

# UNITED STATES PATENT OFFICE.

LUDWIG GRABAU, OF HANOVER, GERMANY.

## PROCESS OF OBTAINING METALLIC SODIUM.

SPECIFICATION forming part of Letters Patent No. 464,097, dated December 1, 1891.

Application filed October 1, 1890. Serial No. 366,753. (No specimens.) Patented in Germany September 20, 1890, No. 56,230; in France September 20, 1890, No. 208,354; in Belgium September 20, 1890, No. 92,055; in England October 9, 1890, No. 16,060, and in Austria-Hungary December 12, 1890, No. 42,222 and No. 67,608.

*To all whom it may concern:*

Be it known that I, LUDWIG GRABAU, a subject of the Emperor of Germany, residing at Hanover, Germany, have invented certain  
5 new and useful Improvements in the Process of Obtaining Metallic Sodium, (for which I have obtained Letters Patent in the following countries, to wit: in Germany, September 20, 1890, No. 56,230; in Austria-Hungary, Decem-  
10 ber 12, 1890, No. 42,222 and No. 67,608; in Great Britain, October 9, 1890, No. 16,060; in France, September 20, 1890, No. 208,354, and in Belgium, September 20, 1890, No. 92,055;) and I do hereby declare the following to be a full,  
15 clear, and exact description of the invention, such as will enable others skilled in the art to make and use the same.

This invention relates to the production of sodium from its chloride by electrolysis; and  
20 it consists in a novel method or process of obtaining this metal, as will now be fully described.

The methods heretofore proposed or followed for obtaining metallic sodium from mol-  
25 ten sodium chloride have invariably proven unsuccessful in a commercial sense, owing to the high melting-point of the sodium chloride, which has to be heated to a bright-red heat before it can be melted. At this temperature  
30 the metallic sodium in its nascent state combines at once with the excess of its chloride and forms the subchloride, ( $\text{Na}_2\text{Cl}$ ), discovered by Rose and Bunsen, which combines with and is distributed in the molten chloride, and  
35 is in part oxidized on the surface thereof and in part reconverted into chloride by chlorine liberated at the positive pole, so that but little or no metal is actually separated. This oxidation of the subchloride may take place on the  
40 surface of the molten chloride by contact with atmospheric moisture, a hydroxide resulting from the reaction according to the following equation:  $\text{Na}_2\text{Cl} + \text{H}_2\text{O} = \text{NaHO} + \text{NaClH}$ .

From experience I have become satisfied  
45 that sodium could be obtained on a commercial scale, or, at least, to a largely-increased extent, if it were possible to electrolytically treat the fused or molten chloride at a temperature so low as to avoid the formation of  
50 the subchloride referred to and its partial oxidation and reversion into chloride. Af-

ter long experiments I have discovered that the melting-point of the sodium chloride can be materially reduced by combining there-  
55 with a chloride of the metals of the alkalies—such as potassium chloride, for instance—and a chloride or chlorides of the metals of the alkaline earths, thus forming a trisalt combination, the melting-point of which is consid-  
60 erably lower than that of the sodium chloride itself. Such a trisalt combination will melt when heated to a dull-red heat, and at this temperature no subchlorides are formed.

While but a very small quantity of sodium is obtained electrolytically from the pure  
65 fused or molten chloride by the known processes or methods, owing to the subchloride formation above referred to, I have by practical operations obtained ninety-five per cent.  
70 of the theoretical quantity of the metal obtainable according to the law of Faraday, provided the temperature of the molten chloride is sufficiently low to prevent the formation of a subchloride, and this I attain by  
75 the use of the trisalt combination above referred to. I have obtained the best results when the chlorides of the metals of the alkalies—as, for instance, potassium and sodium  
80 chloride—are employed in the proportion of molecule for molecule and the chloride or chlorides of the metals of the alkaline earths in the proportion of one molecule of the latter to three molecules of the chlorides of the  
85 metals of the alkalies. Such a trisalt combination will melt at a much lower temperature than that required to melt sodium chloride alone.

Among the chlorides of the metals of the alkaline earths I prefer to use strontium chlo-  
90 ride, because its melting-point is lower than that of barium chloride, while it is difficult to free the calcium chloride from the water held thereby. On the other hand, I have found that when a chloride of the metals of  
95 the alkalies other than potassium chloride is employed in the trisalt combination the melting-point thereof is higher than when potassium chloride is used and the percentage of sodium obtained is correspondingly re-  
100 duced, practical experiments having shown a reduction of sodium from ninety-five per cent. when potassium chloride is present to fifty



per cent. and below when another chloride of the metals of the alkalies is substituted therefor. Notwithstanding the comparatively small proportion of sodium chloride present in the trisalt combination, the sodium obtained is nearly pure, without a trace of a metal of the alkaline earths and containing but three per centum (3%) of potassium, which in the most important uses of the sodium will not prove detrimental, and even this small percentage of potassium can be eliminated by an oxidizing fusion—that is to say, if it is desired to eliminate the potassium from the sodium the oxidizing fusion of said sodium may be effected by simply melting the sodium while covered with petroleum and by injecting air into the molten metal. In this manner the potassium will be oxidized first. A very small proportion of the sodium will, however, also be oxidized, so that a loss of this small proportion of sodium will be unavoidable. In the more important uses of the sodium it is in fact not necessary that the potassium should be removed; but if this is desired or necessary the above-described method by oxidizing fusion may be resorted to.

It will be readily comprehended by those conversant with this branch of the arts that the composition of the molten bath may be readily maintained by the addition of sodium chloride in proportion to the sodium separated and by the addition of such small quantities of potassium chloride as will replace the very small percentage of potassium separated with the sodium, thus rendering it possible to produce sodium on a commercial scale. On the other hand, great technical difficulties are overcome by the reduc-

tion of the melting-point of the sodium chloride, in that the durability of the melting-pots and of the carbon electrodes is greatly enhanced, while the process is essentially practical and simple and readily carried out.

Having thus described my invention, what I claim as new therein, and desire to secure by Letters Patent, is—

1. The process of obtaining sodium from its chloride, which consists in forming a trisalt by combining sodium chloride with another chloride of the metals of the alkalies and a chloride or chlorides of the metals of the alkaline earths, melting the trisalt, and separating the sodium by electrolysis.

2. The process of obtaining sodium from its chloride, which consists in combining sodium chloride with another chloride of the metals of the alkalies and with a chloride or chlorides of the metals of the alkaline earths in the proportion of one molecule of the latter to three molecules of the chlorides of the metals of the alkalies, melting the trisalt, and separating the sodium by electrolysis.

3. The process of obtaining pure sodium from its chloride, which consists in forming a trisalt by combining sodium chloride with potassium chloride and a chloride or chlorides of the metals of the alkaline earths, melting the trisalt so obtained, separating the sodium by electrolysis, and eliminating the potassium by oxidizing fusion, as set forth.

In testimony whereof I affix my signature in presence of two attesting witnesses.

LUDWIG GRABAU.

Witnesses:

AL C. PEPPERMÜLLER,  
W. KETTLER.