concentration of 25 % by weight of leucoindigo, approx. 44 % by weight water may be distilled off in order to concentrate the solution up to 45 % by weight (Example 4).

[0015] Example 5 of WO 00/04100 discloses that from an aqueous solution containing 20 % by weight leucoindigo about 34 % water are distilled off in order to concentrate the solution to 45 % by weight.

[0016] WO 00/04100 is silent regarding aniline and N-methylaniline concentrations.

OBJECTS OF THE INVENTION

[0017] There is an ongoing need in the industry for providing concentrated leucoindigo salt solutions in which the content of said aromatic amine is as low as possible. Thus, the problem to be solved by the present the invention was to provide purified concentrated leucoindigo salt solutions and indigo made therefrom having an aniline content of far below 200 ppm, i.e. at least less than 100 ppm and in particular at least less than 40 ppm or 30 ppm, preferably less than 20 or 10 ppm or less than 5 ppm or even 0 ppm.

SUMMARY

[0018] The inventors discovered that the amount of water which is distilled off from a leucoindigo salt solution has a crucial influence on the residual amount of aromatic amine in the leucoindigo solution. The inventors discovered that aqueous leucoindigo solutions having a very low content of aromatic amine such as aniline and N-methylaniline may be prepared if water is added to a non-purified leucoindigo solution comprising said amine commonly used in the industry in order to obtain a diluted leucoindigo solution, and subsequently water is distilled off in a weight which at least equals the weight of said aqueous leucoindigo solution prior to the addition of water. The more water is added and is subsequently distilled off the lower is the content of

aromatic amine. The thus obtained concentrated solutions contain less than 40 ppm aromatic amine, preferably less than 30 ppm or less than 10 ppm or less than 5 ppm. In a preferred example, solutions may be prepared in which said aromatic amine is no longer detectable. Such solutions may be termed as aniline-free. Accordingly, the term "*less than 100 ppm and in particular less than 40 ppm or less than 30 ppm or less than 20 ppm or less than 10 ppm*" encompasses as lower limit a limit at which the aromatic amine is no longer detectable, i.e. 0 ppm measured according to ISO 14362-1:2017(E).

[0019] A further particular advantage of the method according to the disclosure is that concentrated solution may be prepared, e.g. in the range of from 10 to 65 % by weight such as 15 to 60 % by weight or 20 to 55 % by weight or 25 to 50 % by weight, which comply with the dyeing requirements in terms of middle and deep shades, and which are aniline-free. Also the requirements of a reduced wastewater contamination in dyeing factories may be met.

[0020] Accordingly, the disclosure relates to the following items:

1. In one aspect, the present disclosure refers to method of removing an aromatic amine in the form of aniline or aniline and N-methylaniline from an aqueous leucoindigo solution comprising said aromatic amine in order to obtain a purified leucoindigo solution, wherein said leucoindigo is in the form of an alkali metal salt, the method comprising step (A1):

   (A1) subjecting the aqueous leucoindigo solution to distillation;

   wherein step (A1) further comprises one of the following steps (B1), (C1) or (D1):

   (B1) adding water to the aqueous leucoindigo solution prior to the distillation according to step (A1);

(C1) adding water to the aqueous leucoindigo solution simultaneously to the distillation according to step (A1);

(D1) adding water to the aqueous leucoindigo solution after the distillation according to step (A1) and redistilling the obtained solution;

wherein water is distilled off in a weight which at least equals the weight of said aqueous leucoindigo solution used in step (A1).

2. In another aspect, the present disclosure refers to method of making an aqueous aniline-free or aniline-free and N-methylaniline leucoindigo solution from an aqueous leucoindigo solution comprising aniline or aniline and N-methylaniline, the concentration of aniline or aniline and N-methylaniline being determined according to ISO 14362-1:2017(E), wherein said leucoindigo is in the form of an alkali metal salt, the method comprising step (A1):

(A1) subjecting the aqueous leucoindigo solution to distillation;

wherein step (A1) further comprises one of the following steps (B1), (C1) or (D1):

(B1) adding water to the aqueous leucoindigo solution prior to the distillation according to step (A1);

(C1) adding water to the aqueous leucoindigo solution simultaneously to the distillation according to step (A1);

(D1) adding water to the aqueous leucoindigo solution after the distillation according to step (A1) and redistilling the obtained solution;

wherein water is distilled off in a weight which at least equals the weight of said aqueous leucoindigo solution used in step (A1).

(γ2) feeding said water stream

into a distillation column configured to bring into contact said liquid stream with said aqueous stream;

(δ) discharging from the device water comprising aniline or aniline and N-methylaniline; and

(ε) discharging from the device aqueous an aniline-free or aniline-free and N-methylaniline-free leucoindigo solution.

14. Method of item 12 or 13, wherein said (γ1) liquid stream and said (γ2) water stream are fed into the column via inlets in the sidewalls of the column, and said (δ) water comprising aniline or aniline and N-methylaniline is discharged at the top of the column, and the (ε) aqueous aniline-free or aniline-free and N-methylaniline-free leucoindigo solution is discharged at the bottom of the column.

15. Method of any one of claims 13 or 14, wherein the distillation column is a column provided with trays or packing materials.

16. In yet another aspect, the present disclosure refers to stable aqueous leucoindigo solution comprising an aromatic amine in the form of aniline or aniline and N-methylaniline, wherein said leucoindigo is in the form of an alkali metal salt;

wherein the concentration of the aromatic amine is below 40 ppm determined according to ISO 14362-1:2017(E); and

wherein the concentration of the leucoindigo salt is in a concentration range of from 10 to 45 % by weight based on the total weight of the solution, and wherein the stability of the solution is measured at a temperature of 23 °C; or

wherein the concentration of the leucoindigo salt is in a concentration range of from 45 to 65 % by weight based on the total weight of the solution, and wherein the stability of the solution is measured at a temperature of 60 °C.

17. Stable aqueous leucoindigo solution of item 16, wherein the concentration of the aromatic amine is below 30 ppm or 20 ppm or 5 ppm.

18. In yet another aspect, the present disclosure refers to stable aqueous leucoindigo solution as defined herein, obtainable by a method as defined herein.

19. In yet another aspect, the present disclosure refers to method of making indigo, comprising step (D):

    (D) oxidizing the aqueous leucoindigo solution as defined herein.

20. Method of item 19, comprising step (C) prior to step (D):

    (C) treating a textile with the leucoindigo solution as defined in any one of items 16 to 18.

21. In yet another aspect, the present disclosure refers to method of making indigo, comprising steps (I) and (III)):

    (I) performing a method as defined herein;

    (III) oxidizing the leucoindigo solution obtained in step (I).

22. Method of claim 21, comprising step (II) prior to step (III):

    (II) treating a textile with the leucoindigo solution obtained in step (I).