Synthesis of hexaamminecobalt(III) chloride

Summary of the analysed protocols

$2CoCl_2 + 10NH_3 + 2NH_4Cl + H_2O_2 \rightarrow 2[Co(NH_3)_6]Cl_3 + 2H_2O$ (1)
$4CoCl_2 + 4NH_4Cl + 20NH_3 + O_2 \rightarrow 4[Co(NH_3)_6]Cl_3 + 2H_2O$ (2)
$4Co(C_2H_3O_2)_2.4H_2O + 4NH_4C_2H_3O_2 + 20NH_3 + O_2 + 12HCl \rightarrow 4[Co(NH_3)_6]Cl_3 + 18H_2O + 12CH_3COOH$ (3)
Protocol A[1,2] **Reaction (R_1):** equation (1), 195% exc. ammonium chloride, 608% exc. ammonia solution, 271% exc. hydrogen peroxide, water and activated charcoal (auxiliary substances), 0 ºC < T < 100 ºC **Isolation (I_1):** filtration (suction) **Purification (Pu_1):** recrystallization – dissolution (hot water and concentrated hydrochloric acid) → heating → filtration → cooling (ice bath) → filtration → washing (cold ethanol) → drying (air)
Protocol B[3] **Reaction (R_1):** ≡ Pr A (scale decreased to half) **Isolation (I_1):** ≡ Pr A **Purification (Pu_1):** ≡ Pr A (scale decreased to half)
Protocol C[4] **Reaction (R_2):** equation (1), 195% exc. ammonium chloride, 608% exc. ammonia solution, 462% exc. hydrogen peroxide, water and activated charcoal (auxiliary substances), 0 ºC < T < 100 ºC **Isolation (I_1):** ≡ Pr A **Purification (Pu_1):** ≡ Pr A
Protocol D[5] **Reaction (R_3):** equation (1), 195% exc. ammonium chloride, 74% exc. ammonia solution, 84% exc. hydrogen peroxide, water and activated charcoal (auxiliary substances), 0 ºC < T < 100 ºC **Isolation (I_1):** ≡ Pr A **Purification (Pu_2):** recrystallization – dissolution (water and boiling concentrated hydrochloric acid) → filtration → cooling (ice bath) → filtration (suction) → drying (air)
Protocol E[6,7] **Reaction (R_4):** equation (1), 195% exc. ammonium chloride, 74% exc. ammonia solution, 66% exc. hydrogen peroxide, water and activated charcoal (auxiliary substances), T ~ 100 ºC **Isolation (I_1):** ≡ Pr A **Purification (Pu_3):** recrystallization – dissolution (water and boiling concentrated hydrochloric acid) → filtration → cooling (ice bath) → filtration
Protocol F[8] **Reaction (R_5):** equation (1), 195% exc. ammonium chloride, 74% exc. ammonia solution, 66% exc. hydrogen peroxide, water and activated charcoal (auxiliary substances), T ~ 100 ºC **Isolation (I_1):** ≡ Pr A **Purification (Pu_4):** recrystallization – dissolution (water and boiling concentrated hydrochloric acid) → filtration → cooling (ice bath) → filtration (suction) → washing (acetone) → drying (desiccator)
Protocol G[9] **Reaction (R_6):** equation (1), 185% exc. ammonium chloride, 103% exc. ammonia solution, 808% exc. hydrogen peroxide, water and activated charcoal (auxiliary substances), T ~ 100 ºC **Isolation (I_2):** filtration (suction) → washing (cold water) **Purification (Pu_5):** recrystallization – dissolution (hot water and concentrated hydrochloric acid) → filtration → cooling (ice bath) → filtration (gravity) → washing (ethanol → ethyl ether) → drying (air)
Protocol H[10] **Reaction (R_7):** equation (2), 200% exc. ammonium chloride, 65% exc. ammonia solution, water and activated charcoal (auxiliary substances), room temperature **Isolation (I_1):** ≡ Pr A **Purification (Pu_6):** recrystallization – dissolution (water and concentrated hydrochloric acid) → heating → filtration → cooling (ice bath) → filtration → washing (ethanol) → drying (oven at 80-100 ºC)

Protocol I[11]
Reaction (R_7): ≡ Pr H (scale enlarged 5 times) **Isolation (I_1):** ≡ Pr A **Purification (Pu_6):** ≡ Pr H (scale enlarged 5 times)
Protocol J[12]
Reaction (R_8): equation (2), 200% exc. ammonium chloride, 72% exc. ammonia solution, water and activated charcoal (auxiliary substances), room temperature **Isolation (I_1):** ≡ Pr A **Purification (Pu_7):** recrystallization – dissolution (water and concentrated hydrochloric acid) → heating → filtration → cooling (ice bath) → filtration (suction) → washing (ethanol) → drying (air)
Protocol K[13,14]
Reaction (R_9): ≡ Pr H (scale enlarged 25 times, but with different amount of activated charcoal) **Isolation (I_1):** ≡ Pr A **Purification (Pu_8):** ≡ Pr H (but with different amounts of solvents)
Protocol L[15]
Reaction (R_{10}): equation (3), stoichiometric proportions of cobalt(II) acetate, ammonium acetate and hydrochloric acid, methanol and activated charcoal (auxiliary substances), room temperature **Isolation (I_3):** filtration → washing (ethanol → ethyl ether) → drying (air) **Purification (Pu_9):** recrystallization – dissolution (hot water and concentrated hydrochloric acid) → filtration → washing (ethanol → ethyl ether) → drying (air)

$^{\alpha}$ → – Sequential

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