United States Patent Office.

HENRY CARL FEHRLIN, OF ST. LOUIS, MISSOURI, ASSIGNOR TO THE FINK & FEHRLIN CHEMICAL COMPANY, OF MILWAUKEE, WISCONSIN.

PROCESS OF MANUFACTURING SALOL.

SPECIFICATION forming part of Letters Patent No. 622,456, dated April 4, 1899.

Application filed February 25, 1898. Serial No. 671,691. (No specimens.)

To all whom it may concern:

Be it known that I, HENRY CARL FEHRLIN, doctor of philosophy, a citizen of the Republic of Switzerland, residing at St. Louis, State 5 of Missouri, have invented certain new and useful Improvements in the Manufacture of Salicylate of Phenyl, of which the following is a full, clear, and exact description, which will enable others skilled in the art to which 10 it appertains to use the same.

In the various processes for the manufacture of phenyl-salicylic-acid ester (or the socalled "salol") the same is formed as described in the patents now to be referred to and in-15 dicated by the following equations:

In Letters Patent No. 350,012, dated the 28th day of September, 1886, to Marcel von Nencki:

$${}^{20} 2C_{6}H_{4}-COOH+2C_{6}H_{5}OH+POCl_{3}= \\ -OH \\ 2C_{6}H_{4}-COOC_{6}H_{5}+HPO_{3}+3HCl$$

or; -OH $2C_6H_4 - COONa + 2C_6H_5OH + POCl_3 =$ 2C₆H₄-COOC₆H₅+NaPO₉+NaCl+2HCl

30 or
$$-OH \\ 2C_{6}H_{4}-COONa+2C_{6}H_{5}ONa+POCl_{3}=\\ -OH \\ 2C_{6}H_{4}-COOC_{6}H_{5}NaPO_{3}+3NaCl$$

In Letters Patent No. 377,311, dated the 31st day of January, 1888, to Carl Kolbe:

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$$-OH_{40} C_{6}H_{4}-COOH+C_{6}H_{4}OH+COCl_{2}=\\ -OH_{C_{6}H_{5}}-COOC_{6}H_{5}+CO_{2}+2HCl$$

In Letters Patent No. 383,306, dated the 22d day of May, 1888, also to Carl Kolbe:

$$-OH 3C_{6}H_{4}-COOH+3C_{6}H_{5}OH+PCl_{3}= -OH 3C_{6}H_{4}-COOC_{6}H_{5}+3HCl+P(OH)_{3}$$

In Letters Patent No. 291,248, dated the 50 16th day of October, 1888, to Panajota W. Hofman:

$$C_{6}H_{5}ONa - OH$$
 $C_{6}H_{5}ONa + COCl_{2} = 2NaCl + C_{6}H_{4} - COOC_{6}H_{5}$ 55
or;
 $-OH$
 $C_{6}H_{4} - COONa + C_{6}H_{5}ONa + COCl_{2} =$
 $-OH$
 $2NaCl + CO_{2} + C_{6}H_{4} - COOC_{6}H_{5}$

According to the above patents salol could only heretofore be produced if salicylic acid or the neutral salts of same, such as 65 C₆H₄(OH)COONa or C₆H₄(OH)COOK, and phenol or phenolate of soda are used as raw materials, and, as stated in the Patents Nos. 377,311, and 383,306, the result obtained is a very impure product if in place of phenol 7° salts of phenol are used. I have now discovered that salol or salicylate of phenyl can also be produced by employing a mixture of phenol and crude basic salicylate of soda instead of salicylic acid or the neutral salts thereof. 75 The manufacture of salol is thus very materially facilitated, for this process dispenses with the preliminary production of salicylic acid or its neutral salts, which was heretofore necessary, and can easily be combined with 80 the manufacture of basic salicylate of soda, such as carried out in Kolbe's process for the manufacture of salicylic acid.

The mode of carrying out the process of my invention is to make a mixture of one equiva- 85 lent of basic salicylate of soda and one molecule of phenol and to subject the same to the action of phosphorus oxychlorid at a temperature of 120° to 140° Celsius, or in case it is convenient to combine this process with that of 90 making basic salicylate of soda to allow the temperature which was necessary to form the latter to cool down to about 100° Celsius, subsequently adding so much phenol that there is exactly one equivalent of the same to one 95 equivalent of the basic salicylate of soda, and then treating the mixture with POCl₃ or the other reagents at a temperature of 120° to 140°

Celsius, as indicated, for instance, by the following equation:

 $5 \begin{array}{c} -\text{ONa} \\ 2\text{CH}_{4} - \text{COONa} + 2\text{CH}_{5}\text{OH} + \text{POCl}_{3} = \\ \text{OH} \\ \text{NaPO}_{3} + 2\text{C}_{6}\text{H}_{4} - \text{COOC}_{6}\text{H}_{5} + 3\text{NaCl.} \end{array}$

The product thus obtained will then give a pure colorless salol if it is treated with a solution of carbonate of soda and the salol distilled off in a current of steam.

I wish it distinctly understood that I do not limit myself to the employment of phosphorous oxychlorid in carrying out my process, as any other ingredient reacting similarly upon such mixtures may be used.

I do not claim anything disclosed in the patents referred to; but

What I claim as new, and desire to protect 20 by Letters Patent, is—

1. The within-described process of producing salol from basic salicylate of soda and phenol, which consists, in treating one mole-

cule of basic salicylate of soda and one molecule of phenol with phosphorous oxychlorid 2 at a suitable temperature.

2. The within-described process of producing salol from basic salicylate of soda and phenol, which consists, in treating one molecule of basic salicylate of soda and one molecule of phenol with phosphorous oxychlorid at a temperature of from 120° to 140° Celsius.

3. The within-described process of producing salol which consists in treating basic salicylate of soda with the necessary quantity 3 of phenol, and phosphorous oxychlorid at a temperature of from 120° to 140° Celsius, subsequently adding a solution of carbonate of soda, and finally distilling off the salol in a current of steam.

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

HENRY CARL FEHRLIN.

Witnesses:

ALFRED A. MATHEY, C. F. KELLER.