

# UNITED STATES PATENT OFFICE.

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PROCESS OF MAKING OXALATES FROM FORMATES.

973,832.

Specification of Letters Patent.

Patented Oct. 25, 1910.

No Drawing.

Application filed September 13, 1907. Serial No. 392,781.

To all whom it may concern:

Be it known that I, ARNOLD WIENS, a subject of the German Emperor, residing at Bitterfeld, in the Empire of Germany, whose  
5 post-office address is Bitterfeld, Germany, have invented a certain new and useful process for manufacturing oxalates from the corresponding formates by heating in a space from which the air has been partially ex-  
10 hausted, of which the following is a specification.

It has not heretofore been considered practicable to convert in a technically satisfactory manner formates into oxalates by  
15 heating them without any addition whatever.

In a process first mentioned by Merz and Weith wherein there is used a chamber the air of which is partially exhausted, as it  
20 was thought that air exerted a prejudicial influence on the formation of oxalate, a high temperature, from 360° upward, was employed and this was considered essential (see *Reports of the German Chem. Soc.*, Vol.  
25 15 (1882) page 1508 and following). When the temperature is raised to the extent considered essential by Merz and Weith in a vacuum as perfect as can be obtained the reaction becomes so violent as to render the  
30 process impracticable. As, however, it seems to have been their intention to exclude air only to some extent it may be assumed that Merz and Weith operated only in a partially exhausted space. The defect above  
35 referred to is obviated when the conversion of formate is caused to take place in a space from which the air has been partially or completely exhausted at a temperature below 360°. The process of reaction will pro-  
40 ceed quite smoothly and without the occurrence of explosions at a temperature of 280°; it is possible even to interrupt the process, as the development of hydrogen occurring during the process at the low tem-  
45 perature will at once cease as soon as the vacuum is suspended, or become so slight as not to give rise to interruptions. Admixture and the loss inevitably attendant thereon can be entirely dispensed with. In the

process of producing formate wherein solid 50 caustic soda is employed, which, as is well known, directly yields technically pure formate, it is even possible to proceed with the conversion of formate into oxalate in the same apparatus wherein the formate had 55 been obtained. By this means the necessity for transporting the substance is dispensed with, no losses can occur, and the heat contained in the finished formate in the auto-  
60 claves is not lost. The temperature of the reaction is lower than in any of the methods heretofore employed, with the result that there can be effected a considerable saving of fuel. The hydrogen removed by suction may be employed as fuel or as it is pure it 65 may be employed for other purposes. Even if other substances are added to the formate, in accordance with the processes described in the German Patents Nos. 111078 and 161512 the process to which this invention 70 relates offers advantages. The process, is, however, most advantageous when no such additions are made.

What I claim as my invention and desire to secure by Letters Patent is:— 75

1. The process for manufacturing oxalates, which consists in bringing formates into a vessel and heating the formates therein *in vacuo* at a temperature substantially below 360° C.; substantially as described. 80

2. The process for manufacturing oxalates, which consists in bringing formates into a vessel and heating the formates therein *in vacuo* at a temperature in the neighborhood of 280° C.; substantially as de- 85 scribed.

3. The process for manufacturing oxalates which consists in bringing formates into a vessel, exhausting the air as completely as possible therefrom and heating 90 the formates to between 280° C. and 360° C.

In testimony whereof I have hereunto signed my name to this specification in the presence of two subscribing witnesses.

ARNOLD WIENS.

Witnesses:

JULIUS RÜNDLAND,  
KARL KREKEBEN.