

UNITED STATES PATENT OFFICE.

JOSEF ZIEGLER, OF HAMBURG-UHLENHORST, GERMANY, ASSIGNOR TO
FRANZ FRITZSCHE & CO., OF SAME PLACE.

ESSENCE OF VIOLETS.

SPECIFICATION forming part of Letters Patent No. 601,193, dated March 22, 1898.

Application filed December 18, 1896. Serial No. 616,213. (Specimens.)

To all whom it may concern:

Be it known that I, JOSEF ZIEGLER, chemist, of Hamburg-Uhlenhorst, Germany, have invented new and useful Improvements in Manufacturing Artificial Essence or Essential Oil of Violets, of which the following is a specification.

The present invention relates to a process for the manufacture of artificial essence or essential oil of violets and is chiefly remarkable for its great simplicity and cheapness of production as well as for the cheapness of the raw materials used. Hitherto artificial violet scent has been produced in the well-known manner of condensing citral with acetone, whereupon aldehydes and ketones form under the influence of alkalies or alkaline earths new substances with formation of water. The product thus obtained (pseudojonon) is converted by diluted mineral acid into jonon. This process is costly and the amount yielded is very small. The inventors came to the conclusion that the plants must use, when producing this scent, materials other than alkalies or alkaline earths and that perhaps combinations of iron played a part in it under the influence of light and oxygen. Experiments have revealed the surprising fact that in the presence of substances which with water yield oxygen by cold or by hot process a product of condensation is obtained from citral or from oils containing citral and from acetone, which product when boiled with weak oxidizing substances produces a volatile oil having the fragrance of flowers.

The new and essential feature of the present invention consists in citral, or oils containing citral, and acetone being treated in diluted solution of alcohol with such substances as usually have an oxidizing effect, but which in the present case, probably owing to the temporary arrangement of atoms, protect or conduct the reaction in the right direction, such substances as hypochlorites of alkalies or alkaline earths as well as barium peroxid can be used with advantage.

The invention further consists in the product so obtained being simultaneously converted by means of weak oxidizing substances into an oil having the fragrance of violets

and freed from impurities. For this purpose chlorid of iron or arsenic-acid may be used with advantage. The result attained is the more surprising and remarkable since chlorid of lime, for instance, with only acetone or alcohol, yields chloroform.

A practical application of the new process is as follows: One kilogram of acetone and 0.5 kilogram of citral are dissolved in 1.5 kilograms of alcohol; and 1 liter of freshly filtered clear concentrated solution of chlorid of lime is added, the mixture being continually stirred. The mass becomes heated and is left to stand for several days, or the heating may be increased by boiling it in a reflux cooling apparatus and so maintained for about six hours. When the liquid has cooled, it is diluted with about ten liters of water, the oil that collects on the surface is skimmed off, and small quantities of superfluous acetone and citral which did not take part in the reaction are removed from this oil by a weak jet of steam. The oil is then boiled with five liters of diluted solution of chlorid of iron of about 1.025 specific gravity for at least one day. By the action of the solution of chlorid of iron not only is the substance altered into essence of violets, but also simultaneously with this undesirable by-smells are removed. The oil obtained in such manner and separated from the solution of chlorid of iron is at first freed by a strong jet of steam from the small admixture of other oils and then distilled again with steam, and this fractional distillation repeated, if necessary. The volatile oil or essence obtained in such manner does not easily evaporate, is of yellowish color, and has a smell resembling that of sandal-wood or fine cedar-wood; and if properly diluted it has a marked smell of freshly-gathered violets.

Instead of citral a double quantity of Indian verbena oil (lemon-grass) may be used—in the present example, therefore, 1 kilogram. The process remains the same. Instead of a saturated solution of chlorid of lime 0.25 kilogram of barium peroxid may be used, the other proportions remaining as described, said barium peroxid being suspended in 0.5 kilogram of water and added to the solution of cit-

ral and of acetone in alcohol. Then the solution is heated and the process conducted as described.

Having now particularly described and ascertained the nature of the said invention and in what manner the same is to be performed, I declare that what I claim is—

1. The process of manufacturing artificial oil of violets, which consists in treating citral or oils containing citral in a mixture of acetone and diluted alcohol, with an active oxidizing agent such as barium peroxid, and then further oxidizing the product by boiling it with ferric chlorid, substantially as described.

2. The process of manufacturing artificial oil or essence of violets, consisting in dissolving one kilogram of acetone and one-half kilo-

gram of citral in one and one-half kilograms of alcohol and adding one liter of freshly-prepared clear saturated solution of chlorid of lime, the mixture being constantly stirred and then heated for a few hours in a reflux cooling apparatus, the oil so obtained being then boiled for a considerable time in five liters of a diluted solution of ferric chlorid and finally distilled, substantially as described.

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

JOSEF ZIEGLER.

Witnesses:

JOSEF SUHR,

E. H. L. MUMMENHOFF.