CLAIMS

1. Method of making an aqueous aniline-free or aniline-free and N-methylaniline-free 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 at least steps (A) to (C):

(A) providing a liquid stream comprising said aqueous leucoindigo solution comprising said amine(s);

(B) providing a purification stream;

(C) bringing into contact said liquid stream with said purification stream;

wherein step (C) comprises steps (C1) and (C2), and the method further comprises steps (D) and (E):

(C1) feeding said liquid stream and

(C2) feeding said purification stream

into a device configured to bring into contact said liquid stream with said purification stream;

(D) discharging from the device at least a portion of said purification stream that has contacted said liquid stream; and

(E) discharging from the device at least a portion of said liquid stream that has been contacted by said purification stream;

wherein the device is selected from the group consisting of plate column, packed column, or bubble column, or two or three thereof, or two or more of any of said columns, and

wherein the column is operated at a pressure substantially equal to atmospheric pressure, i.e. in a range of from $0.95*10^5$ Pa absolute (0.95 bar) to $1.2*10^5$ Pa absolute (1.2 bar), wherein the temperature of the column is set in a range of from 95 °C to 110 °C; or

the column is operated at a pressure above atmospheric pressure, i.e. in a range of from $1.2*10^5$ Pa absolute (1.2 bar) to $11*10^5$ Pa absolute (11 bar) e.g. at $7*10^5$ Pa absolute (7 bar), wherein the temperature of the column is set in a range of from 110 °C to 190 °C;

wherein the temperatures quoted are measured at the bottom of the column.

2. Method of claim 1, wherein the liquid stream and the purification stream are counter-current streams.

3. Method of any one of the preceding claims, wherein the purification stream is a vapor stream.

4. Method of claim 3, wherein said vapor stream comprises an inert gas, a gaseous organic solvent which is not miscible with water, or steam, or two or three thereof.

5. Method of claim 4, wherein said steam is generated by heating at least a portion of said liquid stream that has been discharged from the device according to step (E).

6. Method of any one of claims 2 to 5, wherein steps (A) to (E) are performed simultaneously.

7. Stable aqueous leucoindigo solution, wherein said leucoindigo is in the form of an alkali metal salt; wherein the solution is aniline-free, wherein the term “aniline-free” defines an aniline concentration below 40 ppm, or is aniline-free and N-methylaniline-free, wherein the term “aniline-free and N-methylaniline-free” defines an aniline concentration and a N-methylaniline concentration below 40 ppm, as 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 40 % 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 40 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.

8. Method of making indigo, comprising step (II):

(II) oxidizing the aqueous leucoindigo solution as defined in claim 7.

9. Method of claim 8, comprising step (I) prior to step (II):

(I) treating a textile with the leucoindigo solution to be oxidized.

10. Method of making indigo, comprising steps (I) and (III):

(I) performing a method as defined in any one of claims 1 to 6;

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

11. Method of claim 10, comprising step (II) prior to step (III):

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

12. Stable aqueous leucoindigo solution according to claim 7,

wherein the concentration of the leucoindigo salt is in a concentration range of from 28 to 32 % by weight or 29 to 31 % by weight or in a concentration of 30 % 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; and

wherein the salt comprises a mixed sodium and potassium salt or is in the form of a mixed sodium and potassium salt, wherein sodium and potassium are present in a molar ratio of from 2.33 : 1 to 1 : 2.33.

13. Stable aqueous leucoindigo solution according to claim 7,

wherein the concentration of the leucoindigo salt is ~~in a concentration range of from 38 to 42 % by weight or 39 to 41 % by weight or~~ in a concentration of 40 % 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; and

wherein the salt comprises a mixed sodium and potassium salt or is in the form of a mixed sodium and potassium salt, wherein sodium and potassium are present in a molar ratio of from 2.33 : 1 to 1 : 2.33.