


**We claim,**

1. A process for industrial preparation of 3,3'-dichlorobenzidine dihydrochloride comprising the steps of:
  - i. reducing o-nitrochlorobenzene using formaldehyde in aqueous caustic lye solution in presence of a reduction promoter and dispersing/foaming agent, followed by water treatment to obtain 2,2'-dichloroazoxybenzene in pure form;
  - ii. hydrogenating 2,2'-dichloroazoxybenzene using activated Raney Nickel in DMF under inert atmosphere in presence of caustic lye to obtain 2,2'-dichlorohydrazobenzene;
  - iii. mixing 2,2'-dichlorohydrazobenzene with toluene to get HBT (2,2'-dichlorohydrazobenzene in toluene);
  - iv. filtering the HBT to remove byproduct o-chloroaniline along with Raney Nickel, followed by washing with HCl solution to get pure HBT;
  - v. treating pure HBT with H<sub>2</sub>SO<sub>4</sub> solution at a temperature of 20<sup>0</sup>C - 25<sup>0</sup>C under chilling to get 3,3'-dichlorobenzidine sulfate;
  - vi. isolating 3,3'-dichlorobenzidine sulfate on hot water treatment at 90<sup>0</sup>C;
  - vii. precipitating 3,3'-dichlorobenzidine dihydrochloride directly from 3,3'-dichlorobenzidine sulfate by the addition of NaCl solution and
  - viii. filtering, centrifuging and drying of 3,3'-dichlorobenzidine dihydrochloride to get it in powder form.
2. The process as claimed in claim 1, wherein Diclone is used as a reduction promoter.
3. The process as claimed in claim 1, wherein Dinol 25 is used as dispersing/foaming agent.

4. The process as claimed in claim 1, wherein caustic lye in step (i) is used in 48% concentration.
5. The process as claimed in claim 4, wherein the said aqueous caustic lye solution is cooled to 40<sup>0</sup>C, before reacting with o-nitrochlorobenzene.
6. The process as claimed in claim 1, wherein pure 2,2'-dichloroazoxybenzene is isolated at temperature of 70<sup>0</sup>C.
7. The process as claimed in claim 1, wherein 2,2'-dichlorohydrazobenzene is isolated from its alkaline mother liquor under inert conditions at 90<sup>0</sup>C.
8. The process as claimed in claim 1, wherein the formaldehyde used is in 37% concentration and is continuously added at a temperature range of 50-55<sup>0</sup>C.
9. The process as claimed in claim 1, wherein the hydrogenation of 2,2'-dichloroazoxybenzene is conducted at a temperature range of 65-85<sup>0</sup>C.
10. The process as claimed in claim 1, wherein, the alkalinity is maintained at 3% during the hydrogenation reaction.
11. The process as claimed in claim 1, wherein 3% HCl is used for washing to remove byproduct o-chloroaniline and Raney Nickel.
12. The process as claimed in claim 1, wherein 56%-58% H<sub>2</sub>SO<sub>4</sub> is used for the rearrangement of pure HBT.
13. The process as claimed in claim 1, wherein the NaCl solution used is in 30% concentration.

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