

UNITED STATES PATENT OFFICE.

MARTIN FREUND, DECEASED, LATE OF FRANKFORT-ON-THE-MAIN, GERMANY; BY
WALTER FREUND, ADMINISTRATOR, OF FRANKFORT-ON-THE-MAIN, AND EDMUND
SPEYER, OF FRANKFORT-ON-THE-MAIN, GERMANY.

PRODUCT OF REDUCTION OF OXYCODEINON AND PROCESS OF PREPARING THE SAME.

No Drawing. Original application filed July 15, 1921, Serial No. 485,130. Divided and this application
filed March 15, 1922. Serial No. 544,010.

(GRANTED UNDER THE PROVISIONS OF THE ACT OF MARCH 3, 1921, 41 STAT. L., 1313.)

To all whom it may concern:

Be it known that MARTIN FREUND, deceased, formerly a resident of Frankfort-on-the-Main, for whom the undersigned
5 WALTER FREUND, residing at Frankfort-on-the-Main, Germany, citizen of the German Republic, is acting administrator, and I,
EDMUND SPEYER, a resident of Frankfort-on-the-Main, Germany, a citizen of the German Republic, have invented certain new
10 and useful Improvements in a Product of Reduction of Oxycodoinon and Processes of Preparing the Same (for which we have made
application in Germany April 14, 1916, Patent No. 296,916; in Austria Dec. 9, 1916, Patent No. 75,740; in Hungary Dec. 9, 1916,
15 Patent No. 71,587; in Switzerland Dec. 7, 1916, Patent No. 75,110; in Norway Dec. 30, 1919, Patent No. 31,350; in Czechoslovakia
Feb. 10, 1920, Patent No. 2228; in Denmark Dec. 29, 1919; in Sweden Jan. 20, 1920; in Italy Feb. 23, 1920, and Netherlands Dec.
20 31, 1919), of which the following is a clear, full, and exact description.

25 The subject of this invention is dihydrooxycodoinon, an unknown derivative of oxycodoinon—a product described in the German Letters Patent No. 286431—and the process of preparing the same.

30 Oxycodoinon, having the formula $C_{18}H_{19}NO_4$ and a melting point of $275^{\circ}C.$, by boiling with a solution of sodium hydrosulphite, is reduced to dihydrooxycodoinon of the formula $C_{18}H_{21}NO_4$. Dihydrooxycodoinon prepared in such manner is actually identical with dihydrooxycodoinon as prepared in accordance with the specification of the patent application Serial Number 485130, filed July 15, 1921, from which this
40 application is a divisional application.

The identity is shown by the following facts. Dihydrooxycodoinon prepared in accordance with this application crystallizes from alcohol in long jagged columns melting at $222^{\circ}C.$ and yields well crystallized
45 salts. Its hydrochloride $C_{18}H_{20}NO_4.HCl$ crystallizes from water or diluted alcohol in

columns melting at 268 to $270^{\circ}C.$ Its free base is precipitated from solutions of the salts by ammonia, soda or alkalies; it is insoluble in excess of alkalies. It combines with methyl iodide to form $C_{18}H_{21}NO_4.CH_3I$ a compound representing prisms which decompose at $251^{\circ}C.$ Its ketone character is shown by the formation of an oxime, the hydrochloride of which: $C_{18}H_{22}N_2O_4.HCl$ is isolated in small sticks decomposing at 275 to $278^{\circ}C.$

The new dihydrooxycodoinon is to be used in medicine and pharmacy.

Example: 10 grms. oxycodoinon are heated with a solution of 20 grms. sodium hydrosulphite $Na_2S_2O_4$ in 60 cc. water. After a short time complete solution takes place. Upon supersaturating with soda or ammonia 6 grams dihydrooxycodoinon are separated in crystalline form.

Having thus described this invention, we claim as new and desire to secure by Letters Patent:

1. The process of preparing dihydrooxycodoinon, melting at $222^{\circ}C.$, which consists in boiling oxycodoinon together with a solution of sodium hydrosulphite.

2. A new product of reduction, obtained by heating oxycodoinon together with a solution of sodium hydrosulphite, dihydrooxycodoinon having the formula $C_{18}H_{21}NO_4$, which crystallizes from alcohol in long jagged columns, melting at $222^{\circ}C.$, and yields well crystallized salts and forms an oxime, the hydrochloride of which is isolated in small sticks decomposing at 275 to $278^{\circ}C.$

In testimony whereof we have hereunto signed our names in the presence of the two subscribing witnesses.

DR. WALTER FREUND,
Acting administrator upon the estate of the deceased inventor, Martin Freund.

PRIVATE AGENT DR. EDMUND SPEYER.

Witnesses:

FRITZ MAYER,
FRIEDRICH L. HAHN.