essential to good basing.

For miniature lamps, where a white cement is desirable, cements containing white shellac, dammar gum, and rosin or combinations of these and a marble flour filler are commonly used.

## Inside Frosting

Most of the lamps now used for general lighting purposes are frosted on the inside in order to produce diffused light (*11*). Although outside frosting was used for many years, inside frosting of bulbs was, until recently, impractical owing to the extreme fragility imparted to the bulbs by the frosting process. In 1924 a method was discovered whereby the strength of inside-frosted bulbs could be restored by a subsequent treatment, which consists essentially of treating the bulbs with a diluted frosting solution.

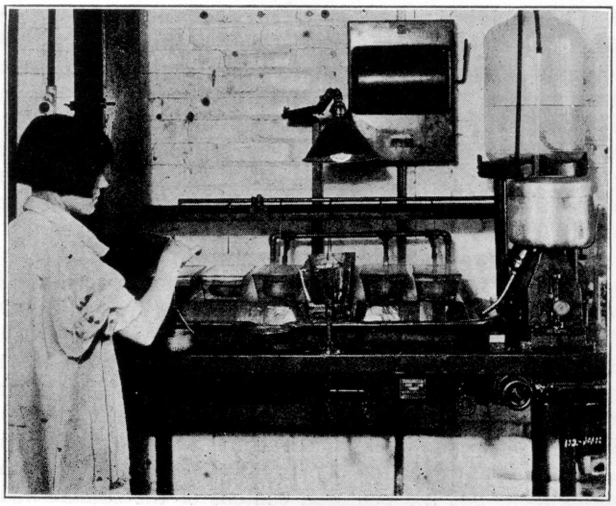
Lamp bulbs are inside-frosted with a solution containing ammonium bifluoride 42 per cent, dextrin 7 per cent, barium sulfate 20 per cent, sodium bisulfate 3.5 per cent, hydrofluoric acid 27.5 per cent, and water. The acidity should fall between 18 and 25 per cent hydrofluoric acid.


**Coating Filaments with "Getter"**

## Lamp Coatings

One of the problems of the lamp chemist has been to develop suitable colored coatings. On large lamps, where considerable heat is developed during operation, organic resins have not been practical owing to charring and to the fact that they lose their adhesive properties under the continued heat treatment. In addition, they do not stand weather conditions well.

Coatings containing sodium silicate as a binder have been used extensively for lamps and have been fairly satisfactory. These coatings are made by ball-milling a mixture of sodium silicate with kaolin and a colored pigment. Other



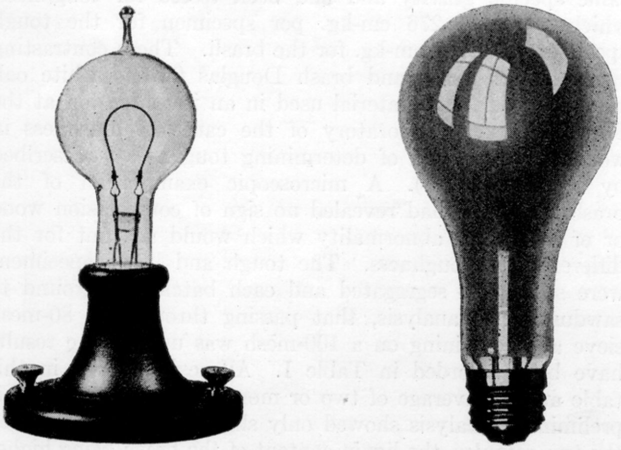
Immersing Coiled Tungsten Filament in Nitric Acid in Modern Lamp Factory

materials, such as the oxides of zinc, tin, and titanium, are sometimes added to give certain characteristics. Kaolin appears to be an essential part of the mixtures, since coatings containing it seldom crack or chip off. The coatings are invariably applied to lamps by spraying, since the mixtures are not adapted to dipping.

Sodium silicate coatings deteriorate rapidly on standing, because of the action of water and carbon dioxide in the air. This difficulty is overcome by dipping the lamps after spraying and baking in a fixing solution. A number of materials are satisfactory for this purpose, but usually a concentrated solution of ammonium chloride or aluminum sulfate is used. The solutions are used hot in order to accelerate the reaction. If the solutions are acidified, the fixing can be carried out still more rapidly.

Recently the American manufacturers have started the manufacture of lamps with coating sprayed on the inside of the bulb (*10*). The composition of the coating is similar to that used for outside coatings, although less sodium silicate is required. Fixing of the inside coating is unnecessary because the bulbs are made into lamps shortly after spraying.

Miniature lamps, such as those used for Christmas tree lighting, are sprayed with mixtures containing shellac as the binding agent. In addition to shellac, these coatings contain kaolin and a colored pigment.



**The Miracles of 1879 and 1929**

At the left the first practical incandescent lamp as invented by Thomas A. Edison. In the main it consisted of a glass chamber exhausted of its air and containing a carbon filament made from bristol board paper. Platinum lead-in wires carried the current to the carbon burner. Note the crude wood stand equipped with metal binding posts.

At the right the new 100-watt inside-frosted Mazda lamp, which is a result of constant improvements and contributions of chemical science, gives seventy-six times as much light for the same money as its illustrious ancestors of fifty years ago.

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# Chemical Composition of Wood in Relation to Physical Characteristics[1]

## A Preliminary Study


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The chemical nature of wood has been the subject of much research and samples of different species have been analyzed for cellulose, lignin, pentosans, extractives, and various other constituents. The results of such analyses have formed a basis for comparing the different woods—as, for example, hardwoods with softwoods, or woods of high extractive content with those of low. Ritter and Fleck (*6*), advancing a step further, have analyzed for comparison the distinctive portions of certain trees—namely, the heartwood and the sapwood, the springwood and the summerwood. Little research has been carried out, however, to determine whether wood samples from the same tree, but having distinct differences in physical properties, are different in chemical composition. The only work of this kind is that of Johnson and Hovey (*3*), who have reported on the chemical composition of a disk cut from a balsam fir and containing (*a*) slow-growth annual rings, (*b*) rapid-growth annual rings, and (*c*) "Rotholz" ("compression" wood). Their analytical data showed that the "compression" wood portion was much lower in cellulose and higher in lignin than the other portions of the disk.

The object of this work is to show whether there are other cases in which differences in physical characteristics are reflected in chemical constitution, and in this way bring

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